**COAL SURFACE CONTROL FOR
ADVANCED FINE COAL FLOTATION**

Project No. DE-AC22-88PC88878

Final Report
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Final Technical Report

(October 1, 1988 - December 31, 1991)

EXECUTIVE SUMMARY

This final report, submitted to DOE for the project entitled "Coal Surface Control for Advanced Fine Coal Flotation," contains a comprehensive summary of work completed during the contract period, October 1, 1988 through December 31, 1991. The project team consisted of research and engineering groups at the University of California at Berkeley, Columbia University, the University of Utah, and Praxis Engineers, Inc., with the University of California acting as the prime contractor with DOE.

The initial goal of the research project was to develop methods of coal surface control in advanced froth flotation to achieve 90% pyritic sulfur rejection, while operating at Btu recoveries above 90% based on run-of-mine quality coal. Moreover, the technology is to concomitantly reduce the ash content significantly (to six percent or less) to provide a high-quality fuel to the boiler (ash removal also increases Btu content, which in turn decreases a coal's emission potential in terms of lbs SO₂/million Btu).

Upon the award of the contract, the initial effort was focused on the collection of samples from six coal seams, three designated as base coals and three as additional coals, to provide samples for research work, coal weathering studies and washability testing. The three base coals, which were selected as representative of important high-pyritic-sulfur coals, were Illinois No. 6 from Randolph County, Illinois; Pittsburgh No. 8 from Belmont County, Ohio; and Upper Freeport PA from Indiana County, Pennsylvania. The additional coal samples, comprised of two eastern high-sulfur coals and one western low-sulfur sub-bituminous coal, were Kentucky No. 9 from Hopkins County, Kentucky; Upper Freeport WV from Grant County, West Virginia; and Wyodak from Campbell County, Wyoming. In order to ensure uniformity of samples for the duration of this project, as well as for the selective oil agglomeration project being conducted at the University of Pittsburgh, DOE consolidated the sample procurement task into a single effort

referred to as Coal Procurement and Weathering (Task 2). This aspect of the program was carried out by the team from Praxi Engineers. About 23 tons of each base coal and five tons of each additional coal were collected from six operating open-pit mines. Care was taken to remove the overburden and floor shale material. Each base coal sample was reduced to a topsize of 4 to 6 inches, homogenized and split into two parts, one for use in the weathering studies and the other for research and washability studies. The research and washability sample was split into 400-lb lots using 20-lb increments and packed into 55 gallon drums, inerted with argon and then distributed. The additional coal samples were also reduced to a topsize of 4 to 6 inches, homogenized and packed in 400-lb lots, and inerted with argon prior to shipping. Approximately 9 tons of coal (research and weathering samples) was shipped to the University of California, 6 tons to the University of Pittsburgh, and 6 tons of coal to commercial testing laboratories for washability testing. Over 42 tons of coal was used to set up the weathering studies of the three base coals at the three mine sites. Another 21 tons was stored in inerted drums as a reserve sample for future use on this project or on other projects funded by DOE.

Bulk Characterization of the Coal Samples

The three base coals were characterized by a variety of different techniques. Bulk characterization studies included the determination of chemical composition by proximate, ultimate and sulfur-forms analyses. From the proximate and calorific value analyses, the three base coals have the following order for their characteristics:

Coal Characteristic	Illinois No. 6	Pittsburgh No. 8	Upper Freeport PA
Moisture	9.5%	2.3%	1.0%
Volatile Matter	36.2	35.7	26.2
Ash	17.5	11.8	12.0
Fixed Carbon	46.3	52.5	61.8
Calorific value, Btu/lb	11,300	12,400	13,400

Sulfur forms analysis showed the following order for the three base coals:

Sulfur Forms	Illinois No. 6	Pittsburgh No. 8	Upper Freeport PA
Total sulfur	5.37%	4.71%	2.35%
Pyritic sulfur	2.56	2.82	1.62
Organic sulfur	2.53	1.64	0.63

The pyritic sulfur as a percentage of the total sulfur is the highest in Upper Freeport PA (69 wt%), followed by Pittsburgh No. 8 (60 wt%) and Illinois No. 6 coal (48 wt%). This implies that the application of physical separation techniques, such as froth flotation and selective agglomeration, should potentially be most effective for lowering the sulfur content of Upper Freeport PA coal, followed by Pittsburgh No. 8 and Illinois No. 6 coals, assuming equal liberation and rejection of the pyritic sulfur.

Examination of the ultimate (elemental) analysis results shows that the bulk carbon-to-oxygen ratio (number in the parentheses) follows the order:

Upper Freeport PA (19.9) > Pittsburgh No. 8 (11.1) > Illinois No. 6 (10.5).

The dry mineral matter free (dmmf) carbon content of the three coals is:

Upper Freeport PA (85.9% dmmf C) > Pittsburgh No. 8 (80.5%) > Illinois No. 6 (77.3%).

This order agrees with the hydrophobicity and the flotability of the three base coals.

Qualitative analysis of the mineralogical content of the ash minerals contained in the coal was conducted using diffuse reflectance infrared Fourier transform (DRIFT) spectroscopy. It was concluded that all three samples contain kaolinite and quartz. In addition, peaks characteristic of calcite were present in the spectra of the ash of Illinois No. 6 coal but were not present in the other two base coals. Neutron activation analysis shows that the Illinois No. 6 sample contains 0.7% calcium whereas Pittsburgh No. 8 and Upper Freeport PA samples contain less than 0.1%.

As part of the bulk characterization studies, photomicrographic studies using SEM/EDXRF (scanning electron microscopy/energy dispersive x-ray fluorescence) and optical image analyses were also conducted to determine pyrite and non-pyritic mineral matter association in the coal. The SEM/EDXRF study consisted of a qualitative analysis of the distribution of pyrite and non-pyritic mineral matter in narrow particle size ranges, finer than 28 mesh. The results of this study indicated that there was almost no liberated pyrite in the 28-mesh samples of Illinois No. 6 or Pittsburgh No. 8 coal, whereas free particles of pyrite did exist in the 200-mesh samples. Optical image analysis was carried out to quantitatively analyze the distribution of locked, semi-locked and

free pyrite grains in different size fractions within the minus 200-mesh grind (in volume percent).

Coal	Locked	Semi-Locked	Free
Illinois No. 6	12 %	12 %	76 %
Pittsburgh No. 8	38	23	39
Upper Freeport PA	14	15	71

This analysis showed that the pyrite present in Illinois No. 6 and Upper Freeport PA coal was primarily liberated, whereas that in the Pittsburgh No. 8 coal was not. Petrographic analysis was also conducted to determine the maceral composition of the three base coals. In all three cases, vitrinite was the major component. About 11 to 15 percent inertinite was present in all three coals and 10% liptinite in Pittsburgh No. 8 and Illinois No. 6 coal. In the case of Upper Freeport PA coal, only 0.2% liptinite was present.

Washability Studies

Washability studies were conducted at three different test laboratories on the original 4-inch topsize samples as well as on representative samples that had been comminuted to pass 1-1/2 inch, 1/8-inch, 28 mesh and 200 mesh. The results for the three base coals, which are presented in this report, show that, with a perfect gravity separation, over 90 percent of the pyritic sulfur can be rejected at 90 percent Btu recovery at a specific gravity of 1.6 for all three coals at a nominal 200-mesh grind size. Considerable difficulty was experienced in the sink-float testing of Illinois No. 6 coal at fine particle size and at low specific gravity, presumably due to agglomeration of the fine coal and to the presence of clays. These complications arose even though the sink-float tests for the 28-mesh and 200-mesh grinds were performed following DOE PETC's recommended procedure for all tests, which included the addition of a dispersant to prevent agglomeration and centrifugation to help effect a sharp gravity split. A comparison of the liberation-characteristics of each coal can be made from the pyrite rejection at a given Btu recovery as a function of particle size. At 90% Btu recovery, the pyritic sulfur content of the float product and the pyritic sulfur rejection have the following dependence on the topsize of each coal:

Top size	Illinois No. 6		Pittsburgh No. 8		Upper Freeport PA	
	<u>Pyr. S</u>	<u>Pyr. S Rej.</u>	<u>Pyr. S</u>	<u>Pyr. S Rej.</u>	<u>Pyr. S</u>	<u>Pyr. S Rej.</u>
4 inches	1.4%	60%	2.1%	40%	0.8%	52%
1 1/2 in.	1.2	65	1.9	45	0.75	52
1/8 in.	0.9	75	1.3	60	0.7	63
28 mesh	0.6	83	0.8	75	0.3	84
200 mesh	0.25	94	0.3	93	0.1	94

The results show that when the top size of the crushed material is 28 mesh or coarser, none of the three base coals exhibits significant pyrite liberation, and the pyritic sulfur rejection is the least for Pittsburgh No. 8 coal at 90% Btu recovery, indicating a greater degree of pyrite dissemination throughout the coal in this material. However, all of the samples show that more than 90% of the pyritic sulfur can be rejected by sink-float techniques at a specific gravity of 1.6 if they are ground to minus 200 mesh (74 microns). Similar behavior was observed in the washability tests of the additional coals (Upper Freeport WV, Kentucky No. 9 and Wyodak).

Because liberation occurs at fine sizes and in view of heightened interest in fine coal beneficiation, a more detailed study of the washability of fine coal was undertaken. Reserve samples of the coal, which had been used in the original washability study and had been stored in sealed "inerted" 55-gallon drums, were comminuted to minus 100, 200, 325, and 400 mesh for these tests. For all three coals, there was enough liberation of pyrite at the 100-mesh grind size to achieve 90% pyritic sulfur rejection with 90% Btu recovery. There were only slight differences in the washability results between the coals comminuted to 100, 200, 325, and 400-mesh grind sizes, as can be seen from comparison of the pyritic sulfur rejection at 90% Btu recovery for each coal:

Top size	Illinois No. 6	Pittsburgh No. 8	Upper Freeport PA
100 mesh	90%	91%	93%
200 mesh	91	91	96
325 mesh	94	93	96
400 mesh	95	94	96

These results indicate that by grinding the coals to minus 100 mesh (150 microns), it is possible to achieve 90% pyritic sulfur rejection at 90% Btu recovery. However, it is important to note that these separations are based only on the specific gravity of the particles and provide

a indication of mineral liberation, but not on pyrite separability by processes based on other phenomena, such as surface behavior. Because the separation in washability tests is based on the specific gravity of the entire particle, a locked coal-pyrite particle can also be rejected if the overall specific gravity of the locked particle is higher than the liquid density used in the separation.

Wettability and Surface Characterization

The second part of the general characterization studies was directed at delineating the nature of surface properties and included analysis of both macroscopic and microscopic properties. Macroscopic properties evaluated included the wettability behavior (by film flotation, contact angle and induction time measurements) and surface charge characteristics (as given by zeta potential measurements). Microscopic characterization of the coal included surface elemental composition analysis by ESCA (Electron Spectroscopy for Chemical Analysis) and determination of organic functional groups by DRIFT.

Results of the wettability analyses indicate, among other things, that the hydrophobicity of the coal follows the order:

Upper Freeport PA > Pittsburgh No. 8 > Illinois No. 6.

This order was confirmed by film flotation, contact angle measurements and induction time measurements. Contact angles calculated from film flotation results and measured by the captive bubble techniques and the induction time for bubble-particle contact for each of the three coals exhibited the following behavior:

Wettability Parameters	Illinois No. 6	Pittsburgh No. 8	Upper Freeport PA
Contact angle, degrees (film flotation)	52	63	63
Contact angle, degrees (captive bubble)	44	60	62
Induction time, μ sec	475	180	75

The very sensitive induction time measurements show that Illinois No. 6 coal is considerably more hydrophilic than is Upper Freeport PA coal.

Zeta potential measurements show that the isoelectric point (IEP), that is the condition (pH) under which the zeta potential is reversed, of the three base coals decreases in the following order:

Upper Freeport PA (pH 5.5) > Pittsburgh No. 8 (pH 3.5) > Illinois No. 6 (pH 2.5).

The order of both hydrophobicity and the IEP of the three base coals is the same as that of the bulk carbon-to-oxygen ratio and the dmmf carbon content of the three coals.

The surface area measurements using carbon dioxide as an adsorbate gas show that Illinois No. 6 coal has the highest specific surface area ($172 \text{ m}^2/\text{g}$), followed by Pittsburgh No. 8 ($150 \text{ m}^2/\text{g}$) and Upper Freeport PA ($147 \text{ m}^2/\text{g}$). The surface elemental composition determined by ESCA and the oxygen functional groups determined by DRIFT of the three base coals were not very conclusive.

Standard Grinding Tests

For preparing the feed for the flotation experiments, the coal was first crushed to minus 1/4-inch under inert conditions, and then ground in a stainless steel laboratory rod mill to produce material that was either 95% minus 28 mesh or 95% minus 200 mesh. For each grind, 500 grams of coal was charged to the mill so that four 125-gram samples could be produced to be used in each series of flotation tests. Stainless steel rods rather than balls were used as the grinding media because the product size distribution produced by rod action more closely simulates the product produced by an industrial ball mill/classifier circuit. For each coal and each grind, whether wet or dry, a standard grinding condition was developed, including rod charge and grind time, to prepare flotation feed with a size distribution of 95% passing either 28 mesh or 200 mesh. Identical mills and rods were located at each research site so that identical test feeds could be prepared at each site. To increase the time (for better control) required for grinding the coal to 28 mesh, the mill was charged with only 24 rods, whereas for grinding to 200 mesh, the number of rods was increased to 42. From the standard grinding tests, the relative grindability

of the three coals can be seen (here relative grindability is expressed as mass of product produced per unit of energy relative to dry grinding Illinois No. 6 coal either to minus 28 or 200 mesh).

Grinding Mode	Illinois No. 6	Pittsburgh No. 8	Upper Freeport PA
Dry grind to 28 mesh	1.0	1.7	2.8
Wet grind to 28 mesh	2.4	2.7	4.6
Dry grind to 200 mesh	1.0	1.6	2.7
Wet grind to 200 mesh	3.2	2.9	4.2

Clearly, wet grinding is more energy efficient than dry grinding for all three coals, and Upper Freeport PA coal requires the least amount of grinding energy, either dry or wet. The order of the grindability of these three coals is:

Dry: Upper Freeport PA > Pittsburgh No. 8 > Illinois No. 6

Wet: Upper Freeport PA > Pittsburgh No. 8 ≈ Illinois No. 6

Standard Flotation Tests

A standard flotation test was developed in order to evaluate the effects of the grinding environment and the addition of surface modifying reagents on the flotation response of the coals. For these standard tests, a 2-liter Denver Laboratory flotation machine was used and configured according to the DOE (PETC) design. The standard tests developed for each coal are essentially identical to each other except for the dosage of collector and frother added to the flotation cell. Pure dodecane was selected as the collector and MIBC (methylisobutylcarbinol) as the frother. Extensive preliminary studies showed that the amount of collector and frother required are coal-specific and depend on the inherent hydrophobicity of the coal. The standard dosages were selected so that the flotation recovery was between 60 and 80 percent, but no attempt was made initially to optimize these tests. Further, separate standard tests were developed for each coal sample with respect to whether it had been ground dry or wet or to a nominal topsize of 28 mesh or 200 mesh. This resulted in four standard flotation reagent additions for each coal. Except for the collector and frother dosage, all other variables (both physical and operating variables) were the same for all three coals, regardless of the grind size or grinding environment. How coal hydrophobicity determined the amount of reagents required

to achieve the desired yield under standard operating conditions can be seen from a summary of the standard reagent additions for the three coals dry ground to 28 mesh and to 200 mesh.

Reagent, grind	Illinois No. 6	Pittsburgh No. 8	Upper Freeport PA
Collector, 28 mesh	4.80 lb/T	1.90 lb/T	0.24 lb/T
Frother	1.17	0.39	0.26
Collector, 200 mesh	5.76	2.16	0.48
Frother	1.17	0.39	0.21

Clearly, the reagent requirements show that the order of hydrophobicity for the three coals is

Upper Freeport PA > Pittsburgh No. 8 > Illinois No. 6

The results from the Standard Flotation Tests for the three base coals, including the efficiency indices, are the following:

Grind	Flotation Yield	Illinois No. 6	Pittsburgh No. 8	U Freeport PA
28 mesh, dry grind	Yield, %	75	82	81
	CMR, %	80	87	86
	Pyr. S, %	1.76	1.99	0.69
	Ash Rej., %	54	55	57
	Pyr. S Rej., %	49	42	66
	El _H	29	29	52
200 mesh, dry grind	Yield, %	60	78	59
	CMR, %	65	83	63
	Pyr. S, %	1.36	1.37	0.67
	Ash Rej., %	64	63	70
	Pyr. S Rej., %	68	62	76
	El _H	33	46	39
200 mesh, wet grind	Yield, %	77	76	68
	CMR, %	83	82	74
	Pyr. S, %	1.57	1.20	0.57
	Ash Rej., %	58	70	71
	Pyr. S Rej., %	53	68	79
	El _H	36	50	52

An extensive QA/QC program shows that the results of both the standard flotation tests and coal analysis obtained in the three different research sites (universities) agree quite well. Although increased efficiency of separation is always achieved by refloating the concentrate, the standard test was designed to be single-stage only for simplicity.

The so-called Hancock efficiency index was used for assessing the separation efficiency of the various flotation experiments. This index simplifies down to the sum of the combustible matter recovery plus the pyritic sulfur rejection minus 100. Thus, if the initial project objective of 90% Btu recovery at 90% pyritic sulfur rejection were achieved, the efficiency index (EI_H) would be 80. For 200-mesh wet-ground coal samples, the separation efficiency for the flotation separation of Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA coals under the standard test conditions is 36, 50 and 52, respectively.

Optimization of the reagent addition with Illinois No. 6 indicated that the reagent dosages used in the Standard Flotation Test were almost identical to the optimized dosage, while the results of optimization tests of flotation machine parameters with Pittsburgh No. 8 showed that the flotation machine parameters had little effect on the efficiency of separation and that the standard tests also produced results close to the optimum.

In order to determine the best results achievable with dodecane and MIBC using flotation, release analysis curves were determined for 200-mesh grinds of each base coal. The maximum separation efficiency obtained from the release analysis for Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA coals is 65, 56 and 64, respectively. These numbers are to be compared with those obtained from the standard flotation tests, namely 36, 50 and 52, respectively. As expected, the results of the release analysis (flotation separation) for all three base coals are not as good as those of the gravity-based washability tests, which can achieve a pyritic sulfur rejection of 90% at a Btu recovery of 90% with a separation efficiency of 80 at a 200-mesh grind. This is because washability tests (if done correctly) make a separation based on particle composition (specific gravity) whereas flotation makes a separation based on surface composition and relative surface hydrophobicity. Release analysis curves provide a good base for assessing the flotation performance of various reagents used in comparing flotation separations.

A procedure was developed for estimating the pyritic sulfur content of coal separation products from their total sulfur and ash content. By assuming that the organic sulfur content in the combustible material in a coal is constant, a linear relationship is found among the pyritic sulfur, total sulfur and the ash content of a coal sample. This linear relationship has been confirmed from analysis of the products obtained from the various flotation and washability tests.

The values of the proportionality constants in the linear equation for a specific coal are first determined from the experimental pyritic sulfur, total sulfur and ash contents of several coal separation products of the same coal using linear regression analysis. After the parameters in the equation have been established for a particular coal, the pyritic sulfur content of other separation products of the same coal can be calculated based on their total sulfur and ash content using the equation. The results have shown that the calculated pyritic sulfur contents of various coal samples agree very well with their experimental values. The main advantage of this procedure is that it is reliable and rapid because total sulfur and ash content of coal can be determined easily using microprocessor-controlled machines.

Effect of Grinding Environment

A study of the grinding environment indicated that wet grinding resulted in a higher flotation yield and combustible material recovery than dry grinding for 200-mesh Illinois No. 6 and Upper Freeport PA coals at the same standard reagent dosages, respectively. A further study on behavior of 200-mesh Pittsburgh No. 8 coal showed that the separation efficiency of wet-ground samples is significantly higher than that of dry-ground samples. On the other hand, grinding in an inert atmosphere and floating with either air or argon as the carrier gas had little effect on the flotation performance for all three coals, as compared to grinding and floating the coal in an uncontrolled atmosphere. Therefore, it was concluded that grinding and floating the coal in air (uncontrolled atmosphere) should be satisfactory for industrial purposes. Grinding the coal wet should also be used industrially because of the reduced energy consumption for grinding. With respect to the points of reagent addition, both the flotation yield and the flotation rate constant of hydrophilic Illinois No. 6 coal were higher when the collector was added to the mill before grinding than when the collector was added to the cell after grinding. However, for Pittsburgh No. 8 and Upper Freeport PA coals (more hydrophobic coals), the flotation yield is lower when the standard dosage of the collector is added to the mill before grinding. Similarly, the flotation yield is significantly reduced when the standard dosage of the frother was added to the mill for all three coals. Finally, grinding coal in a ceramic mill was not found to improve the flotation performance of the coal studied.

Coal Flotation Kinetics

A flotation kinetic study was undertaken to systematically delineate the effect of particle size on the recovery and rate constants for the flotation of coal and the associated mineral matter. For this purpose, the three bituminous coals were ground wet to produce flotation feed at three different top sizes, namely 28, 100 and 200 mesh. This study showed that in the batch flotation, the kinetic behavior of coal flotation can most simply be described by a first-order kinetic model with a sine function (in the range of 0 to $\pi/2$) of flotabilities for the floatable particles. The ultimate recovery and flotation rate constant obtained by fitting experimental results with this kinetic model represent the ultimate recovery of coal and the rate at which this final state can be reached, respectively.

The ultimate recovery of the combustible material is significantly higher than that of ash and pyrite, whereas the flotation rate constant of the combustible material is only slightly higher than that of ash and pyrite. This may indicate that the floated portion of ash and pyrite is either locked with coal or carried over by the froth with the coal.

At constant reagent dosages, the flotation rate constant decreases significantly as the grind size of the flotation feed is decreased, due not only to the low collector coverage on the surface of the coal particles but also to froth overloading during the first half minute of flotation of the fine feed.

The effect of particle size on the flotation recovery and rate constant of coal is not as pronounced as in mineral flotation due to the low specific gravity and the hydrophobic nature of coals. The effect of particle size is small for the more hydrophobic coals, higher reagent dosages and smaller top feed size of coal. For minus 28-mesh coal, the intermediate size fractions (48 x 100 and 100 x 200 mesh) exhibit higher ultimate combustible material recovery than the larger (plus 48 mesh) and the smaller (minus 200 mesh) size fractions for all three coals. The flotation rate constant of Illinois No. 6 coal also exhibits a maximum at intermediate sizes, whereas the rate constant of Pittsburgh No. 8 and Upper Freeport PA coals increases monotonically with the increasing particle size. For 100-mesh grinds, the ultimate CMR increases and the flotation rate constant decreases with decreasing particle size. For 200-mesh grinds, both the ultimate CMR and the rate constant increase as the particle size is decreased.

An increase in reagent dosage for minus 200-mesh coal increases the flotation recovery and the rate constant of the plus 400-mesh size fraction more than that of the minus 400-mesh size fraction. At the same time, the increase in pyrite recovery is greater than that of coal, resulting in an overall decrease in the separation efficiency. In general, low recovery of the relatively coarse coal particles and high recovery of fine pyrite particles are the major problems in separating pyrite from fine coal using froth flotation.

Surface Modification

Surface modifiers, which are reagents that interact with or adsorb on the surface of coal or on pyrite and thereby should make the coal surface more hydrophobic or pyrite more hydrophilic, were tested to enhance the separation of pyrite from coal by advanced froth flotation. The effect of pH, controlled by the addition of lime, was studied as a first step and the results showed that the flotation yield of 28-mesh dry-ground Pittsburgh No. 8 coal decreased when the pH was raised above 10. At the same time, there was a steady increase in the pyritic sulfur rejection from minus 28-mesh coal, prepared either by dry grinding or wet grinding. The drop in the yield and the efficiency index above pH 6 was more dramatic for the minus 200-mesh dry-ground material and above pH 8 for the minus 200-mesh wet-ground coal. Similar results were obtained for Illinois No. 6 coal. The Upper Freeport PA sample did not exhibit any drop in yield up to pH 10. Because the flotability of both coal and pyrite decreases at pH's above 8, rejection of pyrite at high Btu recovery can not be accomplished solely by controlling the pH.

Although the effect of pH on the flotation behavior was easily observed, the physical changes occurring on the surfaces of the coal and pyrite upon change in pH, were not obvious and an extensive study was undertaken to try to elucidate the underlying phenomena. The major finding of this investigation was that the pH at which the grinding step is carried out is extremely important. When coal is ground at low pH, various soluble metal cations are introduced into the coal slurry and if the pH is subsequently increased, these cations begin to precipitate as hydroxides, often onto the surface of coal and pyrite particles, causing the yield to decrease. On the other hand, if the coal is ground at a higher pH, metal cations are not solubilized and therefore do not subsequently precipitate during flotation. It was found that when

Pittsburgh No. 8 coal was ground and floated at relatively high pH (6-10), the flotation yield is higher than when the coal was ground at natural pH and subsequently floated at high pH. The overall effect of grinding and floating at the same pH results in an increase in the separation efficiency.

One series of modifiers tested involved various anionic surfactants. Coal surface modification was attempted by adding these surfactants to the flotation cell and evaluating their effect on the flotation product. Of the anionic reagents tested, only Aerosol OT (2-ethylhexylsulfosuccinate) resulted in a small improvement in the flotation response of Pittsburgh No. 8 coal, using the standard collector and frother dosages. Other anionic surfactants tested, 4-t-butylpyridine and 2-(1-butylpentyl)pyridine, do not appear to have promise as flotation reagents for aiding coal desulfurization.

A number of nonreactive nonpolymerizable reagents, such as methanol, ethanol, alkylbenzaldehyde and glyoxal, were also tested to determine their effect on the flotation performance of the three base coals. The addition of methanol during the wet-grinding of Upper Freeport PA coal to minus 200 mesh resulted in a combustible material recovery of 86% and a pyritic sulfur rejection of 81% ($EI_H = 67$), representing a substantial improvement over the standard flotation test. However, no such improvement was observed for Pittsburgh No. 8 or for Illinois No. 6 coal. The improvement obtained with methanol suggested that other alcohols may also have beneficial effects on flotation. While the improvements in performance for Upper Freeport PA coal using ethanol were comparable to methanol, some improvements were achieved with Pittsburgh No. 8 coal but none with Illinois No. 6 coal. Tests with butylbenzaldehyde, another non-polymerizable reagent, did not result in any improvement in performance over the standard test. In addition to these non-polymerizable reagents, a number of other organic reagents that have functional groups which might interact with coal surface sites and possibly alter the flotation behavior were also examined. These included ethylene glycol, polyethylene glycol, succinic acid, pentyl ether, butylamine and dipentylamine. However, none of these reagents was found to improve the pyrite separation efficiency over that obtained in the standard tests.

Organic monomers, which can polymerize to form large hydrocarbon macromolecules, were added during grinding to determine if mill conditions were conducive for the polymerization

reaction to occur between the monomer and newly generated coal surface sites. For a number of practical considerations vinyl acetate, styrene, diisobutylene and methyl methacrylate were selected for testing. The coals were dry ground with the monomers in an argon-purged rod mill. Some increase in yield was observed. After extensive testing, observed increases in flotation yield were attributed to an increase in the amount of hydrocarbons present during flotation and not due to graft polymer formation because saturated analogs of the monomers such as ethyl benzene, ethyl acetate and 1,1,4-trimethylpentane affected flotation to the same extent. Commercially available monomers are generally available only with an inhibitor added to prevent violent polymerization from occurring. Since the inhibitor may prevent polymerization with coal surface sites, vinyl acetate was distilled to remove the inhibitor, thereby obtaining a pure monomer for testing. The tests using vinyl acetate without inhibitor did not result in any improved flotation performance over that in which the inhibitor was present. This research indicates that without the addition of external free-radical initiators, the formation of in-situ graft polymers on the coal surface does not appear to be a technically feasible approach in advanced coal flotation.

The function of dodecane in the flotation of coal is to enhance the hydrophobicity of the coal particles. However, a detailed study of dodecane addition on coal flotation showed that dodecane adsorption is non-selective and at high dosages, pyrite and ash are also rendered hydrophobic. In order to obtain better separation efficiency, lower dodecane dosages and higher MIBC dosages should be used in the flotation. For a relatively hydrophilic coal such as Illinois No. 6, larger dodecane additions are required to recover the combustible material, which contributes to increasing the flotation of pyrite and ash in the clean coal. For this reason, a series of nonionic surfactants were tested to determine if they provided better flotation selectivity than dodecane for hydrophilic coal. These reagents, oxygenated alkyl aromatic compounds, were found to be powerful collecting reagents for Illinois No. 6 coal at reagent additions as low as 1/10th of that of dodecane. The oxygen groups tended to decrease the separation efficiency of the reagents, with nonylbenzene giving the best separation with the purely alkyl aromatic series. These reagents have a similar effect on Pittsburgh No. 8 coal. Because the interaction of oxygenated compounds seems to be dependent on oxygen sites on the coal surface, this investigation was extended to the flotation of oxidized coals. Laboratory-oxidized

Pittsburgh No. 8 coal was floated with dodecane and with two different compounds of this series. Dodecane alone was not able to render the surface of the coal sufficiently hydrophobic, resulting in low combustible matter recovery (CMR values). However, the addition of oxygenated compound resulted in large CMR's at relatively low dosages as compared to that of dodecane.

The effect of commonly used dispersants, namely sodium silicate and sodium hexametaphosphate, on the separation efficiency of Pittsburgh No. 8 coal was also studied. The results show that neither of them has much effect on pyritic sulfur rejection or on ash rejection.

Pyrite Depressants

A number of organic reagents having different functional groups were tested to determine their pyrite-depressing effect. The results show that glycerol monothioglycolate, mercaptoacetic acid, 2-mercaptoethanesulfonic acid, aminoacetic acid, aminoethanesulfonic acid and sulfosalicylic acid improve the separation efficiency when added to the mill in the grinding stage. For instance, grinding Pittsburgh No. 8 coal with 2-mercaptoethanesulfonic acid (1.0 lb/T) results in an improvement of pyrite rejection of about 10 percent at similar CMR with single-stage flotation, an improvement of about 20% with two-stage flotation.

However, a number of effective ore pyrite depressants, such as calcium cyanide, xanthated polyacrylic acid, xanthated sucrose, and xanthated citric acid, were found to be ineffective depressants for pyrite in coal flotation. In order to reduce the hydrophobicity of pyrite, hydrogen peroxide (H_2O_2) was also tested as a selective oxidant for pyrite in coal flotation. The results show that the addition of hydrogen peroxide reduces the flotability of pyrite, but it also reduces the flotability of coal to the same extent. Thus the addition of hydrogen peroxide to coal flotation has no effect on pyritic sulfur rejection and ash rejection.

Because Illinois No. 6 coal is relatively hydrophilic and almost no material floats without the addition of an oily collector, flotation of pyrite from this coal, known as reverse flotation, using xanthate and dithiophosphates as collector was tested in order to increase the separation efficiency. However, the results were not promising because the collector dosages required were high and substantial amounts of coal were also recovered in the froth.

Flotation Optimization and Circuits

The effect of frother conditioning time on separation efficiency and flotation kinetics of the base coals was studied. The results show that the separation efficiency does not vary with the frother conditioning time whereas the flotation rate constant decreases with the increase in the frother conditioning time (in the range 0.5 to 3 minutes). At optimized reagent dosages of dodecane and MIBC, the maximum separation efficiency of 100-, 200- and 325-mesh Upper Freeport PA coal is about 65 with a single-stage flotation. This indicates that grinding this coal finer than 100 mesh does not result in significant improvement of the separation efficiency, which is in accordance with the results obtained in the washability tests.

The effect of various flotation circuit configurations on the separation efficiency of the base coals was investigated. These circuits consisted of closed-circuit flotation, cleaner-scavenger circuits, and circuits with staged-reagent additions. In general, two-stage flotation (recleaning the concentrates) can improve the separation efficiency significantly over single-stage flotation. The the best results were obtained by refloating the middlings (the tailings from the cleaning stage) with new flotation feed (closed-circuit flotation).

The flotation column is generally considered to be more efficient than conventional flotation machines for fine coal cleaning. The separation efficiency in a single-stage column flotation was found to be the same as that obtained in two-stage flotation with the conventional mechanical flotation cell. By pre-cleaning 28-mesh coal with a shaking table and floating the table concentrates after they had been reground to minus 200 mesh, the separation efficiency can be improved significantly over that obtained by the standard flotation of 200-mesh coal. This separation efficiency was found to be equivalent or better than that obtained by two-stage grinding along with two-stage flotation.

Under the best flotation conditions, the maximum separation efficiency obtained for 200-mesh Upper Freeport PA coal is 75 (combustible material recovery of 89% and pyritic sulfur rejection of 86%). This value, reported by the University of Utah, was achieved by floating wet-ground Upper Freeport PA coal at natural pH in a closed circuit for five cycles with 4.0 lb/T butanol added to the grinding stage. This efficiency index is significantly higher than that obtained in the standard flotation test ($El_H = 52$) and in the release analysis using dodecane and

MIBC ($EI_H = 64$). For 200-mesh Pittsburgh No. 8 coal, the maximum efficiency index is 71 (CMR = 91% and PSR = 80%), reported by Columbia University. This result was obtained by two-stage flotation of 200-mesh Pittsburgh No. 8 coal, which had been ground wet at pH 8 (with sodium hydroxide added to the grinding stage). The flotation test was conducted using 1.95 lb/T of MIBC at the same pH as in the grinding stage. The maximum efficiency index for Pittsburgh No. 8 is also significantly higher than that obtained with the standard flotation procedure ($EI_H = 50$) and in the release analysis using dodecane and MIBC ($EI_H = 56$). For 200-mesh Illinois No. 6 coal, the maximum efficiency index that was obtained is 56 (CMR = 78% and PSR = 78%), which was achieved by two-stage flotation of a coal sample that had been wet ground with 1.0 lb/T of mercaptoacetic acid at natural pH (about 4). The reagents used in these tests were the standard dosages of dodecane and MIBC. Although this value is low, it is still significantly higher than that obtained in the standard flotation test ($EI_H = 36$) and in the release analysis for the sample that had undergone the same storage time ($EI_H = 42$). However, this value is lower than the value of EI_H obtained in release analysis for the coal samples with a shorter storage time ($EI_H = 65$) where less degradation of the coal samples had taken place during the storage. Illinois No. 6 coal is more susceptible to oxidation than Pittsburgh No. 8 and Upper Freeport PA coals. Because of the lower hydrophobicity, Illinois No. 6 coal is also the most difficult coal to clean among the three base coals.

Coal Weathering

Coal weathering studies were conducted on three size fractions, plus 1-inch, 1-inch x 1/4-inch, and 1/4-inch x 0. Each size fraction was screened in the field and stored in three different modes: i) inert, in drums under an argon atmosphere; ii) covered, in drums without lids but with plywood boards on top; and iii) open, in a stockpile completely exposed to the atmosphere. A total of thirteen weathering increments was collected, the first six on a biweekly basis starting in November 1988, the next four on a monthly basis, and the final three increments on a bimonthly basis. Each increment was inerted with argon before being shipped to Berkeley and to the University of Pittsburgh for characterization studies.

Physical and chemical changes that might occur during the storage of coal were examined by characterizing the weathered coal samples using various techniques. This characterization included size distribution, proximate analysis, DRIFT spectroscopy, zeta potential, standard flotation, film flotation and induction time determinations. The proximate analysis showed that no appreciable decrease in the volatile matter of the weathered samples occurred over the 13-month weathering period. The particle size distribution of the minus 28-mesh fraction screened from the minus 1/4-inch sample stored in open mode indicated that for Illinois No. 6 coal, particles in the intermediate size fractions (such as 28 x 35 mesh) became more friable, contributing to an increase in the amount of material in the finer size fractions as the weathering time increased. Particle size analysis of the 1/4-inch x 28 mesh material indicated a progressive increase in the fines as weathering progressed. Interestingly, the size degradation that was observed for the Illinois No. 6 coal sample did not occur with either Pittsburgh No. 8 or Upper Freeport PA coal.

A comparison of the DRIFT spectra of the research and weathered samples for each of the three base coals showed some differences between them. Spectra for Illinois No. 6 weathered for short periods of time showed the presence of a peak corresponding to the carbonyl group (1655 cm^{-1}), indicating a certain degree of oxidation. No carbonyl peaks were found in the DRIFT spectra of the weathered samples of Pittsburgh No. 8, indicating that this coal is less susceptible to oxidation by weathering for short periods of time. No peaks associated with oxidation products were obtained for Upper Freeport PA weathered samples.

Film flotation tests conducted on the weathered samples of the three base coals showed a slow drop in hydrophobicity initially, followed by a rapid decrease after five months of weathering. Weathering began in the winter, and only after the weather began to warm up in the spring did the Pittsburgh No. 8 and Upper Freeport coals begin to weather. Furthermore, both film flotation and induction time measurements clearly indicated that the hydrophobicity of the weathered samples is a function of the storage environment. Specifically, the hydrophobicity of coal stored in the inert mode is greater than that for coal stored in the covered and open modes for similar periods of weathering. The bubble/particle contact time for Illinois No. 6 stored in the open mode increased from 60 milliseconds for Increment 1 to 1750 milliseconds for

Increment 13. The same trend was observed with the other two coals, but, the increase in induction time was not as dramatic. Wettability tests done with the Hallimond tube (up to Increment 4) indicated that the hydrophobicity of the weathered samples drops only marginally and that the yields were less than that of the research samples, probably due to the lower ash content in the research samples.

The standard flotation test procedure developed for this program for the 28-mesh ground coal was also used to float the minus 28-mesh weathered material screened from minus 1/4-inch fraction. The results showed that there is a decrease in the flotation yield as compared to the freshly ground research samples. This drop was higher for the open mode as compared to the covered and inert modes for the same weathering time. The rate of decrease in the yield is also coal-dependent, with Illinois No. 6 coal yield decreasing very rapidly initially as compared to the other two coals before levelling off at a yield of 28%. The recovery of Pittsburgh No. 8 and Upper Freeport PA coals decreased little during the first 5 months of weathering and then decreased rapidly for coals weathered through the summer months. This behavior seems to correlate quite well with the temperature in the weathering sites. When the weathering temperature rises, the oxidation of the coals is more significant. After 9 months of weathering, the yield for these two coals increased slightly, the reason for which is not clear at this point. Induction time measurements also show an increase in the apparent hydrophobicity of Upper Freeport PA and Pittsburgh No. 8 coals that had been weathered for longer than 9 months. Flotation pulp pH in the standard flotation of the minus 28-mesh weathered coals was also decreased as the weathering time increased.

Grinding of the 1/4-inch x 28-mesh material to minus 28 mesh before flotation restored the yield equivalent to that of the research samples, indicating that the effect of weathering is mainly limited to the external surfaces only. Therefore, coal should be stored as coarse as possible in order to minimize the detrimental effect of oxidation on the surface hydrophobicity of coal. In addition, the largest decreases in the hydrophobicity of the coals occurred at the onset of spring and summer temperatures, suggesting that if coal must be stored, significant changes can be expected to occur and these will be a function of ambient temperature.

Degradation of Stored Coal Samples

Although the research samples were inerted carefully under argon atmosphere, degradation of the stored coal samples has been observed during the course of this research. The separation efficiency under the same flotation conditions decreases for coal samples that had been stored under the "inert" environment for a long time. This suggests that in any research effort extending over a period of time, it is necessary to regularly collect newly mined samples that are as close to the original location in the region as possible. Without this regeneration of coal samples, the results obtained from the latter part of any research project must be viewed with caution.

TABLE OF CONTENTS

	<u>Page</u>
EXECUTIVE SUMMARY	ES-1
1.0 INTRODUCTION	1-1
1.1 Project Team	1-1
1.2 Project Goals	1-3
1.3 Scope of this Document	1-4
2.0 COAL PROCUREMENT	2-1
2.1 Overview and Scope	2-1
2.2 Coal Sample Procurement and Distribution	2-3
2.3 Primary and Secondary Crushing	2-4
3.0 BULK CHARACTERIZATION STUDIES	3-1
3.1 Chemical (compositional) analysis	3-1
3.2 Mineralogical Analysis	3-4
3.3 Photomicrographic Studies of Mineral Matter Association	3-6
3.3.1 Sem/EDXRF Study	3-6
3.3.2 Optical Image Analysis	3-16
3.4 Petrographic Characterization	3-17
3.5 Solubility Behavior of the Base Coals	3-18
3.6 References	3-22
4.0 COAL WASHABILITY STUDIES	4-1
4.1 Sample Preparation and Sink-float Test Procedure	4-2
4.2 Washability Results and Data Analysis	4-4
4.2.1 Illinois No. 6 Sample Washability Results	4-6
4.2.2 Pittsburgh No. 8 Sample Washability Results	4-9
4.2.3 Upper Freeport PA Sample Washability Results	4-12

4.3	Washability QA/QC	4-15
4.3.1	Washability Data Evaluation Criteria	4-16
4.3.2	Data Analysis for Illinois No. 6 Coal	4-17
4.3.3	Data Analysis for Pittsburgh No. 8 Coal	4-19
4.3.4	Data Analysis for Upper Freeport PA Coal	4-21
4.3.5	QA/QC on the Analytical Work	4-22
4.4	Fine Coal Washability Studies	4-23
4.4.1	Sink-float Test Procedure and Data Compilation	4-25
4.4.2	Illinois No. 6 Sample Washability Results	4-26
4.4.3	Pittsburgh No. 8 Sample Washability Results	4-32
4.4.4	Upper Freeport PA Sample Washability Results	4-37
4.4.5	Fine Coal Washability Summary	4-45
4.5	Washability Tests on Additional Coal Samples	4-46
4.5.1	Washability of Upper Freeport WV Coal	4-46
4.5.2	Washability of Kentucky No. 9 coal	4-51
4.5.3	Washability of Wyodak Coal	4-54
4.6	References	4-54
5.0	SURFACE CHARACTERIZATION STUDIES	5-1
5.1	Surface Area Determination	5-1
5.2	Wettability Behavior Studies	5-2
5.2.1	Film Flotation	5-2
5.2.2	Contact Angle Measurements	5-3
5.2.3	Induction Time Measurements	5-6
5.3	Surface Charge Characterization	5-11
5.4	Analysis of Surface Elemental Composition by ESCA	5-16
5.5	Analysis of Surface Functional Groups by DRIFT	5-18
5.6	References	5-23
6.0	DEVELOPMENT OF STANDARD GRINDING TEST	6-1
6.1	Development of Standard Grinding Test	6-1
6.2	Selection of a Laboratory Grinding Mill	6-3
6.3	Effect of Rod Charge on Grinding Time and Particle Size Distribution ...	6-5
6.4	Standard Grinding Procedure	6-12

6.5	Relative grindabilities of base coal samples	6-14
6.6	Cleanability of Standard Ground Coals	6-15
6.7	QA/QC Grinding Tests	6-17
6.8	References	6-18
7.0	DEVELOPMENT OF THE STANDARD FLOTATION TEST	7-1
7.1	Standard Flotation Test Procedure	7-1
7.2	QA/QC for Standard Flotation Tests and Coal Analyses	7-9
7.3	Efficiency Index Comparison	7-12
7.4	Release Analysis	7-18
	7.4.1 Illinois No. 6	7-18
	7.4.2 Pittsburgh No. 8	7-20
	7.4.3 Upper Freeport PA	7-23
	7.4.4 Summary	7-26
7.5	Correlation Between Combustible Material Recovery (CMR) and Btu Recovery	7-26
7.6	Relation Between Total Sulfur and Pyritic Sulfur	7-28
	7.6.1 Testing the Proposed Procedure with Flotation Samples	7-30
	7.6.2 Testing the Proposed Procedure with Washability Samples	7-34
	7.6.3 Summary and Conclusions	7-38
7.7	References	7-39
8.0	EFFECT OF GRINDING ENVIRONMENT	8-1
8.1	Grinding and Flotation under Air and Inert Gases	8-1
8.2	Wet and Dry Grinding Methods	8-2
8.3	Grinding with Ceramic Mill	8-9
8.4	Grinding with Collector and Frother	8-11
	8.4.1 Effect of Grinding with Collector and Frother on Flotation Yield and Separation Efficiency	8-12
	8.4.2 Effect of Grinding with Collector on Flotation Kinetics	8-18
8.5	References	8-30

9.0	SURFACE MODIFICATION	9-1
9.1	Polymerizable Monomers	9-2
9.2	Effect of Anionic Reagents	9-8
9.2.1	Aerosol OT	9-9
9.2.2	Alkylated pyridines	9-13
9.2.3	Summary	9-13
9.3	Flotation with Non-ionic Surfactants	9-16
9.3.1	GH Series of Reagents	9-16
9.3.2	Alkylated thiophene	9-21
9.3.3	Dodecane	9-23
9.4	Reactive non-Polymerizable Reagents	9-26
9.4.1	Alcohol Homologues	9-26
9.4.2	Butyl benzaldehyde	9-33
9.4.3	Glyoxal	9-35
9.4.4	Other Reagents	9-36
9.5	pH Effect	9-36
9.5.1	Pittsburgh No. 8 coal	9-38
9.5.2	Illinois No. 6 coal	9-54
9.5.3	Upper Freeport PA coal	9-57
9.5.4	Summary	9-58
9.5.5	Recommendation	9-58
9.6	Dispersants	9-59
9.7	References	9-62
10.0	PYRITE DEPRESSANTS	10-1
10.1	Organic Pyrite Depressants	10-1
10.2	Calcium Cyanide	10-7
10.3	Polymeric Xanthates	10-12
10.4	Effect of Hydrogen Peroxide	10-14
10.5	Reverse Flotation	10-17
10.6	References	10-22

11.0	KINETICS OF COAL FLOTATION	11-1
11.1	Flotation Kinetics Model Selection	11-2
11.2	Effect of Particle Size on the Flotation of 28-Mesh Ground Coal	11-6
11.3	Effect of Particle Size in 100-mesh Ground Coal	11-12
11.4	Effect of Particle Size in 200-mesh Ground Coal	11-20
11.5	Effect of Particle Topsize on Flotation Kinetics	11-30
11.6	Flotation Kinetics of Coal at Long Flotation Times	11-37
11.7	References	11-40
12.0	FLOTATION OPTIMIZATION AND CIRCUITRY	12-1
12.1	Optimization of Dodecane and MIBC	12-1
12.1.1	Illinois No. 6	12-1
12.1.2	Upper Freeport PA	12-6
12.2	Effect of Frother Conditioning Time on Separation Efficiency and Flotation Kinetics.	12-10
12.3	Flotation Circuitry	12-12
12.3.1	Closed-Circuit Flotation	12-14
12.3.2	Cleaning and Scavenging	12-17
12.3.3	Staged Reagent Addition	12-18
12.4	Column Flotation	12-20
12.4.1	Comparison of Performances of Mechanically Agitated Cell and Flotation Column	12-21
12.4.2	Effect of bubble size and wash water	12-21
12.5	Combination of Tabling and Flotation	12-25
12.6	References	12-26
13.0	WEATHERING STUDIES OF THE BASE COALS	13-1
13.1	Weathering Site Preparation and Sampling	13-2
13.2	Periodic Collection of Weathering Samples	13-4
13.2.1	QA/QC Analysis of Coal Weathering Samples	13-6
13.3	Collection of Weather Data	13-8

13.4	Proximate Analysis of Weathered Samples	13-10
13.5	Size Consistency of the Weathered Samples	13-14
13.6	Assessing the Hydrophobicity of Weathered Samples	13-19
13.6.1	Film Flotation	13-20
13.6.2	Induction Time Measurements of Weathered Samples	13-25
13.7	Flotation Behavior of the Weathered Samples	13-27
13.8	Correlation of Wettability Criteria With Weathering	13-34
13.9	pH of Weathered Coal Samples	13-36
13.10	References	13-39
14.0	DEGRADATION OF STORED COAL SAMPLES	14-1
14.1	Tree Analysis of Base Coals	14-1
14.2	Effect of Storage Time on the Behavior of Illinois No. 6 Coal	14-9
14.3	References	14-11
15.0	SUMMARY AND CONCLUSIONS	15-1
15.1	Project Team and Objective	15-1
15.2	Coal Procurement	15-1
15.3	Bulk Characterization of the Coal Samples	15-2
15.4	Washability Studies	15-3
15.5	Wettability and Surface Characterization	15-4
15.6	Standard Grinding Tests	15-5
15.7	Standard Flotation Tests	15-6
15.8	Effect of Grinding Environment	15-8
15.9	Surface Modification	15-8
15.10	Pyrite Depressants	15-12
15.11	Coal Flotation Kinetics	15-12

15.12 Flotation Optimization and Circuits	15-14
15.13 Coal Weathering	15-15
15.14 Degradation of Stored Coal Samples	15-17
16.0 RECOMMENDATIONS FOR FUTURE WORK	16-1

LIST OF FIGURES

	<u>Page</u>
Figure 1.1 - Project organizational chart	1-2
Figure 2.1 - Drums of Upper Freeport PA coal being filled with 20-lb increments at the North Cambria Fuel Co. mine in Indiana County, Pennsylvania. ...	2-4
Figure 3.1 - Diffuse reflectance spectra of ash from the three base coals.	3-5
Figure 3.2 - SEM Micrograph of a 28 x 65-mesh sample of Pittsburgh No. 8 coal. ..	3-7
Figure 3.3 - SEM Micrograph of a 28 x 65-mesh sample of Illinois No. 6 coal.	3-7
Figure 3.4 - SEM Micrograph of a 28 x 65-mesh sample of Upper Freeport PA coal.	3-8
Figure 3.5 - SEM Micrograph of 65 x 100-mesh sample of Pittsburgh No. 8 coal. ...	3-8
Figure 3.6 - SEM Micrograph of a 65 x 100-mesh sample of Illinois No. 6 coal.	3-9
Figure 3.7 - SEM Micrograph of a 65 x 100-mesh sample of Upper Freeport PA coal.	3-9
Figure 3.8 - SEM Micrograph of a 100 x 200 mesh sample of Pittsburgh No. 8 coal.	3-11
Figure 3.9 - SEM Micrograph of a 100 x 200 mesh sample of Illinois No. 6 coal. ...	3-11
Figure 3.10 - SEM Micrograph of a 100 x 200-mesh sample of Upper Freeport PA coal.	3-12
Figure 3.11 - SEM Micrograph of a minus 200-mesh sample of Pittsburgh No. 8 coal.	3-12
Figure 3.12 - SEM Micrograph of a minus 200-mesh sample of Illinois No. 6 coal. ...	3-13
Figure 3.13 - SEM Micrograph of a minus 200-mesh sample of Upper Freeport PA coal.	3-13
Figure 3.14 - X-ray fluorescence spectra of iron sulfide particles in a minus 200-mesh sample of Pittsburgh No. 8 sample.	3-14
Figure 3.15 - X-ray fluorescence spectra of iron sulfide particles in a minus 200-mesh sample of Illinois No. 6 sample.	3-14
Figure 3.16 - X-ray fluorescence spectra of iron sulfide particles in a minus 200-mesh sample of Upper Freeport PA sample.	3-15

Figure 3.17 - SEM Micrograph of a Pittsburgh No. 8 coal particle with an epigenetic phase of pyrite in the form of veins.	3-15
Figure 4.1 - Coal washability sample splitting and processing method.	4-2
Figure 4.2 - Fraction of float material at 1.3 specific gravity for the three base coals comminuted to various designated sizes.	4-5
Figure 4.3 - Cumulative weight percent of float material (yield) at various top sizes of Illinois No. 6 as a function of specific gravity.	4-7
Figure 4.4 - Btu recovery of the cumulative float product as a function of the ash content for Illinois No. 6 coal at various top sizes.	4-7
Figure 4.5 - Btu recovery of the cumulative float product as a function of the pyritic sulfur content for Illinois No. 6 coal at various sizes.	4-8
Figure 4.6 - Btu recovery of the cumulative float product as a function of pyritic sulfur rejection for Illinois No. 6 coal at various sizes.	4-9
Figure 4.7 - Cumulative weight percent float (yield) at various specific gravities for Pittsburgh No. 8 coal at various top sizes.	4-10
Figure 4.8 - Btu recovery of the cumulative float product as a function of the ash content for Pittsburgh No. 8 coal.	4-11
Figure 4.9 - Btu recovery of the cumulative float product as a function of the pyritic sulfur content for Pittsburgh No. 8 coal.	4-11
Figure 4.10 - Btu recovery of the cumulative float product as a function of pyritic sulfur rejection for Pittsburgh No. 8 coal at various topsizes.	4-12
Figure 4.11 - Cumulative weight percent float (yield) at various specific gravities for Upper Freeport PA coal.	4-13
Figure 4.12 - Btu recovery of the cumulative float product as a function of the ash content for Upper Freeport PA coal.	4-14
Figure 4.13 - Btu recovery of the cumulative float product as a function of the pyritic sulfur content for Upper Freeport PA coal.	4-14
Figure 4.14 - Btu recovery of the cumulative float product as a function of pyritic sulfur rejection for Upper Freeport PA coal.	4-15
Figure 4.15 - Cumulative weight percent float at various specific gravities for Illinois No. 6 coal.	4-28
Figure 4.16 - Btu recovery vs. ash percent for Illinois No. 6 coal.	4-28

Figure 4.17 -	Calorific value of the cumulative float product as a function of the ash content for Illinois No. 6 coal.	4-29
Figure 4.18 -	Btu recovery of the cumulative float product as a function of pyritic sulfur rejection for Illinois No. 6 coal.	4-30
Figure 4.19 -	Particle size distribution for Illinois No. 6 coal washability test samples ground to 100, 200 and 400 mesh top size fraction.	4-33
Figure 4.20 -	Cumulative weight percent float at various specific gravities for Pittsburgh No. 8 coal.	4-33
Figure 4.21 -	Btu recovery of the cumulative float product as a function of the ash content for Pittsburgh No. 8 coal.	4-34
Figure 4.22 -	Btu recovery of the cumulative float product as a function of pyritic sulfur rejection for Pittsburgh No. 8 coal.	4-35
Figure 4.23 -	Particle size distribution for Pittsburgh No. 8 coal washability test samples ground to 100, 200 and 400 mesh top size fraction.	4-38
Figure 4.24 -	Cumulative weight percent float at various specific gravities for Upper Freeport PA coal.	4-39
Figure 4.25 -	Btu recovery of the cumulative float product as a function of the ash content for Upper Freeport PA coal.	4-39
Figure 4.26 -	Calorific value of the cumulative float product as a function of the ash content for Upper Freeport PA washability samples comminuted to various top sizes.	4-40
Figure 4.27 -	Btu recovery of the cumulative float product as a function of pyritic sulfur rejection for Upper Freeport PA coal.	4-42
Figure 4.28 -	Particle size distribution for Upper Freeport PA coal washability test samples ground to 100, 200 and 400-mesh top size fraction.	4-45
Figure 4.29 -	Cumulative weight percent (yield) float at various specific gravities for Upper Freeport WV coal.	4-47
Figure 4.30 -	Btu recovery of the cumulative float product as a function of the ash content for Upper Freeport WV coal.	4-48
Figure 4.31 -	Btu recovery of the cumulative float product as a function of the total sulfur content for Upper Freeport WV coal.	4-49
Figure 4.32 -	Btu recovery of the cumulative float product as a function of the pyritic sulfur content for Upper Freeport WV coal.	4-50

Figure 4.33 - Btu recovery of the cumulative float product as a function of pyritic sulfur rejection of Upper Freeport WV coal.	4-50
Figure 4.34 - Btu recovery plotted as a function of ash for various topsizes of Kentucky No. 9 coal.	4-52
Figure 4.35 - Btu recovery plotted as a function of total sulfur for various topsizes of Kentucky No. 9 coal.	4-52
Figure 4.36 - Btu recovery plotted as a function of cumulative pyritic sulfur for various topsizes of Kentucky No. 9 coal.	4-53
Figure 4.37 - Btu recovery plotted as a function of pyritic sulfur rejection for various top sizes of Kentucky No. 9 coal.	4-53
Figure 4.38 - Cumulative weight percent yield (float) at various specific gravities for the Wyodak seam coal from Campbell County, Wyoming.	4-54
Figure 5.1 - Film flotation partition curves for the three base coals.	5-3
Figure 5.2 - Induction time curve indicating the percentage of fruitful contacts as a function of contact time in microseconds for a 100 x 150 mesh sample of Pittsburgh No. 8 coal.	5-8
Figure 5.3 - Induction time curve indicating the percentage of fruitful contacts as a function of contact time in microseconds for 100 x 150 mesh samples of the three base coals.	5-8
Figure 5.4 - Induction time curves indicating the percentage of fruitful contacts as a function of contact time for Illinois No. 6 coal both undeslimed and deslimed fresh ground samples.	5-10
Figure 5.5 - Induction time curves indicating the percentage of fruitful contacts as a function of contact time for Pittsburgh No. 8 coal both undeslimed and deslimed fresh ground samples.	5-10
Figure 5.6 - Induction time curves indicating the percentage of fruitful contacts as a function of contact time for Upper Freeport PA coal both undeslimed and deslimed fresh ground samples.	5-11
Figure 5.7 - Zeta Potential versus pH curve for Illinois No. 6 coal	5-13
Figure 5.8 - Zeta Potential versus pH Curve for Pittsburgh No. 8 coal.	5-14
Figure 5.9 - Zeta Potential versus pH Curve for Upper Freeport PA coal.	5-14
Figure 5.10 - Diffuse reflectance spectra of the three base coals.	5-19
Figure 5.11 - Comparison of intensities of aliphatic CH stretching and bending peaks in the three base coals.	5-19

Figure 5.12 - Comparison of intensities of aromatic C=C peak of coal and Si-O peak of clay in the three base coals.	5-20
Figure 5.13 - Effect of heating time on the intensity of C=O stretching peak of Pittsburgh No. 8 coal.	5-21
Figure 5.14 - Effect of heating time on the intensity of C=C stretching peak (negative peak signifying reduction) from Pittsburgh No. 8 coal.	5-22
Figure 6.1 - Comparison of the size distribution of the products obtained by grinding Illinois No. 6 coal in ball and rod mills for four minutes.	6-4
Figure 6.2 - Particle size distribution of Illinois No. 6 coal ground in the rod mill for different grind times.	6-5
Figure 6.3 - Particle size distribution of Pittsburgh No. 8 coal ground in the rod mill for different grind times.	6-7
Figure 6.4 - Particle size distribution of Upper Freeport PA coal ground in the rod mill for different grind times.	6-7
Figure 6.5 - Effect of the number of rods on the particle size distribution of the ground product of Illinois No. 6 coal conforming to 95 percent passing through 28-mesh.	6-8
Figure 6.6 - Effect of the number of rods on the production of minus 200-mesh Illinois No. 6 coal for a grind time of 8 minutes.	6-9
Figure 6.7 - Effect of the number of rods on 28-mesh material produced during the dry grinding of Illinois No. 6 coal as a function of grinding time.	6-10
Figure 6.8 - Effect of the number of rods on 200-mesh material produced during the dry grinding of Illinois No. 6 coal as a function of grinding time.	6-10
Figure 6.9 - Effect of the number of rods on 200-mesh material produced during the dry grinding of Upper Freeport PA coal as a function of grinding time.	6-11
Figure 6.10 - Effect of the number of rods on 200-mesh material produced during the wet grinding of Upper Freeport PA coal as a function of grinding time.	6-11
Figure 6.11 - Size distributions of Illinois No. 6 coal prepared for washability testing and 200-mesh wet and dry-ground products prepared by the standard grinding procedure.	6-14
Figure 6.12 - Measured and SEM-AIA predicted washability results for 200-mesh Illinois No. 6 coal.	6-16

Figure 6.13 - Measured and SEM-AIA predicted washability results for 200-mesh Pittsburgh No. 8 coal.	6-16
Figure 6.14 - Measured and SEM-AIA predicted washability results for 200-mesh Upper Freeport PA coal.	6-17
Figure 7.1 - Two-paddle flotation cell used in The Standard Flotation Test.	7-3
Figure 7.2 - Standard flotation apparatus showing the flotation cell and Denver Laboratory Model Flotation Machine.	7-3
Figure 7.3 - Effect of collector and frother dosage on the flotation yield of 200-mesh wet-ground Pittsburgh No. 8 coal.	7-6
Figure 7.4 - Flotation yields of the three base coals as a function of flotation time using the standard collector and frother dosages tabulated in Table 7.1.	7-8
Figure 7.5 - Comparison of the Hancock and DOE efficiency indexes as a function of pyritic sulfur rejection at a constant CMR of 60 percent.	7-15
Figure 7.6 - Comparison of the Hancock and DOE efficiency indexes as a function of yield at a constant pyritic sulfur rejection of 80 percent.	7-16
Figure 7.7 - Comparison of the pyritic sulfur rejection as a function of yield at constant efficiency for Hancock and DOE indexes.	7-16
Figure 7.8 - Release analysis flotation scheme for Illinois No. 6 coal.	7-19
Figure 7.9 - Release analysis, washability data, and SEM-AIA data showing CMR (or BTU) recovery as a function of pyritic sulfur rejection for Illinois No. 6 coal.	7-19
Figure 7.10 - Ash rejection as a function of (100-Yield) for Illinois No. 6 coal for the kinetic flotation tests, release analysis and washability studies.	7-21
Figure 7.11 - Pyritic sulfur rejection as a function of (100-Yield) for Illinois No. 6 for the kinetic flotation tests, release analysis and washability studies.	7-21
Figure 7.12 - Release analysis flotation scheme for Pittsburgh No. 8 coal.	7-22
Figure 7.13 - Release analysis, washability data, and SEM-AIA data showing CMR (or Btu) recovery as a function of pyritic sulfur rejection for Pittsburgh No. 8 coal.	7-22
Figure 7.14 - Ash rejection as a function of (100-Yield) for Pittsburgh No. 8 coal for the one-step flotation tests, release analysis and washability studies. ...	7-24

Figure 7.15 - Pyritic sulfur rejection as a function of (100-Yield) for Pittsburgh No. 8 coal for the one-step flotation tests, release analysis and washability studies.	7-24
Figure 7.16 - Release analysis flotation scheme for Upper Freeport PA coal.	7-25
Figure 7.17 - Release analysis, washability data, and SEM-AIA data showing CMR (or BTU) recovery as a function of pyritic sulfur rejection for Upper Freeport PA coal.	7-25
Figure 7.18 - Relation between Combustible Material Recovery and Btu Recovery for Illinois No. 6 coal.	7-27
Figure 7.19 - Relation between Combustible Material Recovery and Btu Recovery for Pittsburgh No. 8 coal.	7-27
Figure 7.20 - Relation between Combustible Material Recovery and Btu Recovery for Upper Freeport PA coal.	7-28
Figure 7.21 - Correlation between the pyritic sulfur content determined experimentally using the ASTM Procedure and the value calculated using Equation 3 for flotation samples of Illinois No. 6 coal.	7-32
Figure 7.22 - Correlation between the pyritic sulfur content determined experimentally using the ASTM Procedure and the value calculated using Equation 4 for flotation samples of Pittsburgh No. 8 coal.	7-32
Figure 7.23 - Correlation between the pyritic sulfur content determined experimentally using the ASTM Procedure and the value calculated using Equation 5 for flotation samples of Upper Freeport PA coal.	7-33
Figure 7.24 - Correlation between the pyritic sulfur content determined experimentally using the ASTM Procedure and the value calculated using Equation 6 for washability samples of Pittsburgh No. 8 coal.	7-35
Figure 8.1 - Effect of the grinding mode on the separation of non-pyritic minerals from Pittsburgh No. 8 coal and a comparison of washability and flotation selectivity curves for 28 mesh grind.	8-3
Figure 8.2 - Effect of the grinding mode on the separation of non-pyritic minerals from Pittsburgh No. 8 coal and comparison of washability and flotation selectivity curves for 200-mesh grind.	8-5
Figure 8.3 - Comparison of flotation selectivity and washability curves of the 28 mesh and 200 mesh grinds of Pittsburgh No. 8 coal.	8-6
Figure 8.4 - Effect of grinding mode on the separation of pyrite from Pittsburgh No. 8 coal and a comparison of washability and flotation selectivity curves for the 28-mesh grind.	8-8

Figure 8.5	- Effect of grinding mode on the separation of pyrite from Pittsburgh No. 8 coal and a comparison of the washability and flotation selectivity curves for the 200-mesh grind.	8-8
Figure 8.6	- Particle size distribution for Illinois No. 6 research sample grind under standard dry and wet conditions and in a ceramic and steel ball mill. ...	8-11
Figure 8.7	- Effect of adding collector to the grinding mill on the yield, ash and total sulfur content of the concentrate for minus 28-mesh wet-ground Illinois No. 6 coal.	8-12
Figure 8.8	- Effect of adding part of the collector to the grinding mill and the balance to the flotation cell on the yield, ash and total sulfur content of the concentrate of minus 28-mesh wet-ground Illinois No. 6 coal.	8-14
Figure 8.9	- Effect of adding collector to the grinding mill on the yield, ash and total sulfur content of the concentrate of minus 200-mesh wet-ground Illinois No. 6 coal.	8-14
Figure 8.10	- Effect of adding part of the collector to the grinding mill and the balance to the flotation cell on the yield and ash content of the concentrate of minus 200-mesh wet-ground of Illinois No. 6 coal.	8-15
Figure 8.11	- Effect of adding frother (MIBC) to the grinding mill and in the flotation cell on the yield of the concentrate obtained using Illinois No. 6 coal. .	8-15
Figure 8.12	- Flotation yield of 200-mesh Illinois No. 6 coal obtained by adding 5.8 lb/T of dodecane to the grinding mill or flotation cell.	8-20
Figure 8.13	- Flotation yield of 200-mesh Pittsburgh No. 8 coal obtained by adding 1.9 lb/T of dodecane to the grinding mill or flotation cell.	8-20
Figure 8.14	- Flotation yield of 200-mesh Upper Freeport PA coal obtained by adding 0.5 lb/T of dodecane to the grinding mill or flotation cell.	8-21
Figure 8.15	- Flotation yield of 200-mesh Illinois No. 6 coal obtained by adding 0.7 lb/T of dodecane to grinding mill or flotation cell.	8-23
Figure 8.16	- Flotation yield of 200-mesh Illinois No. 6 coal obtained by adding 2.9 lb/T of dodecane to grinding mill or flotation cell.	8-23
Figure 8.17	- Flotation yield of 200-mesh Pittsburgh No. 8 coal obtained by adding 0.7 lb/T of dodecane to grinding mill or flotation cell.	8-24
Figure 8.18	- Flotation yield of 200-mesh Pittsburgh No. 8 coal obtained by adding 2.9 lb/T of dodecane to grinding mill or flotation cell.	8-24
Figure 8.19	- Flotation yield of 200-mesh Upper Freeport PA coal obtained by adding 0.24 lb/T of dodecane to grinding mill or flotation cell.	8-25

Figure 8.20 - Flotation yield of 200-mesh Upper Freeport PA coal obtained by adding 2.9 lb/T of dodecane to grinding mill or flotation cell.	8-25
Figure 8.21 - The effect of aging after grinding on the flotation yield of 200-mesh Illinois No. 6 coal obtained by adding 2.9 lb/T of dodecane to grinding mill or flotation cell.	8-28
Figure 8.22 - The effect of aging after grinding on the flotation yield of 200-mesh Pittsburgh No. 8 coal obtained by adding 2.9 lb/T of dodecane to flotation cell.	8-28
Figure 8.23 - Ash and pyritic sulfur rejection as a function of combustible material recovery of 200-mesh Illinois No. 6 coal obtained by adding 2.9 lb/T of dodecane to grinding mill or flotation cell.	8-29
Figure 9.1 - Effect of Aerosol OT addition on the rejection of pyrite from wet ground minus 200-mesh Pittsburgh No. 8 coal.	9-10
Figure 9.2 - Effect of Aerosol OT addition on the rejection of non-pyritic minerals from wet ground minus 200-mesh Pittsburgh No. 8 coal.	9-10
Figure 9.3 - Effect of Aerosol OT addition on the rejection of pyrite from wet ground minus 200-mesh Illinois No. 6 coal.	9-11
Figure 9.4 - Effect of Aerosol OT addition on the rejection of non-pyritic minerals from wet ground minus 200-mesh Illinois No. 6 coal.	9-11
Figure 9.5 - Effect of Aerosol OT addition on the rejection of pyrite from wet ground minus 200-mesh Upper Freeport PA coal.	9-12
Figure 9.6 - Effect of Aerosol OT addition on the rejection of non-pyritic minerals from wet ground minus 200-mesh Upper Freeport PA coal.	9-12
Figure 9.7 - Effect of 4- tert. butylpyridine addition on the rejection of pyrite from wet-ground minus 200-mesh Pittsburgh No. 8 coal.	9-14
Figure 9.8 - Effect of 4- tert. butylpyridine addition on the rejection of non-pyritic minerals from wet ground minus 200 mesh Pittsburgh No. 8 coal.	9-14
Figure 9.9 - Effect of 4-(1- butylpentyl)pyridine addition on the rejection of pyrite from wet ground minus 200-mesh Pittsburgh No. 8 coal.	9-15
Figure 9.10 - Effect of 4-(1- butylpentyl)pyridine addition on the rejection of non-pyritic minerals from wet ground minus 200-mesh Pittsburgh No. 8 coal.	9-15
Figure 9.11 - Comparison of the collecting ability of the GH series of reagent with that of dodecane in the flotation of 200-mesh wet-ground Illinois No. 6 coal.	9-18

Figure 9.12 - Comparison of the collecting ability of GH series with that of dodecane in the flotation of 200-mesh wet-ground Pittsburgh No. 8 coal.	9-19
Figure 9.13 - Effect of 2,n-butylthiophene addition on the rejection of pyrite from wet ground minus 200 mesh Pittsburgh No. 8 coal.	9-22
Figure 9.14 - Effect of 2,n-butylthiophene addition on the rejection of non-pyritic minerals from wet ground minus 200 mesh Pittsburgh No. 8 coal.	9-22
Figure 9.15 - Effect of dodecane addition on the rejection of pyrite from wet ground minus 200-mesh Pittsburgh No. 8 coal - single stage flotation results. .	9-24
Figure 9.16 - Effect of dodecane addition on the rejection of pyrite from wet ground minus 200-mesh Pittsburgh No. 8 coal - two-stage flotation results. ...	9-24
Figure 9.17 - Effect of dodecane addition on the rejection of non-pyritic minerals from wet ground minus 200-mesh Pittsburgh No. 8 coal - single stage flotation results.	9-25
Figure 9.18 - Effect of dodecane addition on the rejection of non-pyritic minerals from wet ground minus 200 mesh Pittsburgh No. 8 coal - two-stage flotation results.	9-25
Figure 9.19 - Comparison of performances of butanol, hexanol and MIBC in terms of reagent consumption.	9-31
Figure 9.20 - Effect of frother type on pyrite rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal -- single-stage flotation.	9-31
Figure 9.21 - Effect of frother type on pyrite rejection from wet-ground minus 200 mesh Pittsburgh No. 8 coal -- two-stage flotation.	9-32
Figure 9.22 - Effect of frother type on non-pyritic minerals rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal -- single-stage flotation.	9-32
Figure 9.23 - Effect of frother type on non-pyritic minerals rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal -- two-stage flotation.	9-33
Figure 9.24 - Solution - species diagram of aqueous iron sulfide system.	9-37
Figure 9.25 - Comparison of effects of sodium hydroxide addition to the mill and to the cell on coal flotation recovery.	9-38
Figure 9.26 - Effect of mode of sodium hydroxide addition (mill or cell) on pH - time relationship.	9-40
Figure 9.27 - Effect of pH (adjusted by adding sodium hydroxide in mill) on dissolved species concentration in the supernatant of wet-ground minus 28 mesh Pittsburgh No. 8 coal.	9-41

Figure 9.28 - Effect of pH (adjusted by adding sodium hydroxide in cell) on dissolved species concentration in the supernatant of wet-ground minus 200 mesh Pittsburgh No. 8 coal.	9-41
Figure 9.29 - Effect of ferrous chloride addition on washed coal flotation recovery. .	9-43
Figure 9.30 - Zeta potential behaviors of coal samples prepared by grinding coal in mill and in cell, and metal-hydroxides precipitated from coal supernatant.	9-44
Figure 9.31 - Effect of sodium hydroxide addition in cell on pyrite rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal.	9-45
Figure 9.32 - Effect of sodium hydroxide addition in mill on pyrite rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal.	9-45
Figure 9.33 - Comparison of effects of sodium hydroxide additions in mill and cell on pyrite rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal.	9-46
Figure 9.34 - Zeta potential behaviors of coal and pyrite samples prepared by grinding the feeds in mill, and metal-hydroxides precipitated from coal supernatant.	9-46
Figure 9.35 - Solution - species diagram of aqueous iron sulfide system: location of experimental data.	9-47
Figure 9.36 - Effect of sodium hydroxide addition in cell on the rejection of non-pyritic minerals from wet ground minus 200 mesh Pittsburgh No. 8 coal.	9-49
Figure 9.37 - Effect of sodium hydroxide addition in mill on the rejection of non-pyritic minerals from wet ground minus 200 mesh Pittsburgh No. 8 coal.	9-49
Figure 9.38 - Effect of frother dosage on coal flotation recovery under precipitation (sodium hydroxide in cell) and non-precipitation conditions (sodium hydroxide in mill).	9-51
Figure 9.39 - Effect of frother dosage on total water flow out of cell.	9-51
Figure 9.40 - Comparison of effects of sodium and calcium hydroxides in mill on pyrite rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal.	9-52
Figure 9.41 - Comparison of effects of sodium and calcium hydroxides in mill on non-pyritic minerals rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal.	9-52
Figure 9.42 - Effect of frother dosage on coal flotation recovery: comparison of frother consumptions in the presence of sodium hydroxide and calcium hydroxide.	9-53

Figure 9.43 - Effect of calcium hydroxide addition in cell on pyrite rejection from dry ground minus 200 mesh Pittsburgh No. 8 coal.	9-53
Figure 9.44 - Effect of calcium hydroxide addition in cell combustible material recovery and efficiency index for wet-ground minus 28 mesh Pittsburgh No. 8 coal.	9-55
Figure 9.45 - Effect of calcium hydroxide addition in cell on combustible material recovery and efficiency index for dry-ground minus 28 mesh Pittsburgh No. 8 coal.	9-55
Figure 9.46 - Effect of calcium hydroxide addition in cell on combustible material recovery and efficiency index for wet-ground minus 200 mesh Illinois No. 6 coal.	9-56
Figure 9.47 - Effect of calcium hydroxide addition in cell on combustible material recovery and efficiency index for wet-ground minus 200 mesh Upper Freeport PA coal.	9-57
Figure 9.48 - Effect of sodium silicate addition on pyrite rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal.	9-60
Figure 9.49 - Effect of sodium silicate addition on non-pyritic minerals rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal.	9-60
Figure 9.50 - Effect of sodium hexametaphosphate addition on pyrite rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal.	9-61
Figure 9.51 - Effect of sodium hexametaphosphate addition on non-pyritic minerals rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal.	9-61
Figure 10.1 - Flowsheet of the experiment to test GMTG as a pyrite depressant. ...	10-2
Figure 10.2 - Effect of grinding with GMTG on combustible material recovery and ash and pyritic sulfur rejection in the flotation of Pittsburgh No. 8 coal. ...	10-5
Figure 10.3 - Effect of grinding with GMTG on combustible material recovery and ash and pyritic sulfur rejection in the flotation of Illinois No. 6 coal.	10-6
Figure 10.4 - Effect of grinding with GMTG on combustible matter recovery and ash and pyritic sulfur rejection in the flotation of Upper Freeport PA coal.	10-7
Figure 10.5 - Effect of grinding with depressants on combustible material recovery and pyritic sulfur rejection in the flotation of Pittsburgh No. 8 coal.	10-8
Figure 10.6 - Effect of grinding with pyrite depressants on the kinetics of combustible material and pyritic sulfur recovery in the flotation of Pittsburgh No. 8 coal.	10-9

Figure 10.7 - Effect of xanthated polyacrylic acid and xanthated sucrose additions on pyrite rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal.	10-15
Figure 10.8 - Effect of xanthated polyacrylic acid and xanthated sucrose additions on non-pyritic minerals rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal.	10-15
Figure 10.9 - Effect of xanthated sucrose and xanthated citric acid additions on pyrite rejection from wet ground minus 325 mesh Pittsburgh No. 8 coal. ...	10-16
Figure 10.10 - Effect of xanthated sucrose and xanthated citric acid additions on non-pyritic minerals rejection from wet ground minus 325 mesh Pittsburgh No. 8 coal.	10-16
Figure 10.11 - Effect of hydrogen peroxide addition on pyrite rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal.	10-18
Figure 10.12 - Effect of hydrogen peroxide addition on non-pyritic minerals rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal.	10-18
Figure 11.1 - Effect of particle size on the flotation rate constant and the ultimate recovery of combustible materials of 28-mesh Illinois No. 6 coal.	11-9
Figure 11.2 - Effect of particle size on the flotation rate constant and the ultimate recovery of combustible materials of 28-mesh Pittsburgh No. 8 coal. ...	11-9
Figure 11.3 - Effect of particle size on the flotation rate constant and the ultimate recovery of combustible materials of 28-mesh Upper Freeport PA coal.	11-10
Figure 11.4 - Flotation recovery of combustible material, ash and pyritic sulfur from 100-mesh Illinois No. 6 coal as a function of flotation time along with the fitting curves of the first-order kinetic model with sine distribution of flotabilities.	11-16
Figure 11.5 - Flotation recovery of combustible material, ash and pyritic sulfur of 100-mesh Pittsburgh No. 8 coal as a function of flotation time along with the fitting curves of the first-order kinetic model with sine distribution of flotabilities.	11-16
Figure 11.6 - Flotation recovery of combustible material, ash and pyritic sulfur of 100-mesh Upper Freeport PA coal as a function of flotation time along with the fitting curves of the first-order kinetic with sine distribution of flotabilities.	11-17

Figure 11.7 - Combustible material recovery of plus 200, 200x400 and minus 400 mesh fractions of 100 mesh Illinois No. 6 coal as a function of flotation time along with the fitting curves of the first-order kinetic model with sine distribution of flotabilities.	11-17
Figure 11.8 - Combustible material recovery of various size fractions of 100-mesh Pittsburgh No. 8 coal as a function of flotation time along with the fitting curves of the first-order kinetic model with sine distribution of flotabilities.	11-18
Figure 11.9 - Combustible material recovery of various size fractions of 100 mesh Upper Freeport PA coal as a function of flotation time along with the fitting curves of the first-order kinetic model with sine distribution of flotabilities.	11-18
Figure 11.10 - Flotation recovery of combustible material, ash and pyritic sulfur of 200-mesh Illinois No. 6 coal as a function of flotation time along with the fitting curves of the first-order kinetic model with sine distribution of flotabilities.	11-23
Figure 11.11 - Flotation recovery of combustible materials, ash and pyritic sulfur of 200-mesh Pittsburgh No. 8 coal as a function of flotation time along with the fitting curves of the first-order kinetic model with sine distribution of flotabilities.	11-23
Figure 11.12 - Flotation recovery of combustible materials, ash and pyritic sulfur of 200-mesh Upper Freeport PA coal as a function of flotation time along with the fitting curves of the first-order kinetic model with sine distribution of flotabilities.	11-24
Figure 11.13 - Combustible material recovery of the plus and minus 400-mesh size fractions of 200-mesh Illinois No. 6 coal as a function of flotation time along with the fitting curves of the first-order kinetic model with sine distribution of flotabilities.	11-25
Figure 11.14 - Combustible material recovery of various size fractions of 200-mesh Pittsburgh No. 8 coal as a function of flotation time along with the fitting curves of the first-order kinetic model with sine distribution of flotabilities.	11-26
Figure 11.15 - Combustible material recovery of various size fractions of 200-mesh Upper Freeport PA coal as a function of flotation time along with the fitting curves of the first-order kinetic model with sine distribution of flotabilities.	11-26
Figure 11.16 - The difference between combustible material recovery of the plus and minus 400 mesh size fractions as a function of flotation time for the three base coals.	11-27

Figure 11.17 - The effect of reagent dosage on the difference between the combustible material recovery of the plus and minus 400 mesh size fractions as a function of flotation time for 200-mesh Illinois No. 6 coal.	11-28
Figure 11.18 - The effect of reagent dosage on the difference between the combustible material recovery of the plus and minus 400 mesh size fractions as a function of flotation time for 200-mesh Pittsburgh No. 8 coal.	11-29
Figure 11.19 - The effect of reagent dosage on the difference between combustible material recovery of the plus and minus 400 mesh size fractions as a function of flotation time for Upper Freeport PA coal.	11-29
Figure 11.20 - Combustible material recovery of 28, 100 and 200-mesh Illinois No. 6 coal as a function of flotation time along with the fitting curves of the first-order kinetic model with sine distribution of flotabilities.	11-36
Figure 11.21 - Combustible material recovery of 28, 100 and 200-mesh Pittsburgh No. 8 coal as a function of flotation time along with the fitting curves of the first-order kinetic model with sine distribution of flotabilities.	11-36
Figure 11.22 - Combustible material recovery of 28, 100 and 200-mesh Upper Freeport PA coal as a function of flotation time along with the fitting curves of the first-order kinetic model with sine distribution of flotabilities. ...	11-37
Figure 11.23 - Flotation results of minus 200-mesh Illinois No. 6 coal fitted with Lai's proportionality law of flotation kinetics.	11-41
Figure 11.24 - Flotation results of 200-mesh Pittsburgh No. 8 coal fitted with Lai's proportionality law of flotation kinetics.	11-42
Figure 11.25 - Flotation results of 200-mesh Upper Freeport PA coal fitted with Lai's proportionality law of flotation kinetics.	11-43
Figure 12.1 - Combustible material recovery as a function of time for Upper Freeport PA coal ground to minus 100-mesh, minus 200-mesh and minus 325-mesh.	12-8
Figure 12.2 - Separation efficiency as a function of time for Upper Freeport PA coal ground to minus 100-mesh, minus 200-mesh and minus 325-mesh.	12-8
Figure 12.3 - Effect of frother conditioning time on the flotation rate constant and ultimate recovery of combustible material of Illinois No. 6 coal.	12-11
Figure 12.4 - Effect of frother conditioning time on the flotation rate constant and ultimate recovery of combustible material of Pittsburgh No. 8 coal. ...	12-11
Figure 12.5 - Effect of frother conditioning time on the flotation rate constant and ultimate recovery of combustible material of Upper Freeport PA coal.	12-12
Figure 12.6 - Closed circuit flotation	12-15

Figure 12.7 - Cleaner-Scavenger Circuit	12-16
Figure 12.8 - Flow sheet for staged reagent addition.	12-19
Figure 12.9 - Comparison of performances of mechanically agitated cell and flotation column for the rejection of non-pyritic minerals from Pittsburgh No. 8 coal.	12-22
Figure 12.10 - Comparison of performances of mechanically agitated cell and flotation column for the rejection of pyrite from Pittsburgh No. 8 coal.	12-22
Figure 12.11 - Effect of bubble size on the rejection of pyrite from Pittsburgh No. 8 coal.	12-23
Figure 12.12 - Effect of bubble size on the rejection of non-pyritic minerals from Pittsburgh No. 8 coal.	12-23
Figure 12.13 - Effect of wash water on the rejection of pyrite from Pittsburgh No. 8 coal - tests with coarse frit.	12-24
Figure 12.14 - Effect of wash water on the rejection of pyrite from Pittsburgh No. 8 coal - tests with medium frit.	12-24
Figure 13.1 - Splitting scheme for the weathered coal samples.	13-3
Figure 13.2 - Coal weathering stockpiles of Upper Freeport PA coal at the Indiana County, Pennsylvania mine site.	13-4
Figure 13.3 - The maximum and minimum temperatures from October 1988 through November 1989 in Ohio County, West Virginia, located 15 miles from the Pittsburgh No. 8 coal weathering site in Belmont County, Ohio. ...	13-9
Figure 13.4 - The maximum and minimum temperatures of St. Louis, Missouri from October 1988 through November 1989.	13-9
Figure 13.5 - The maximum and minimum temperatures at the Upper Freeport PA coal weathering site in Indiana County, Pennsylvania from October 1988 through November 1989.	13-10
Figure 13.6 - Comparison of the particle size distribution of the rod milled research sample (95 percent passing 28 mesh) and Increment 7 of the weathered samples (inert, covered and open) for Illinois No. 6 coal.	13-15
Figure 13.7 - Effect of weathering time on the weight percent of material in different size intervals obtained by sieving the minus 28-mesh material of Illinois No. 6 coal weathered under open mode.	13-16
Figure 13.8 - Effect of weathering on the size distribution of the 1/4-inch x 28-mesh Illinois No. 6 weathered coal samples (inert and open samples of Increment 1, and open samples of Increments 7 and 9).	13-17

Figure 13.9	- Comparison of the particle size distribution of rod mill ground research sample (95 percent passing 28 mesh) and Increment 8 of the weathered samples (inert, covered and open) for Pittsburgh No. 8 coal.	13-17
Figure 13.10	- Effect of weathering on the size distribution of the 1/4-inch x 28-mesh weathered samples (inert and open samples of Increment 1, and open samples of Increments 8 and 9) for Pittsburgh No. 8 coal.	13-18
Figure 13.11	- Comparison of the particle size distribution of rod mill ground research sample (95 percent passing 28 mesh) and Increment 8 of the weathered samples (inert, covered and open) for Upper Freeport PA coal.	13-19
Figure 13.12	- Film flotation partition curves of as-received samples of Increment 3 for Illinois No. 6 weathered under inert, covered and open conditions. .	13-20
Figure 13.13	- Film flotation partition curves of deslimed samples of Increment 3 for Illinois No. 6 coal samples weathered under inert, covered and open conditions.	13-21
Figure 13.14	- Film flotation partition curves of deslimed Illinois No. 6 coal samples weathered under open mode for 0.5, 1.5 and 4 months.	13-22
Figure 13.15	- Film flotation partition curves of deslimed Pittsburgh No. 8 coal weathered under open mode for 0.5, 2 and 5 months.	13-23
Figure 13.16	- Film flotation partition curves of deslimed Upper Freeport PA coal weathered under open mode for 0.5, 2 and 5 months.	13-23
Figure 13.17	- Induction time curves indicating the percentage of fruitful contacts as a function of contact time in milliseconds for 100 x 150 M Illinois No. 6 weathered samples stored under open mode. Two different sample preparation procedures were used.	13-26
Figure 13.18	- Induction Time Curves indicating the percentage of fruitful contacts as a function of contact time for 100 x 150 M Illinois No. 6 weathered samples Incr. 1 stored under inert, covered and open modes.	13-26
Figure 13.19	- Induction Time Curve indicating the percentage of fruitful contacts as a function of contact time for 100 x 150 M Illinois No. 6 weathered sample Incr. 13 stored under open mode.	13-27
Figure 13.20	- Effect of weathering time on the flotation yield for Illinois No. 6 coal stored under inert and open modes.	13-29
Figure 13.21	- Effect of weathering time on the flotation yield for Pittsburgh No. 8 coal stored under inert and open modes.	13-31
Figure 13.22	- Effect of weathering time on the flotation yields for Upper Freeport PA coal stored under inert and open modes.	13-32

Figure 13.23 - Comparison of the effect of weathering on the combustible matter recovery for Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA coals weathered under open condition.	13-33
Figure 13.24 - Induction time, standard flotation and film flotation yields for Illinois No. 6 weathered samples stored under open mode as a function of weathering time.	13-34
Figure 13.25 - Induction time, standard flotation and film flotation yields for Pittsburgh No. 8 weathered samples stored under open mode as a function of weathering time.	13-36
Figure 13.26 - Induction time, standard flotation and film flotation yields for Upper Freeport PA weathered samples stored under open mode as a function of weathering time.	13-37
Figure 13.27 - Flotation pulp pH of the 28 mesh x 0 weathered samples as a function of weathering time.	13-38
Figure 14.1 - Tree analysis flotation scheme for Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA coals.	14-2
Figure 14.2 - Release analysis and tree analysis results showing ash rejection as a function of CMR for Illinois No. 6 coal.	14-3
Figure 14.3 - Release analysis and tree analysis showing pyritic sulfur rejection as a function of CMR for Illinois No. 6 coal.	14-3
Figure 14.4 - Release analysis and tree analysis results showing ash rejection as a function of CMR for Pittsburgh No. 8 coal.	14-5
Figure 14.5 - Release analysis and tree analysis results showing pyritic sulfur rejection as a function of CMR for Pittsburgh No. 8 coal.	14-5
Figure 14.6 - Release analysis and tree analysis results showing ash rejection as a function of CMR for Upper Freeport PA coal.	14-8
Figure 14.7 - Release analysis and tree analysis results showing pyritic sulfur rejection as a function of CMR for Upper Freeport PA coal.	14-8

LIST OF TABLES

	<u>Page</u>
Table 2.1 - List of coal seams sampled and corresponding mine locations.	2-2
Table 3.1 - Proximate analysis of the three base coals.	3-2
Table 3.2 - Analysis of sulfur-forms for the three base coal samples.	3-2
Table 3.3 - Ultimate analysis of the three base coals.	3-3
Table 3.4 - Infrared wavenumbers (cm^{-1}) for various minerals present in coal.	3-6
Table 3.5 - Pyrite association and distribution (in volume percent) in the three base coals comminuted to minus 200-mesh.	3-16
Table 3.6 - Maceral analysis of the three base coals.	3-18
Table 3.7 - Results of Pittsburgh No. 8, Illinois No. 6, and Upper Freeport PA leaching tests in distilled water at a solids concentration of 10% at 27°C.	3-20
Table 4.1 - Commercial laboratories used for washability testing of various coals. ...	4-1
Table 4.2 - Sink-float tests conducted on various size fractions for each coal.	4-3
Table 4.3 - Statistical analysis of composite values of ash, total and pyritic sulfur and calorific values for Illinois No. 6 sample.	4-17
Table 4.4 - Comparison of the float products at 1.3 and 1.6 specific gravity for the Illinois No. 6 sample.	4-18
Table 4.5 - Comparison of float product at 1.4 specific gravity for Illinois No. 6 sample.	4-19
Table 4.6 - Statistical analysis of the composite values of ash, total and pyritic sulfur and calorific values for Pittsburgh No. 8 sample.	4-20
Table 4.7 - Comparison of the float products at 1.3 and 1.6 specific gravity for Pittsburgh No. 8 sample.	4-20
Table 4.8 - Statistical analysis of composite values of ash, total and pyritic sulfur and calorific values for Upper Freeport PA.	4-21
Table 4.9 - Comparison of the float products at 1.3 and 1.6 specific gravity for the Upper Freeport PA sample.	4-22
Table 4.10 - Comparison of proximate and sulfur analyses* of base coals.	4-23

Table 4.11	- Comparison of the elemental analyses* of the base coals.	4-24
Table 4.12	- Analysis of sink-float fractions preselected for testing.	4-24
Table 4.13	- Comparison of ash, sulfur, and Btu values of the Illinois No. 6 coal samples used in the additional washability samples with those used for coarse coal washability.	4-31
Table 4.14	- Proximate, total sulfur and pyritic sulfur analyses for the additional fine washability test samples for Illinois No. 6 coal.	4-31
Table 4.15	- Comparison of float products at 1.3 and 1.6 specific gravity for Illinois No. 6 coal.	4-32
Table 4.16	- Comparison of ash, sulfur and Btu values of the Pittsburgh No. 8 coal samples used in the fine and coarse coal washability tests.	4-36
Table 4.17	- Proximate, total sulfur and pyritic sulfur analyses for the additional fine washability test samples for Pittsburgh No. 8 coal.	4-37
Table 4.18	- Comparison of floats at 1.3 and 1.6 specific gravity for Pittsburgh No. 8 seam samples tested.	4-37
Table 4.19	- Comparison of ash, sulfur, and Btu values of the Upper Freeport PA coal samples used in the fine and coarse washability testing.	4-43
Table 4.20	- Proximate, total sulfur and pyritic sulfur analyses for the additional fine washability test samples for Upper Freeport PA coal.	4-44
Table 4.21	- Comparison of floats at 1.3 and 1.6 specific gravity for Upper Freeport PA coal samples tested.	4-44
Table 4.22	- Ash, total sulfur, pyritic sulfur content and calorific value of the additional coals.	4-46
Table 5.1	- Determination of the specific surface areas of the three base coals by CO ₂ adsorption at room temperature.	5-1
Table 5.2	- Air/water advancing contact angles (in degrees) measured on the three base coals by the sessile-drop method on surfaces prepared by different methods.	5-4
Table 5.3	- Air/water contact angles (in degrees) measured on the three base coals by the captive-bubble method on surfaces wet-polished in air.	5-5
Table 5.4	- Air/water contact angle measurements on the three base coals calculated from film flotation results and advancing contact angles measured by the sessile-drop and the captive-bubble methods on surfaces wet-polished in air.	5-5

Table 5.5	- Contact angle measurements of the three base coals as a function of the time after placing a water drop on a pellet surface (using the sessile-drop method).	5-6
Table 5.6	- Calculated difference between zeta potential values in triply distilled water and 2×10^{-3} M KNO_3 solution for the three base coals.	5-15
Table 6.1	- Number of rods tested and rod mix used for various grinding tests.	6-6
Table 6.2	- Measured struck volume for the various rod charges used.	6-9
Table 6.3	- Standard wet and dry grinding conditions for the three base coals.	6-12
Table 6.4	- Size distribution and ash analysis of Upper Freeport PA 28-mesh standard ground material.	6-13
Table 6.5	- Size distribution and ash analysis of Upper Freeport PA 200-mesh standard ground material.	6-13
Table 6.6	- Relative grindabilities* of the three base coals.	6-15
Table 6.7	- Results of QA/QC tests of the standard grinding procedure showing the percent passing the desired grind size for grinding conducted at various sites for the three base coals.	6-18
Table 7.1	- Standard flotation test conditions.	7-5
Table 7.2	- Results of standard flotation tests using Illinois No. 6 coal.	7-6
Table 7.3	- Results of the standard flotation tests using Pittsburgh No. 8 coal.	7-7
Table 7.4	- Results of standard flotation tests for Upper Freeport PA coal.	7-7
Table 7.5	- Reproducibility of the standard flotation tests (dodecane 5.75 lb/T, MIBC 1.16 lb/T, pH 6.6) of 200-mesh, wet-ground Illinois No. 6.	7-10
Table 7.6	- Reproducibility of the standard flotation tests (dodecane 1.92 lb/T, MIBC 0.30 lb/T, pH 3.8) of 200-mesh, wet-ground Pittsburgh No. 8 coal.	7-10
Table 7.7	- Reproducibility of standard flotation tests (dodecane 0.48 lb/T, MIBC 0.21 lb/T, pH 3.3) of 200-mesh, wet-ground Upper Freeport PA coal	7-10
Table 7.8	- Comparison of split samples from the 200-mesh wet grind of the three base coals.	7-11
Table 7.9	- Comparison of ash, total-sulfur, and pyritic-sulfur analyses conducted at different universities.	7-12

Table 7.10	- Comparison of CMR, ash rejection, pyritic-sulfur rejection, and efficiency index of three base coal calculated using different methods based on the results obtained by the different research groups	7-13
Table 7.11	- Comparison of the pyritic sulfur content determined experimentally using the ASTM procedure and calculated using Eq. 3 for flotation samples of 200-mesh Illinois No. 6 coal.	7-31
Table 7.12	- Comparison of the pyritic sulfur content determined experimentally using the ASTM Procedure and calculated using Eq. 4 for flotation samples of 200-mesh Pittsburgh No. 8 coal.	7-33
Table 7.13	- Comparison of the pyritic sulfur content determined experimentally using the ASTM Procedure and calculated using Eq. 5 for Flotation samples of 200-mesh Upper Freeport PA coal.	7-34
Table 7.14	- Comparison of the pyritic sulfur content determined experimentally using the ASTM Procedure and calculated using Eq. 6 for washability samples of 200x28 mesh size fraction of Pittsburgh No. 8 coal (1.5 inch top size).	7-36
Table 7.15	- Comparison of the pyritic sulfur content determined experimentally using the ASTM Procedure and calculated using Eq. 6 for washability samples of 28 mesh x 1/8 inch size fraction of Pittsburgh No. 8 coal (1.5 inch top size).	7-36
Table 7.16	- Comparison of the pyritic sulfur content determined experimentally using the ASTM Procedure and calculated using Eq. 6 for washability samples of 1.5 x 1/8-inch size fraction of Pittsburgh No. 8 coal (1.5 inch top size).	7-37
Table 7.17	- Comparison of the pyritic sulfur content determined experimentally using the ASTM Procedure and calculated using Eqs. 4 and 6 for washability samples of 28 x 200 mesh size fraction of Pittsburgh No. 8 coal (1.5 inch top size).	7-37
Table 7.18	- Comparison of the pyritic sulfur content determined experimentally using the ASTM Procedure and calculated using both Eq. 4 and Eq. 6 for flotation samples of 100-mesh Pittsburgh No. 8 coal.	7-38
Table 8.1	- The effect of grinding atmosphere and flotation gas composition on the combustible material recovery of 200-mesh feed.	8-2
Table 8.2	- Particle size distribution of various grinding and washability samples of Pittsburgh No. 8 coal.	8-4
Table 8.3	- Effect of hydrogen peroxide addition on flotation performance of 200-mesh wet-ground Pittsburgh No. 8 coal.	8-7

Table 8.4	- Experimental procedure for step grinding tests using ceramic and steel ball mills.	8-10
Table 8.5	- Flotation results of different grinding products for Illinois No. 6 research samples.	8-13
Table 8.6	- The effect of collector and/or frother addition to mill during grinding on 28-mesh Illinois No. 6 coal.	8-16
Table 8.7	- The effect of collector and/or frother addition to the mill during wet grinding on the flotation of 200-mesh Illinois No. 6 coal.	8-17
Table 8.8	- The effect of collector and/or frother addition to the mill during wet grinding on 28-mesh Pittsburgh No. 8 coal.	8-17
Table 8.9	- The effect of collector and/or frother addition to the mill during wet grinding on 200-mesh Pittsburgh No. 8 coal.	8-17
Table 8.10	- The effect of collector and/or frother addition to the mill during wet grinding on 28-mesh Upper Freeport PA coal.	8-18
Table 8.11	- The effect of collector and/or frother addition to the mill during wet grinding on 200-mesh Upper Freeport PA coal.	8-18
Table 8.12	- Summary of the effect of collector addition on the ultimate recovery and flotation rate constant for the three base coals obtained by fitting the first-order kinetic model with sine distribution of flotabilities.	8-22
Table 9.1	- Summary of the effect of various polymerizable monomers and their saturated equivalents (4.8 lb/T except where noted) on the flotation efficiency index of the three base coals. Standard frother and collector dosages were used in all tests.	9-4
Table 9.2	- Effect of polymerizable monomer addition on the flotation of 200-mesh dry-ground Illinois No. 6 coal (4.8 lb/T of modifier used except where noted).	9-5
Table 9.3	- Effect of polymerizable monomer addition on the flotation of 200-mesh dry-ground Pittsburgh No. 8 coal (4.8 lb/T of modifier used for all tests).	9-5
Table 9.4	- Effect of polymerizable monomer addition on the flotation of 200-mesh dry-ground Upper Freeport PA coal (4.8 lb/T modifier used for all tests).	9-6
Table 9.5	- Effect of vinyl acetate (4.8 lb/T) with and without inhibitor on the flotation response of 200-mesh dry-ground Illinois No. 6 coal.	9-6
Table 9.6	- Effect of vinyl acetate (4.8 lb/T) with and without inhibitor on the flotation of 200-mesh dry-ground Pittsburgh No. 8 coal.	9-7

Table 9.7	- Effect of diisobutylene (4.8 lb/T) addition to the mill on the flotation response of 200-mesh wet and dry-ground Pittsburgh No. 8 coal.	9-7
Table 9.8	- Comparison of the effect of diisobutylene addition (4.8 lb/T) to the mill with that to the cell on the flotation response of 200-mesh wet-ground Pittsburgh No. 8 coal.	9-8
Table 9.9	- Contact angle measurement by the sessile drop method on flotation feed samples of Pittsburgh No. 8 and Upper Freeport PA coal treated with diisobutylene.	9-8
Table 9.10	- Effect of GH reagents on the flotation of 200-mesh wet-ground Illinois No. 6 coal.	9-17
Table 9.11	- Effect of GH reagents on the flotation of 200-mesh wet-ground Pittsburgh No. 8 coal.	9-17
Table 9.12	- The effect of GH0 and GHB on the flotation of 200-mesh wet-ground Illinois No. 6 coal.	9-20
Table 9.13	- The effect of dodecane, GH0 and GH4 reagents on the flotation of lab-oxidized 200-mesh wet-ground Pittsburgh No. 8 coal.	9-21
Table 9.14	- Effect of methanol addition to the grinding mill on the flotation response of 200-mesh wet-ground Upper Freeport PA coal.	9-26
Table 9.15	- Effect of methanol addition on the flotation response of 200-mesh wet-ground Pittsburgh No. 8 coal.	9-27
Table 9.16	- Effect of ethanol addition on the flotation response of 200-mesh wet-ground Upper Freeport PA coal.	9-28
Table 9.17	- Effect of ethanol addition on the flotation response of 200-mesh wet-ground Pittsburgh No. 8 coal.	9-28
Table 9.18	- Flotation results using alcohol homologs in the mill with 200-mesh wet-ground Upper Freeport PA coal. 6 lb/T of alcohol was used for each test.	9-29
Table 9.19	- Optimized flotation results using butanol added to the mill with 200-mesh wet-ground Upper Freeport PA coal.	9-29
Table 9.20	- Effect of butyl benzaldehyde addition on the flotation response of 28-mesh wet-ground Upper Freeport PA coal.	9-34
Table 9.21	- Effect of butyl benzaldehyde addition on the flotation response of 200-mesh wet-ground Upper Freeport PA coal.	9-34
Table 9.22	- Effect of glyoxal addition on the flotation response of 28-mesh wet-ground Upper Freeport PA coal.	9-35

Table 9.23	- Effect of glyoxal addition on the flotation of 28-mesh wet-ground Illinois No. 6 coal.	9-36
Table 10.1	- Two-stage flotation test results for Pittsburgh No. 8 coal with and without GMTG addition during grinding.	10-3
Table 10.2	- Single and two-stage flotation test results for Illinois No. 6 coal, in the presence and absence of GMTG.	10-3
Table 10.3	- Two-stage flotation test results for Upper Freeport PA coal with and without GMTG addition during grinding.	10-3
Table 10.4	- Effect of pH and calcium cyanide on pyrite depression of Pittsburgh No. 8 coal with 2 minutes additional conditioning time.	10-11
Table 10.5	- Effect of conditioning time on pyrite depression for Pittsburgh No. 8 coal in the absence of calcium cyanide.	10-11
Table 10.6	- Flotation results for Pittsburgh No. 8 coal as a function of calcium cyanide addition at pH 8.5 ± 0.5 (reagents added to the cell).*	10-11
Table 10.7	- Flotation results for Illinois No. 6 in the presence and absence of calcium cyanide at pH 7.8 ± 0.1 . Conditioning time was the standard time plus an additional 2 minutes with a collector dosage of 5.76 lb/T and frother dosage of 1.17 lb/T.	10-12
Table 10.8	- Flotation results for Upper Freeport PA in the presence and absence of calcium cyanide at pH 8.5 ± 0.3 . Conditioning time was the standard time plus an additional 2 minutes with a collector dosage of 0.48 lb/T and frother dosage of 0.21 lb/T.	10-13
Table 10.9	- Flotation of pyrite from Illinois No. 6 coal 200 mesh grind as a function of MIBC addition in the presence of 0.57 lb/Ton potassium ethyl xanthate (KEX) at pH 8 ± 0.5	10-19
Table 10.10	- Flotation of pyrite from Illinois No. 6 coal 200 mesh grind as a function of MIBC addition in the presence of 0.57 lb/T potassium ethyl xanthate (KEX) at pH 4 ± 0.1 . Frother conditioning time: 15 seconds.	10-19
Table 10.11	- Step flotation of pyrite from Illinois No. 6 coal 200 mesh grind in the presence of potassium ethyl xanthate (KEX) and MIBC at pH 4 ± 0.1	10-20
Table 10.12	- Step flotation results for pyrite flotation from Illinois No. 6 coal at 200 mesh grind in the presence of potassium ethyl xanthate (KEX), hydrogen peroxide (H_2O_2), and 0.78 lb/T of MIBC.	10-21
Table 10.13	- Step flotation results for pyrite flotation from Illinois No. 6 coal at 200 mesh grind in the presence of Aerofloat 25 Promoter, hydrogen peroxide (H_2O_2), and MIBC.	10-22

Table 11.1	- Reagent dosages used in the flotation kinetics tests.	11-1
Table 11.2	- Comparison of the ultimate recovery, flotation rate constant and the standard error (SE) of estimation of combustible materials of 100-mesh Upper Freeport PA coal obtained by fitting different kinetic models (dodecane 0.24 lb/T, MIBC 0.26 lb/T and pH 3.3).	11-4
Table 11.3	- Ranking of the standard error (SE) of estimation of the ultimate combustible material recovery of the three base coals obtained by fitting different flotation kinetic models.	11-5
Table 11.4	- Combustible material recovery (CMR), ash and pyritic sulfur rejection, and separation efficiency of Illinois No. 6 coal for 28-mesh grind (pH 5.5).	11-6
Table 11.5	- Combustible material recovery (CMR), ash and pyritic sulfur rejection, and separation efficiency of Pittsburgh No. 8 coal for 28-mesh grind (pH 3.3).	11-7
Table 11.6	- Combustible material recovery (CMR), ash and pyritic sulfur rejection, and separation efficiency of Upper Freeport PA coal for 28-mesh grind.	11-7
Table 11.7	- Compositional analysis of the 28-mesh samples of Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA coals.	11-8
Table 11.8	- Ultimate recoveries and flotation rate constants of combustible material, ash and pyritic sulfur of 28-mesh Illinois No. 6 coal (pH 5.5).	11-11
Table 11.9	- Ultimate recoveries and flotation rate constant of combustible material, ash and pyritic sulfur of 28-mesh Pittsburgh No. 8 coal (pH 3.3). ...	11-11
Table 11.10	- Ultimate recoveries and flotation rate constant of combustible material, ash and pyritic sulfur of 28-mesh Upper Freeport PA coal.	11-11
Table 11.11	- Combustible material recovery (CMR), ash and pyritic sulfur rejection, and separation efficiency of 100-mesh Illinois No. 6 coal.	11-12
Table 11.12	- Combustible material recovery (CMR), ash and pyritic sulfur rejection, and separation efficiency of 100-mesh Pittsburgh No. 8 coal.	11-13
Table 11.13	- Combustible material recovery (CMR), ash and pyritic sulfur rejection, and separation efficiency of 100-mesh Upper Freeport PA coal.	11-14
Table 11.14	- Compositional analysis of the 100-mesh samples of Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA coals.	11-15
Table 11.15	- Ultimate recovery, flotation rate constant and the maximum separation efficiency of 100-mesh Illinois No. 6 coal.	11-15

Table 11.16 - Ultimate recovery, flotation rate constant and the maximum separation efficiency of 100-mesh Pittsburgh No. 8 coal.	11-19
Table 11.17 - The ultimate recovery, flotation rate constant and the maximum separation efficiency of 100-mesh Upper Freeport PA coal.	11-19
Table 11.18 - Combustible material recovery (CMR), ash and pyritic sulfur rejection, and separation efficiency of 200-mesh Illinois No. 6 coal.	11-20
Table 11.19 - Combustible material recovery (CMR), ash and pyritic sulfur rejection, and separation efficiency of 200-mesh Pittsburgh No. 8 coal.	11-21
Table 11.20 - Combustible material recovery (CMR), ash and pyritic sulfur rejection, and separation efficiency of 200-mesh Upper Freeport PA coal.	11-21
Table 11.21 - Compositional analysis of the 200-mesh samples of Illinois No. 6 coal, Pittsburgh No. 8 and Upper Freeport PA coals.	11-22
Table 11.22 - The ultimate recovery, flotation rate constant and the maximum separation efficiency of 200-mesh Illinois No. 6 coal (Dodecane 4.92 lb/T, MIBC 0.98 lb/T).	11-30
Table 11.23 - The ultimate recovery, flotation rate constant and the maximum separation efficiency of 200-mesh Illinois No. 6 coal (Dodecane 9.84 lb/T, MIBC 0.98 lb/T).	11-30
Table 11.24 - The ultimate recovery, flotation rate constant and the maximum separation efficiency of 200-mesh Illinois No. 6 coal (Dodecane 4.92 lb/T, MIBC 1.95 lb/T).	11-31
Table 11.25 - The ultimate recovery, flotation rate constant and the maximum separation efficiency of 200-mesh Pittsburgh No. 8 coal (Dodecane 1.92 lb/T, MIBC 0.30 lb/T and pH 3.71).	11-31
Table 11.26 - The ultimate recovery, flotation rate constant and the maximum separation efficiency of 200-mesh Pittsburgh No. 8 coal (Dodecane 3.84 lb/T, MIBC 0.30 lb/T).	11-31
Table 11.27 - The ultimate recovery, flotation rate constant and the maximum separation efficiency of 200-mesh Pittsburgh No. 8 coal (Dodecane 1.92 lb/T, MIBC 0.60 lb/T).	11-31
Table 11.28 - The ultimate recovery, flotation rate constant and the maximum separation efficiency of 200-mesh Upper Freeport PA coal (Dodecane 0.24 lb/T, MIBC 0.26 lb/T).	11-32
Table 11.29 - The ultimate recovery, flotation rate constant and the maximum separation efficiency of 200-mesh Upper Freeport PA coal (Dodecane 0.48 lb/T, MIBC 0.26 lb/T).	11-32

Table 11.30	- The ultimate recovery, flotation rate constant and the maximum separation efficiency of 200-mesh Upper Freeport PA coal (Dodecane 0.24 lb/T, MIBC 0.52 lb/T).	11-32
Table 11.31	- Combustible material recovery (CMR), ash and pyritic sulfur rejection, and separation efficiency of Illinois No. 6 coal for different grind sizes.	11-33
Table 11.32	- Combustible material recovery (CMR), ash and pyritic sulfur rejection, and separation efficiency of Pittsburgh No. 8 coal for different grind sizes.	11-33
Table 11.33	- Combustible material recovery (CMR), ash and pyritic sulfur rejection, and separation efficiency of Upper Freeport PA coal for different grind sizes.	11-34
Table 11.34	- Effect of grind size on the efficiency index, ultimate recovery and flotation rate constants of the combustible material, ash and pyritic sulfur of Illinois No. 6 coal.	11-34
Table 11.35	- Effect of grind size on the efficiency index, ultimate recovery and flotation rate constants of the combustible material, ash and pyritic sulfur of Pittsburgh No. 8 coal.	11-35
Table 11.36	- Effect of grind size on the efficiency index, ultimate recovery and flotation rate constants of the combustible material, ash and pyritic sulfur of Upper Freeport PA coal.	11-35
Table 11.37	- Collector and frother dosages used in the flotation tests.	11-38
Table 11.38	- Flotation yield of minus 200-mesh Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA coals at different flotation times.	11-38
Table 11.39	- The fitting parameters of equation 11.4c for the flotation data of the three base coals.	11-39
Table 12.1	- Combustible material recovery (CMR), ash and pyritic sulfur rejection, and separation efficiency obtained in the first step flotation optimization tests with 200-mesh wet-ground Illinois No. 6 coal.	12-2
Table 12.2	- Effect of collector (dodecane) and frother dosage (MIBC) on the separation efficiency index of Illinois No. 6 in the first step optimization tests.	12-3
Table 12.3	- Combustible material recovery (CMR), ash and pyritic sulfur rejection, and separation efficiency obtained in the second step flotation optimization tests with 200-mesh wet-ground Illinois No. 6 coal.	12-4
Table 12.4	- Effect of collector (dodecane) and frother dosage (MIBC) on the separation efficiency index obtained in the second step optimization tests with Illinois No. 6 coal.	12-4

Table 12.5	- Flotation rate constant and ultimate recoveries of combustible materials, ash and pyritic sulfur of 200-mesh wet-ground Illinois No. 6 coal obtained from the second set of optimization tests.	12-5
Table 12.6	- Statistical analysis of the effect of collector (dodecane) and frother dosage (MIBC) on the flotation rate constant and ultimate recovery of combustible materials, ash and pyritic sulfur of Illinois No. 6 coal in the second set of optimization tests.	12-5
Table 12.7	- Flotation reagent optimization results for 200-mesh wet-ground Upper Freeport PA coal.	12-6
Table 12.8	- Optimization of standard flotation test results for 200-mesh wet-ground Upper Freeport PA coal.	12-6
Table 12.9	- Optimization flotation test results for 100-mesh wet-ground Upper Freeport PA coal.	12-7
Table 12.10	- Optimization flotation test results for 325-mesh wet-ground Upper Freeport PA coal.	12-7
Table 12.11	- The test results of flotation kinetics using dodecane 0.25 lb/T and MIBC 0.70 lb/T for 100-mesh wet-ground Upper Freeport PA coal.	12-9
Table 12.12	- The results of flotation kinetics using MIBC 1.0 lb/T for wet-ground minus 200-mesh Upper Freeport PA coal.	12-9
Table 12.13	- The test results of flotation kinetics using dodecane 0.30 lb/T and MIBC 1.30 lb/T for 325-mesh wet-ground Upper Freeport PA coal.	12-9
Table 12.14	- Effect of frother conditioning time on flotation of Illinois No. 6 coal (200 mesh grind) at collector (dodecane) dosage 4.92 lb/T, frother (MIBC) dosage 0.98 lb/T and pH 7.2.	12-13
Table 12.15	- Effect of frother conditioning time on flotation of Pittsburgh No. 8 coal (200-mesh grind at collector (dodecane) dosage 1.92 lb/T, frother (MIBC) dosage 0.39 lb/T and pH 3.9.	12-13
Table 12.16	- Effect of frother conditioning time on flotation of Upper Freeport PA coal (200 mesh grind) at collector (dodecane) dosage 0.48 lb/T, frother (MIBC) dosage 0.21 lb/T and pH 3.4.	12-14
Table 12.17	- Flotation results of five cycles using MIBC 1.0 lb/T for wet-ground 200-mesh Upper Freeport PA coal.	12-16
Table 12.18	- Flotation results of 200-mesh wet-ground Upper Freeport PA coal for five cycles with 4.0 lb/T butanol added to the mill.	12-17
Table 12.19	- Flotation results with cleaning and scavenging for 200-mesh wet-ground Upper Freeport PA coal ground with 6.0 lb/T ethanol.	12-17

Table 12.20	- Flotation results with cleaning and scavenging for 200-mesh wet-ground Upper Freeport PA coal ground with 4.0 lb/T butanol.	12-18
Table 12.21	- Flotation results of 200-mesh wet-ground Upper Freeport PA coal with 6 lb/T ethanol added to the mill with staged collector and frother addition.	12-19
Table 12.22	- Flotation results using 3 lb/T butanol in the mill and staged collector addition in the cell for 200-mesh wet-ground Upper Freeport PA coal.	12-20
Table 12.23	- Flotation results using 4 lb/T butanol in the mill and staged collector addition in the cell for 200-mesh wet-ground Upper Freeport PA coal.	12-20
Table 12.24	- Results of combined tabling and flotation for pyritic sulfur removal from the base coals.	12-25
Table 13.1	- Coal weathering sampling schedule for the three base coals.	13-5
Table 13.2	- Ash and total sulfur of the minus 28-mesh screened fraction of Pittsburgh No. 8 weathering samples reconstituted from flotation product analysis.	13-7
Table 13.3	- Ash and total sulfur of the minus 28-mesh screened fraction of Illinois No. 6 weathering samples reconstituted from flotation product analysis.	13-7
Table 13.4	- Ash and total sulfur of the minus 28-mesh screened fraction of Upper Freeport PA weathering samples reconstituted from flotation product analysis.	13-7
Table 13.5	- Proximate analysis of weathered samples of Illinois No. 6 coal.	13-11
Table 13.6	- Proximate analyses of weathered samples of Pittsburgh No. 8 coal. ...	13-12
Table 13.7	- Proximate analyses of weathered samples of Upper Freeport PA coal.	13-13
Table 13.8	- Proximate analysis of deslimed 100 x 150 mesh weathered samples stored in the open mode, their film flotation response, induction time for the three weathering modes, and the standard flotation of minus 28-mesh weathered coal.	13-24
Table 14.1	- Flotation results for Illinois No. 6 coal using the Tree Analysis Procedure. Standard flotation conditions, 8.21 lb/T dodecane and 2.63 lb/T were used. The flotation pH was 3.7.	14-4
Table 14.2	- Flotation results for Illinois No. 6 coal using Tree Analysis Procedure. Standard flotation conditions, 7.44 lb/T dodecane and 1.76 lb/T MIBC were used. The flotation pH was 3.5.	14-6

Table 14.3	- Flotation results for Illinois No. 6 coal using Release Analysis Procedure. Standard flotation conditions, 7.83 lb/T dodecane and 1.54 lb/T MIBC were used. The flotation pH was 3.8.	14-6
Table 14.4	- Flotation results for Pittsburgh No. 8 coal using the Tree Analysis Procedure. Standard flotation conditions, 2.52 lb/T dodecane and 0.6 lb/T MIBC were used. The flotation pH was 3.7.	14-7
Table 14.5	- Flotation results for Upper Freeport PA coal using the Tree Analysis Procedure. Standard flotation conditions, 0.57 lb/T dodecane and 0.36 lb/T MIBC were used. The flotation pH was 3.5.	14-9
Table 14.6	- Comparison of flotation behavior of Illinois No. 6 coal (200 mesh wet grind) in drum 3 (B1-03) and drum 4 (B1-04) using standard flotation conditions (4.92 lb/T dodecane and 0.98 lb/T MIBC.)	14-10
Table 14.7	- Comparison of the average initial flotation pH of Illinois No. 6 coal samples stored in Drum 3 (B1-03) and Drum 4 (B1-04).	14-11

COAL SURFACE CONTROL FOR ADVANCED FINE COAL FLOTATION

DOE PROJECT NO. DE-AC22-88PC88878

Final Technical Report

1.0 INTRODUCTION

Historically, coal surface characterization and control have not been considered critical to coal cleaning because of the prior emphasis on maintaining particle size as coarse as possible. However, the current goal of near-total removal of pyritic sulfur necessitates fine grinding of coal to liberate pyrite. At these fine sizes coal surface behavior plays an increasingly dominant role and consequently the need for surface characterization and control is growing sharply.

In order to investigate the properties of coal surfaces and their role in coal flotation, DOE awarded a contract entitled "Coal Surface Control for Advanced Fine Coal Flotation" to the University of California at Berkeley in October 1988. The main goal of the project was to characterize the surface and control the behavior of coal during advanced flotation processing in order to achieve an overall objective of removing 90% of the pyritic sulfur at 90% Btu recovery. Also, the investigation of the effects of weathering on the surface characteristics of coal was another important aspect of this project.

1.1 Project Team

The project team consisted of research and engineering groups at Berkeley, Columbia University, the University of Utah and Praxis Engineers, Inc., with the University of California acting as the prime contractor. The organizational chart for conducting the program encompassed by this project is presented in Figure 1.1, which also identifies key project personnel.

The work distribution on this project took into consideration the equipment and expertise available at the facilities of the four participating organizations in accordance with the Final Work Plan submitted at the onset of the contract. The main role of Praxis Engineers was in selecting the mine sites, preparing mined coal samples and the weathering sites, collecting weathered samples, implementing the washability tests at various commercial testing laboratories, and helping coordinate the research during the early stages of the program. At the University of Utah, the

Figure 1.1 - Project organizational chart.

coal was crushed to minus 1/4 inch to be sent to the other research groups. Utah also conducted an extensive liberation study, characterized the petrography, developed the standard grinding and flotation tests for Upper Freeport PA coal, conducted studies on surface modification with reactive but non-polymerizable reagents, and tested various flotation circuit designs. At Columbia University, various bulk and surface characterization studies were completed, including surface characterization by zeta-potential measurement, and by surface element and functional group analysis using ESCA and DRIFT. The group at Columbia also developed the standard grinding and flotation tests for Pittsburgh No. 8 coal, conducted surface modification tests using anionic surfactants and pH modifiers, studied the effect of dispersants and pyrite depressants, and investigated the effect of various machine parameters on flotation performance. Research at Berkeley consisted of bulk and surface characterization studies including detailed wettability studies. The Berkeley group worked out the general grinding procedure to be used in this program, developed the standard grinding and standard flotation tests for Illinois No. 6 coal, conducted flotation studies with polymerizable monomers, non-ionic surfactants and numerous pyrite depressants and conducted a detailed flotation kinetics study on 28-, 100- and 200-mesh coal. The effect of grinding atmosphere and grinding media was investigated at Berkeley. In addition, the Berkeley group carried out the detailed characterization and flotation study of weathered coal. Throughout the course of this program, numerous tests and analyses were performed by the participating groups or at commercial laboratories for QA/QC.

The University of California, acting as prime contractor, was also responsible for all technical and administrative reporting. Technical reports were prepared using input submitted by the various research and engineering groups.

1.2 Project Goals

The primary objective of this research project was to develop advanced flotation methods for coal cleaning in order to achieve the highest possible pyritic sulfur removal at high Btu recovery, using coal samples procured from six major U.S. coal seams. Concomitantly, the ash content of these coals was to be reduced to 6% or less. Investigation of mechanisms for the control of coal and pyrite surfaces prior to fine coal flotation was the central aspect of the project

objectives. Detailed experimental and procurement plans for the contract were drafted during the initial months of the contracting period and were submitted to DOE as a Work Plan.

As a part of this contract, large quantities of coal samples were procured from six major seams identified by DOE for use in this project for advanced flotation and weathering studies. Samples of the same coals were also supplied to the University of Pittsburgh for selective agglomeration research under a separate contract by DOE.

A second major objective was to investigate factors involved in the progressive weathering and oxidation of coal that had been stored in three storage modes, namely, open, covered and inerted under argon atmosphere, over a period of twelve months. After regular intervals of weathering, samples of the three base coals (Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA) were collected and shipped to both the University of Pittsburgh and the University of California at Berkeley for characterization studies of the weathered material.

1.3 Scope of this Document

Intensive research efforts were exerted as part of this program, and therefore the amount of data produced were also extensive. Many man hours of labor went into the analysis and preparation of this report by members of the various research organization. Extensive effort was needed to integrate all the results pertaining to a given topic, and present them in a coherent manner. To do this, prior technical reports, particularly Annual Reports 1 and 2, have been incorporated and supplemented with new results where appropriate. This report is broken down into 14 chapters in order to present the results in a logical and cohesive manner. This chapter serves as an introduction and the second chapter presents a short description of the procurement of the coal samples. Chapters 3, 4 and 5 present results of detailed characterization studies, while 6 and 7 discuss the development of the standard grinding and flotation procedures. Chapters 8-12 present various aspects of the flotation research including the effect of grinding environment, surface modifiers, pyrite depressants and flotation circuitry. Chapter 13 gives the results of the weathering study and Chapter 14 shows the effect of storage on laboratory research coals. Chapter 15 is a summary of the major conclusions and findings for the entire report and Chapter 16 includes recommendations for future work.

2.0 COAL PROCUREMENT

2.1 Overview and Scope

The first major task was to provide a large sample of coal from the designated seams for research and testing of the two leading surface-based advanced physical fine coal cleaning technologies that had been selected by DOE, namely advanced froth flotation and selective agglomeration. The flotation contract was awarded to the University of California - Berkeley team and the selective agglomeration research contract was awarded to the University of Pittsburgh team.

To provide the same starting material for both projects and to ensure the uniformity of samples for these research contracts, DOE consolidated the sampling requirements--along with the weathering program--into a single effort. This work, which was executed by Praxis Engineers, Inc., acting as subcontractors to University of California, involved obtaining samples from six different coal seams.

Three of the seams are designated as base coals in this program and three as additional coals. These seams, which are either major U.S. bituminous or subbituminous coal seams currently being mined, represent a broad range of ash and pyritic sulfur contents. The seams were designated by DOE but mine sites were selected by Praxis Engineers. To expedite the procurement effort due to the approach of winter, work on coal collection was started by Praxis Engineers, on November 4, 1988. A list of the coal seams and mine locations is given in Table 2.1.

Extensive site preparation was carried out, particularly at the base coal sites, and the material was collected from fresh mine faces after clearing clay and overburden. The topsize of the coal was reduced to 4-6 inches in the field and care was taken to prepare and homogenize each sample prior to packing it into 55-gallon drums (from 400 to 450 pounds of coal per drum) and "inerting" for shipment to various locations for use for the following purposes:

- Research samples
 - Advanced froth flotation
 - Selective agglomeration (UP)
- Washability and liberation studies
- Weathering studies
- Reference (save) samples

Table 2.1 - List of coal seams sampled and corresponding mine locations.

<u>SEAM</u>	<u>COUNTY, STATE</u>	<u>MINE OPERATOR</u>	<u>MINE LOCATION</u>
<u>Base Coals</u>			
Illinois No. 6	Randolph, IL	Peabody Coal Co.	Marissa, IL
Pittsburgh No. 8	Belmont, OH	R&F Coal Co.	Warnock, OH
Upper Freeport PA	Indiana, PA	N. Cambria Fuel Co.	Blacklick, PA
<u>Additional Coals</u>			
Kentucky No. 9	Hopkins, KY	P&M Coal Mining Co.	Madisonville, KY
Upper Freeport WV	Grant, WV	Buffalo Coal Co.	Mount Storm, WV
Wyodak	Campbell, WY	Sunedco Coal Co.	Gillette, WY

About 23 tons of each base coal (Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA) and 5 tons of each of the additional coals (Kentucky No. 9, Upper Freeport WV and Wyodak) were collected from the six operating open pit mines.

A second objective of this task was to develop a washability data base for these coals, using a representative portion of the gross sample from each mine. Details of the washability studies are discussed in Chapter 4 of this report.

Another objective of this task was to prepare sites for weathering the coal samples on location under various storage modes for a period of up to one year. The overall purpose of this task was to study the weathering characteristics of the three base coals and to delineate the importance of natural surface oxidation on fine coal cleaning. Previous experience with advanced cleaning technologies had indicated that the storage history of the coal after mining can have a major impact on the separation efficiency of the process as well as on coal utilization. In particular, surface oxidation resulting from exposure to oxygen is considered to be detrimental to surface-based fine coal cleaning processes. Thus samples were regularly collected and shipped to the University of California for detailed study of how weathering affected the coal surface and bulk characteristics and its flotation response.

The three storage modes, open, covered and inert, represent high, moderate, and low levels of exposure to oxidation or weathering. A further refinement to the experimental design

was achieved by evaluating weathering behavior as a function of coal size by screening the coal into three size fractions, +1-inch, 1 x 1/4-inch and 1/4-inch x 0, prior to setting up weathering samples. In total there were nine weathering samples for each base coal, that is, three size fractions each stored under the three different conditions. Details are given in Chapter 13 of this report.

2.2 Coal Sample Procurement and Distribution

A gross sample of over 23 tons of each base coal was collected and split into two parts: 40% (about 9 tons) constituting the research sample and 60% (about 14 tons) constituting the weathering sample. The coal was mined from a fresh seam at each site in such a way that the sample was free of coarse shale and slate. In effect, the material was equivalent to a coarse-cleaned sample in each case. The research sample for the three base coals was further split into the following subsamples:

- Advanced flotation research sample for Berkeley, Columbia and Utah (2 drums each)
- Selective agglomeration research sample for the University of Pittsburgh (5 drums)
- Washability sample to a commercial laboratory (5 drums)
- Berkeley save sample (5 drums)
- DOE reserve sample (25 drums)

The additional coals were split into the same sub-samples with the exception of the DOE reserve sample which was omitted.

The splitting of the gross sample from each mine was done manually by using 5-gallon buckets that contained about 20 pounds of coal. Each drum was filled with about 20-25 increments that were taken from the parent sample at random. The drums were then "inerted" by purging with argon gas equivalent to approximately three times the volume of the drum with a copper tube inserted in the polyethylene bag lining the drum. The plastic bag was then tied at the top, argon purged between the bag and drum, the lid closed and the drum clamped shut. Figure 2.1 is a photograph of the drums being filled at the North Cambria Fuel Co. mine (Upper Freeport PA coal) in Indiana County, Pennsylvania.

Figure 2.1 - Drums of Upper Freeport PA coal being filled with 20-lb increments at the North Cambria Fuel Co. mine in Indiana County, Pennsylvania.

2.3 Primary and Secondary Crushing

In the Project Work Plan, the feed for the two series of flotation tests was set at 95% passing 28 mesh and 95% passing 200 mesh. In order to achieve the required size distribution, the feed coal was first crushed in two stages under argon atmosphere by covering the crusher with a plastic tent. The first-stage (primary) size reduction involved crushing the coal sample from a nominal 4-inch top size to 3/4-inch nominal top size in a laboratory jaw crusher. The secondary crushing stage consisted of further reducing the coal to 1/4-inch nominal top size in a laboratory roll crusher. One drum of coal, which is representative of that coal at the 4-6 inch top size, was crushed before preparing sub-samples to ensure that the sub-samples were also representative. The primary crushed product was homogenized and split by coning and quartering under an argon atmosphere and bagged in 30-lb bags. These samples were prepared for the University of California, Columbia University and University of Utah at the University of Utah laboratories.

Secondary crushing of the coal was done by Berkeley and Utah. Columbia received already crushed samples for flotation feed directly from Utah on a bimonthly basis.

3.0 BULK CHARACTERIZATION STUDIES

Characterization of the base coals broadly falls into two categories, bulk and surface characterization. Results of bulk characterization on the three base coals are discussed in this chapter.

Bulk properties reported here include i) the chemical composition as determined from proximate, ultimate and sulfur-forms analyses, ii) mineralogical content obtained using diffuse reflectance infrared Fourier transform (DRIFT) spectroscopy, iii) pyritic and non-pyritic mineral matter determined by SEM/EDXRF (scanning electron microscopy/energy dispersive x-ray fluorescence), iv) optical image analysis and v) the maceral content of the three coals determined by petrographic analysis. The SEM/EDXRF study was a qualitative analysis of the distribution of pyritic and non-pyritic mineral matter present in different size fractions from minus 28 mesh material. This study also qualitatively assessed the iron/sulfur ratios of the pyritic minerals contained in the base coals. Optical image analysis was carried out to quantitatively analyze the distribution of locked, semi-locked and free pyrite in different size fractions contained in coal ground to minus 200 mesh.

3.1 Chemical (Compositional) Analysis

The proximate analysis was carried out using a LECO MAC 400 Automatic Analyzer. The moisture, volatile matter, ash and fixed carbon were determined gravimetrically by heating the samples at different temperatures: 106°C for moisture, 750°C for volatile matter and 950°C for ash content. A nitrogen atmosphere was maintained in the furnace for the moisture and volatile modes whereas an oxygen atmosphere was used for the ash mode. Fixed carbon was determined by difference. Analysis of sulfur-forms was performed following the procedure described in ASTM No. 2492.

Proximate analysis and the sulfur-forms analyses of the three base coal samples are given in Tables 3.1 and 3.2 respectively. It can be seen from Table 3.1 that the volatile matter of Illinois No. 6 and Pittsburgh No. 8 samples are quite close to each other, while that of the Upper Freeport PA sample is much lower. In terms of ash and moisture contents, on the other hand, Pittsburgh No. 8 and Upper Freeport PA coal samples are closer to one another, while the

Table 3.1 - Proximate analysis of the three base coals.

	PROXIMATE ANALYSIS, DRY WT. %			
	As received Moisture %	Vol. matter	Fixed Carbon	Ash
Illinois No. 6	9.5	36.2	46.3	17.5
Pittsburgh No. 8	2.3	35.7	52.5	11.8
Upper Freeport PA	1.0	26.2	61.8	12.0

moisture and ash contents of the Illinois No. 6 sample are higher than those of the other two coal samples. Although the moisture content may be a direct function of the hydrophobicity of the coal, the relationship is much too complex and may vary with the origin of the coal, weather conditions, or a host of other variables. However, from the results given in Table 3.1, one may cautiously infer that the Upper Freeport PA coal is the most hydrophobic of the three coals and Illinois No. 6 the least, with Pittsburgh No. 8 coal falling somewhere between the other two and closer to the former.

Another important observation is that the pyritic sulfur, as a percentage of the total sulfur, is the highest in Upper Freeport PA (69 wt%), followed by Pittsburgh No. 8 (60 wt%) and Illinois No. 6 coal (48 wt%). This implies that the application of physical separation techniques, such as froth flotation and selective agglomeration, will be most effective to desulfurize Upper Freeport PA coal, followed by Pittsburgh No. 8 and Illinois No. 6 coals, assuming equal liberation of the pyritic sulfur.

The ultimate analysis of the three coals was determined using a LECO CHN 600 Analyzer. The carbon, hydrogen and nitrogen contents of the coals were obtained by burning coal samples in pure oxygen at 950°C. The instrument measures the amount of CO₂ and H₂O in the

Table 3.2 - Analysis of sulfur forms for the three base coal samples.

	FORMS OF SULFUR (DRY BASIS)						Btu/lb
	Moisture %	Ash %	Sulfatic %	Pyritic %	Organic %	Total %	
Illinois No. 6	8.87	15.5	0.28	2.56	2.53	5.37	11,320
Pittsburgh No. 8	2.74	12.6	0.25	2.82	1.64	4.71	12,420
Upper Freeport PA	0.86	12.5	0.10	1.62	0.63	2.35	13,370

Table 3.3 - Ultimate analysis of the three base coals.

<u>Coal sample</u>	<u>Moisture</u>	<u>ELEMENT, PERCENT (DRY BASIS)</u>				<u>Sulfur</u>	<u>Oxygen</u>
		<u>Carbon</u>	<u>Hydrogen</u>	<u>Nitrogen</u>			
Illinois No. 6	9.5	63.8	5.7	1.24		5.73	6.10
Pittsburgh No. 8	2.2	71.0	5.1	1.45		4.28	6.40
Upper Freeport PA	1.0	75.6	4.7	1.45		2.38	3.85

combustion gases by passing them through carbon and hydrogen infrared cells. NO_x must first be reduced to N_2 by hot copper and a catalyst, and the elemental nitrogen concentration is then determined with a thermal conductivity cell.

Results of the ultimate (elemental) analysis of the three base coals are summarized in Table 3.3. Examination of these results shows that the bulk carbon-to-oxygen ratio follows the order: Upper Freeport PA (19.7) > Pittsburgh No. 8 (11.1) > Illinois No. 6 (10.5). This ratio can be considered a measure of the hydrophobicity since the major oxygen containing functional groups in coal such as $-\text{COOH}$ (carboxylic) and ArOH (phenolic) are hydrophilic. While hydrophobicity of the coals might be expected to follow the order shown above, it cannot be related rigorously to the flotability of the three coals which is a function of surface hydrophobicity and hence the surface carbon-to-oxygen ratio which may differ significantly from the bulk ratio. As amply demonstrated by Fuerstenau (1) and by Gutierrez-Rodriguez and Aplan (2) using contact angle studies, oxygen reacts instantaneously with some coals and therefore the surface carbon-to-oxygen ratio can be expected to be lower than that of the bulk. The dry mineral matter free (dmmf) carbon content of coal can be considered as one more measure of the hydrophobicity of coal. The contact angle and natural flotability of coals have been reported to pass through a maximum at about 88% carbon content (dmmf) (3-5). Calculations of the dmmf carbon content of the three coals show that the flotability of the three coals should vary in the following order: Upper Freeport (85.9% C dmmf) > Pittsburgh No. 8 (80.5% C dmmf) > Illinois No. 6 (77.3% C dmmf). It is to be noted that the proximate analysis also indicated the same trend with respect to the carbon content.

3.2 Mineralogical Analysis

The ash mineral composition of the coal can have a significant effect on the flotation of coal and pyrite. Therefore, qualitative analysis of the mineralogical content of the three base coals was carried out using diffuse reflectance infrared Fourier transform (DRIFT) spectroscopy. Samples for the DRIFT analysis were prepared by grinding the coals to minus 60 mesh followed by ashing them at 700°C for 4 hours in a muffle furnace. The air flow rate through the furnace was maintained at 4 liters/min. The samples were then cooled inside the furnace, ground in an agate mortar to minus 400 mesh and then stored in glass vials until needed. Two hundred milligrams of IR grade potassium bromide was ground to minus 400 mesh and mixed with six milligrams of the ash and the mixture further ground using an agate mortar in order to intimately mix them. The ground mixture was then placed in the sample cell of the diffuse reflectance accessory of the Perkin-Elmer 1800 FTIR spectrometer. The sample was scanned for 100 cycles in single-ratio mode and the spectra obtained was smoothed and analyzed using a Perkin-Elmer 7500 data processing system.

Diffuse reflectance spectra of three commonly found minerals in coal, namely, illite, quartz and calcite were also obtained for the purpose of peak calibration. Wave numbers at which strong IR absorption is found for other minerals such as kaolinite, montmorillonite, attapulgite, dolomite, siderite, and gypsum were obtained from the literature (6-8). Table 3.4 lists various minerals commonly found in coal and the corresponding wave numbers. Diffuse reflectance spectra of ash fractions obtained from the three base coals are presented in Figure 3.1. It can be seen from this figure that the ash fractions from all the three coals show a major broad peak around 1045 cm^{-1} , peaks of intermediate intensity around 795 cm^{-1} , 565 cm^{-1} and 478 cm^{-1} and a minor peak around 690 cm^{-1} . Matching these peaks with wave numbers in Table 3.4 indicates that all three samples probably contain kaolinite (1090, 1035, 1010, and 490 cm^{-1}) and quartz (1156, 1045, 795 and 695 cm^{-1}). It appears that the 1090 and 1035 cm^{-1} peaks of kaolinite and 1156 and 1045 cm^{-1} peaks of quartz all overlap and form a broad peak around 1045 cm^{-1} . However, the presence of these two minerals can be confirmed from their other characteristic peaks as indicated in Figure 3.1. In addition to kaolinite and quartz, which were identified in all three coals, the ash from Illinois No. 6 coal also shows peaks around 1455 and 873 cm^{-1} ,

Figure 3.1 - Diffuse reflectance spectra of ash from the three base coals.

characteristic of calcite. The presence of calcite in Illinois No. 6 coal is also supported by results from neutron activation analysis (NAA), which yielded a calcium content of 0.7 weight percent for the Illinois No. 6 sample; the calcium content of the other two coals were found to be below the detectable limit of NAA which is 0.1 wt%. Assuming that the calcium content determined by NAA in Illinois No. 6 coal is from calcite, its calcite content can be calculated to be 1.75 weight percent.

Thus, a qualitative mineralogical analysis of ash from the base coals using DRIFT showed that all three coals contained kaolinite and quartz while Illinois No. 6 coal also contained calcite. No other mineral was present at detectable levels.

Table 3.4 - Infrared wavenumbers (cm⁻¹) for various minerals present in coal.

<u>Class</u>	<u>Division</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>Reference</u>
Silicates	Quartz	-	790	1046	1156	695	-	Prev. work
Clays	Kaolinite	1035	1010	535	478	1090	910	Painter (8)
	Montmorillonite	1050	530	-	-	-	-	Painter (8)
	Attapulgit	1030	985	1665	1325	1190	910	Hunt (6)
	Illite	1025	-	-	-	-	-	Prev. work
Carbonates	Calcite	1447	877	712	845	-	-	Prev. work
	Dolomite	882	730	-	-	-	-	Painter (8)
	Siderite	872	740	-	-	-	-	Painter (8)
Sulfates	Gypsum	669	603	-	-	-	-	Painter (8)

3.3 Photomicrographic Studies of Mineral Matter Association

3.3.1 SEM/EDXRF Study

Scanning electron microscopy coupled with energy dispersive x-ray fluorescence (SEM/EDXRF) was used to analyze the nature of association of mineral matter with coal. This work was carried out at the Lamont Geological Observatory (affiliated with Columbia University) using a Cambridge Model 250 scanning electron microscope and a Cevax Model 8000 energy dispersive x-ray fluorescence system. The sample preparation procedure consisted of riffing one kilogram of a minus 1/4-inch sample several times to reduce the sample size to about 40-50 grams. Each subsample was ground with an agate mortar, in a glove box maintained under an argon atmosphere, to minus 28-mesh and the ground material further split into 10-gram samples. Each sample was then sieved to produce 28 x 50-mesh, 65 x 100-mesh, 100 x 200-mesh and minus 200-mesh size fractions which were used for the analysis.

Scanning electron micrographs of the 28 x 65-mesh fractions of Pittsburgh No. 8, Illinois No. 6, and Upper Freeport PA coals are given in Figures 3.2, 3.3 and 3.4, respectively. All SEM micrographs were taken in the back-scatter mode in which the mineral matter appears bright in contrast to dark coal. It can be seen from these photographs that for all the three coals investigated no liberated pyrite or non-pyritic minerals could be seen in the 28 x 65-mesh fraction. Photomicrographs of the 65 x 100-mesh size fraction of Pittsburgh No. 8, Illinois No. 6, and Upper Freeport PA coal samples are shown in Figures 3.5, 3.6 and 3.7, respectively. Only in the

Figure 3.2 - SEM Micrograph of a 28 x 65-mesh sample of Pittsburgh No. 8 coal.

Figure 3.3 - SEM Micrograph of a 28 x 65-mesh sample of Illinois No. 6 coal.

Figure 3.4 - SEM Micrograph of a 28 x 65-mesh sample of Upper Freeport PA coal.

Figure 3.5 - SEM Micrograph of 65 x 100-mesh sample of Pittsburgh No. 8 coal.

Figure 3.6 - SEM Micrograph of a 65 x 100-mesh sample of Illinois No. 6 coal.

Figure 3.7 - SEM Micrograph of a 65 x 100-mesh sample of Upper Freeport PA coal.

case of Upper Freeport PA sample [particles marked 1 (Fe, S) and 2 (Ca) in Figure 3.7] a few liberated mineral particles can be seen. In the 100 x 200 mesh size range, one liberated mineral particle each can be seen in the micrographs of Pittsburgh No. 8 sample [Figure 3.8; area marked 1 (Fe, S) and 2 (Al, Si clays) and Illinois No. 6 coal sample [Figure 3.9; particle marked 1 (Ca)]. On the other hand, at least four liberated particles can be seen in the 100 x 200-mesh fraction of Upper Freeport PA coal [Figure 3.10; particles marked 1 (Fe, Ti), 2 (K, Si, Fe, Ti), 3 (Fe, S) and 4 (Al, Si)]. Photomicrographs of the minus 200-mesh fractions of the three base coals are shown in Figures 3.11 to 3.13. It can be seen from Figure 3.12 that almost all the mineral particles are liberated in the minus 200-mesh fraction of Illinois No. 6 coal, x-ray fluorescence analysis showed that most of these particles are pyrite. The minus 200-mesh fraction of Pittsburgh No. 8 coal (Figure 3.11) also exhibited characteristics similar to that of Illinois No. 6 sample. In the case of the Upper Freeport PA sample, although most of the mineral particles are seen to be liberated in the minus 200-mesh fraction and x-ray fluorescence analysis showed that the majority of these particles are non-pyritic mineral as indicated in Figure 3.13. Typical spectra of the iron sulfide particles identified in the minus 200-mesh fractions of the three base coals are given in Figures 3.14 to 3.16. It can be seen from these figures that the relative intensity of the sulfide peak is higher than that of the iron peak of pyrite from the Illinois No. 6 sample (Figure 3.15) and the Upper Freeport PA sample (Figure 3.16) whereas the intensity of the iron peak is higher for pyrite from the Pittsburgh No. 8 sample. This indicates that Pittsburgh No. 8 sample contains iron-bearing minerals in addition to pyrite, possibly non-stoichiometric iron-sulfide minerals.

SEM/EDXRF analysis thus showed that in the 28-mesh x 0 grind, almost no free pyrite particles exist above 200 mesh in samples of Illinois No. 6 and Pittsburgh No. 8 coals, whereas free pyrite particles in the Upper Freeport PA sample exist also in coarser sizes.

An interesting feature that can be seen in the photomicrograph of the 100 x 200-mesh fraction of the Pittsburgh No. 8 sample (Figure 3.9) is a typical pyrite vein (area marked 3) characteristic of epigenetic growth (9). This growth is thought to occur due to the migration of solutions containing mineral-forming ions through coal fractures. It can be clearly seen from a magnified image of the pyrite veins, Figure 3.17, that the individual grains of pyrite comprising

Figure 3.8 - SEM Micrograph of a 100 x 200 mesh sample of Pittsburgh No. 8 coal.

Figure 3.9 - SEM Micrograph of a 100 x 200 mesh sample of Illinois No. 6 coal.

Figure 3.10 - SEM Micrograph of a 100 x 200-mesh sample of Upper Freeport PA coal.

Figure 3.11 - SEM Micrograph of a minus 200-mesh sample of Pittsburgh No. 8 coal.

Figure 3.12 - SEM Micrograph of a minus 200-mesh sample of Illinois No. 6 coal.

Figure 3.13 - SEM Micrograph of a minus 200-mesh sample of Upper Freeport PA coal.

Figure 3.14 - X-ray fluorescence spectra of iron sulfide particles in a minus 200-mesh sample of Pittsburgh No. 8 sample.

Figure 3.15 - X-ray fluorescence spectra of iron sulfide particles in a minus 200-mesh sample of Illinois No. 6 sample.

Figure 3.16 - X-ray fluorescence spectra of iron sulfide particles in a minus 200-mesh sample of Upper Freeport PA sample.

Figure 3.17 - SEM Micrograph of a Pittsburgh No. 8 coal particle with an epigenetic phase of pyrite in the form of veins.

Table 3.5 - Pyrite association and distribution (in volume percent) in the three base coals comminuted to minus 200-mesh.

	<u>Illinois No. 6</u>	<u>Pittsburgh No. 8</u>	<u>Upper Freeport PA</u>
(A) <u>Liberation of pyrite</u>			
Locked	11.7	38.4	14.2
Semilocked	11.7	22.4	15.4
Free	76.6	39.2	70.4
(B) <u>Distribution of pyrite</u>			
150 x 75 μm	0.7	5.5	0.5
75 x 45	9.9	9.4	14.3
45 x 15	17.6	31.5	28.5
15 x 0	71.8	53.6	56.7
(C) <u>Distribution of locked pyrite</u>			
150 x 75 μm	0.0	0.5	0.0
75 x 45	0.0	3.7	0.0
45 x 15	6.1	22.0	4.2
15 x 0	93.9	73.8	95.8
(D) <u>Distribution of semilocked pyrite</u>			
150 x 75 μm	0.0	6.8	0.0
75 x 45	2.0	12.7	3.2
45 x 15	23.9	52.7	1.6
15 x 0	74.1	27.8	95.2
(E) <u>Distribution of free pyrite</u>			
150 x 75 μm	0.9	9.5	0.6
75 x 45	12.7	14.2	17.3
45 x 15	18.4	33.3	32.7
15 x 0	68.0	43.0	49.4

the veins are less than 5 microns in size. Some submicron grains can also be seen. In practical terms this means that it will be difficult to separate these pyrite grains by froth flotation because entrainment of the submicron pyrite particles into the pulp is difficult to prevent.

3.3.2 Optical Image Analysis

A liberation study of the pyritic sulfur, conducted at the University of Utah, involved counting 2500 points on duplicate pellets made up of coal comminuted to minus 200 mesh. The pyrite was categorized in sizes $\pm$ plus 75 μm , 75 x 45 μm , 45 x 15 μm and minus 15 μm for each of the three states defined as pyrite in free, semi-locked, and locked particles. The results, summarized in Table 3.5, show that the volume percentage of free pyrite is the highest in the

Illinois No. 6 coal sample (77%), closely followed by the Upper Freeport PA sample (70%). However, when the distribution of free pyrite by size (Table 3.5E) is compared, 68 percent of the pyrite in the Illinois No. 6 coal is smaller than 15 microns in size whereas only 49 percent of free pyrite in the Upper Freeport PA sample is less than 15 microns. As the fineness of the free pyrite poses considerable problems during its separation by flotation or any other physical separation method, Illinois No. 6 coal would be expected to present the greatest degree of difficulty for achieving significant reduction of pyritic sulfur. Pittsburgh No. 8 seam (Table 3.5A) coal has the lowest percentage (39%) of liberated pyrite. However, as over 57% of the liberated pyrite is in the plus 15 micron range, its separation appears to pose less of a problem compared to the other two coals. These results can therefore be viewed from two angles: one, the degree of liberation of pyrite and two, the relative size of the liberated pyrite. In terms of these two problematic characteristics, the three coals can be ranked as follows:

- (a) Percentage of liberated pyrite in the minus 200-mesh sample
Pittsburgh No. 8 < Upper Freeport PA < Illinois No. 6
- (b) Size of liberated pyrite
Illinois No. 6 < Upper Freeport PA < Pittsburgh No. 8

3.4 Petrographic Characterization

Samples for petrographic analysis were prepared at the University of Utah following the procedure described in ASTM D2797-72. Samples were comminuted and sized to 28 x 40 mesh and then made into duplicate one-inch diameter pellets using a hardener (epoxy) and black pigment under 4,000 to 5,000 psi for 3 to 5 seconds. The pressing process was repeated about fifteen times. The pellets were polished using #240, #400 and #600 polishing grits and the final polishing was done using a 30-micron metal-bonded diamond lap (disk) followed by a one-micron and then a 0.05-micron alumina slurry. The pellets were then cleaned in an ultrasonic bath containing distilled water for one minute between each step. The finished pellets were placed in a desiccator overnight before analysis. The samples were analyzed using a Leitz orthoplan microscope following the procedure described in ASTM 2799.

Table 3.6 - Maceral analysis of the three base coals.

<u>Maceral Group</u>	<u>MACERAL COMPOSITIONS (Volume %)</u>		
	<u>Illinois No. 6</u>	<u>Pittsburgh No. 8</u>	<u>U.Freeport PA</u>
Vitrinite	75.8	78.9	88.8
Liptinite	9.2	9.8	0.2
- Sporinite	4.4	8.5	0.2
- Cutinite	2.9	0.4	0.0
- Alginite	1.9	0.9	0.0
- Resinite	0.0	0.0	0.0
Inertinite	15.0	11.3	11.0
- Fusinite	1.1	1.5	4.0
- Semifusinite	4.4	3.5	1.7
- Macrinite	2.6	2.2	2.1
- Micrinite	1.4	0.6	0.5
- Inertodetrinite	4.6	3.1	2.4
- Schlerotinite	0.9	0.4	0.3

The macerals of coal are classified into vitrinite, liptinite (exinite), and inertinite groups. Normal incident illumination light was used to identify the vitrinite and inertinite macerals, and blue-light excitation was used for liptinite maceral analysis. The liptinite group is further divided into cutinite, alginite, resinite, sporinite, exsudatinite, suberinite, phlobaphinite, liptodetrinite, fluorinite, and bituminite. Based on size, shape and color, the inertinite group is divided into fusinite, semifusinite, macrinite, micrinite ($<5\ \mu\text{m}$), sclerotinite, and inertodetrinite ($<30\ \mu\text{m}$). The vitrinite group is gray-white, the inertinite group is bright white, and the liptinite is dark or black under normal incident light, but under blue-light it has different colors (yellow, pink, green, brown orange, etc.). The results of the maceral analysis show that the Illinois No. 6, the Pittsburgh No. 8 and the Upper Freeport PA coals have 76, 79 and 89 volume percent vitrinite content, respectively (Table 3.6).

3.5 Solubility Behavior of the Base Coals

Leaching experiments with the three base coals were carried out to determine the nature and concentration of dissolved metal ions. The importance of this study arises from the fact that precipitation of dissolved metal ions, due to changes in pH, on the coal surface can drastically modify the surface of coal and affect its flotation characteristics. For example, in a study by Celik and Somasundaran (10) on the effect of SnCl_2 addition on the electrokinetic and flotation

properties of coal, the authors noticed that depression of coal flotation with SnCl_2 occurred in the pH range where precipitation took place. They also suggested that the poor flotation response of coal observed above pH 12 could be due to the precipitation of hydroxides of multivalent cations such as calcium and iron.

Leaching experiments were conducted on ten gram samples of coal placed in a 150 ml beaker and combined with 90 ml of triply distilled water. The slurry was then stirred using a magnetic stirrer, inside a glove box maintained under argon. After the desired time interval the stirring was stopped, the beaker was removed from the glove box, and the slurry filtered using a Whatman 41 filter paper. Initial and final pH's of the slurry were noted in all experiments. The filtrate was then analyzed for metal cations using a Perkin-Elmer model 6500 inductively coupled plasma (ICP) spectrophotometer. Leaching experiments were carried out with both the minus 28 and the minus 200-mesh grinds of Pittsburgh No. 8 coal. However, no major differences in either the nature or the concentration of dissolved ions were found between the supernatants from the two size fractions, and therefore, the tests with Illinois No. 6 and Upper Freeport PA coals were conducted using only the minus 28-mesh grind.

Results of the leaching experiments conducted with the three base coals are summarized in Table 3.7 in terms of the suspension pH and the concentration of dissolved ions in the supernatant for Pittsburgh No. 8, Illinois No. 6, and Upper Freeport PA coals, respectively. It can be seen from these tables that the concentration of calcium ions is the highest in the supernatant of Illinois No. 6 suspension, followed by Pittsburgh No. 8 and Upper Freeport PA coal suspensions. Also, calcium ion dissolution reached equilibrium within thirty minutes in the case of Upper Freeport PA coal whereas with Illinois No. 6 coal equilibrium does not appear to have been reached even after two hours. In the case of Pittsburgh No. 8 coal, equilibrium is reached within thirty minutes with the minus 200-mesh sample, whereas with the minus 28-mesh sample there is a steady increase in the calcium ion concentration over 90 minutes and then a decrease during the final 30 minutes. The reason for the decrease in calcium after two hours is not clear. The high concentration of calcium ions in the supernatant of Illinois No. 6 suspension should be mainly due to calcite (CaCO_3) since attapulgite clay, the only other calcium-bearing mineral that is possibly present in this coal (DRIFT analysis did not eliminate the presence of

Table 3.7 - Results of Pittsburgh No. 8, Illinois No. 6, and Upper Freeport PA leaching tests in distilled water at a solids concentration of 10% at 27°C.

<u>Size</u>	<u>Time min.</u>	<u>pH</u>		<u>Concentration, ppm</u>				
		<u>Initial</u>	<u>Final</u>	<u>Ca</u>	<u>Mg</u>	<u>Fe</u>	<u>Al</u>	<u>Na</u>
Pittsburgh No. 8								
-28 M	30	3.6	3.7	48.5	10.1	24.3	-	-
	60		4.0	53.7	10.4	19.4	-	-
	90		4.6	61.3	10.4	22.3	-	-
	120		4.1	56.1	10.4	19.9	-	-
-200 M	15	4.5	4.9	61.5	9.5	17.5	-	-
	30		4.8	63.6	9.8	16.3	-	-
	60		4.6	62.7	10.3	10.2	-	-
Illinois No. 6								
-28 M	30	5.8	7.1	50.2	2.9	-	-	-
	60		7.9	63.8	4.3	-	-	-
	90		8.0	73.1	4.7	-	-	-
	120		8.1	81.3	4.5	-	-	-
Upper Freeport PA								
-28 M	30	4.5	4.5	8.3	1.0	~1.0	-	1.6
	60		4.7	7.6	0.9	~1.0	-	1.5
	90		4.4	8.2	1.1	~1.0	-	1.6
	120		4.5	8.3	0.9	~1.0	-	1.5

attapulgitite), is much less soluble than calcite. The solubility of calcite at pH 6 (initial pH of the Illinois No. 6 suspension) is 400 ppm and at pH 8 (the final suspension pH) is about 40 ppm (11), whereas that of attapulgitite is less than 10 ppm in the above pH range (12). The relatively high natural pH of Illinois No. 6 coal suspensions (natural pH = 8.0), compared to Pittsburgh No. 8 and Upper Freeport PA coal suspensions, suggests that the suspension pH of Illinois No. 6 sample is controlled by calcite dissolution since the natural pH of a calcite suspension has been measured in our laboratory to be around pH 9.

The source of calcium ions in the supernatant of Pittsburgh No. 8 sample is not apparent since no calcite was found in this coal at detectable levels by DRIFT. Also, the estimated calcium ion concentration at pH 4 (extrapolated from Vasudevan and Moudgil's data) from attapulgitite is only about 8 ppm. However, the following analysis shows that calcite may be the source of calcium in the Pittsburgh No. 8 supernatant. An examination of calcite solubility data (11) shows that the solubility of calcite at pH 4.5 (natural pH of Pittsburgh No. 8 suspension) is about 20 g/l. Since the concentration of the solids in the slurry is 10 wt% and the ash content of

Pittsburgh No. 8 sample is 12 wt%, the concentration of mineral matter in the suspension should be 1.2 wt%. This means that at pH 4 all the calcite that is possibly present in Pittsburgh No. 8 coal would have dissolved into solution. Based on the dissolved calcium ion concentration, the calcite content of Pittsburgh No. 8 coal should be about 0.06 wt%. This is below the detectable limit of NAA which is about 0.1 wt%; therefore, NAA results would detect no calcium in the Pittsburgh No. 8 coal. Also, DRIFT is probably not sensitive enough to detect such low calcite levels as is apparent from the size of the calcite peak of Illinois No. 6 in which the calcite content is roughly 30 times that of the Pittsburgh No. 8 sample. The source of calcium in the supernatant of Upper Freeport PA coal suspensions may be due to dissolution of calcite or attapulgite.

The magnesium content of the supernatant of the three coals exhibits a slightly different trend from that of calcium. The magnesium ion concentration is higher in the supernatant of Pittsburgh No. 8 suspension followed by Illinois No. 6 coal and then Upper Freeport PA coal. The source of magnesium ions is probably from either clays or the magnesium-bearing carbonate mineral, dolomite. Dolomite was not identified in the DRIFT study of ash from the three base coals, but may be present in small quantities. Common clay minerals that contain major amounts of magnesium are attapulgite (10 wt% MgO) and montmorillonite (3% MgO) (13). The clay mineral clearly identified in our DRIFT study, kaolinite, contains less than 0.1 wt% MgO on average. As discussed earlier, although our DRIFT analysis did not show the presence of montmorillonite, it did not eliminate the presence of attapulgite. Therefore, it appears that attapulgite is the possible source of magnesium in the supernatant of the three coals.

The iron content in the supernatant of Pittsburgh No. 8 suspension is higher than that found in the supernatant of either Upper Freeport PA coal or Illinois No. 6 coal. The source of the soluble iron content is most likely oxidized pyrite. Although the sulfate content of both the Pittsburgh No. 8 and Upper Freeport PA coals are almost equal, there is evidence that the pyrite in Pittsburgh No. 8 coal oxidizes readily at room temperature, even when stored under argon. In this case, the source of oxygen may be pores or dissolved oxygen in the leachate. Because of the pH dependence of calcite solubility, the oxidation of pyrite may also affect the calcium ion concentrations due to the acid which is a product of pyrite oxidation.

Leaching studies conducted with the three base coals provide an indication of the mineralogical complexity of the three base coals. Low dissolved ion concentrations in the supernatant of Upper Freeport PA coal suggest the absence of highly soluble salt-type minerals in this coal. Furthermore, it appears that the natural pH of the Illinois No. 6 suspension is determined by calcite.

3.6 References

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4.0 COAL WASHABILITY STUDIES

In order to determine the liberation and the coal/ash/pyritic sulfur separation characteristics of each of the project coals, washability tests were conducted on each of the three base coals, Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA, and also on the three additional coal samples, Kentucky No. 9, Upper Freeport WV and the Wyodak seam from Wyoming. This work was performed at various commercial testing laboratories, under the supervision of Praxis Engineers, Inc. as a part of Task 2 "Coal Procurement and Weathering". Four different laboratories, located in the general area from which the samples were collected, were selected for this work with two laboratories each conducting the sink float tests analyses. Laboratory work for the base coal was carried out at a minimum of two laboratories. Table 4.1 identifies the commercial laboratories used for sample preparation and basic washability testing as well as QA/QC work. All sink/float products were analyzed for their calorific value and their ash, pyritic sulfur and total sulfur contents.

The material used for the washability and liberation studies was a part of the large sample that had been collected in November 1988 for research studies, as discussed in Section 2.0. Each sample was subjected to washability tests at the sizes and specific gravities given below:

Designated sizes: 4-inch x 0, 1-1/2-inch x 0, 1/8-inch x 0
28-mesh x 0 and 200-mesh x 0

Specific gravity: 1.30, 1.35, 1.40, 1.60 and 1.80

Table 4.1 - Commercial laboratories used for washability testing of various coals.

<u>Laboratory</u>	<u>Basic Tests</u>
Commercial Testing and Engineering Henderson, KY	Illinois No. 6 Upper Freeport PA
Geochemical Co., Testing Somerset, PA	Pittsburgh No. 8 Kentucky No. 9 Illinois No. 6
Gould Energy - Warner Labs Div. Cresson, PA	Upper Freeport PA Upper Freeport WV Illinois No. 6
Core Labs Inc. Casper, WY	Wyodak WY Pittsburgh No. 8

Figure 4.1 - Coal washability sample splitting and processing method.

4.1 Sample Preparation and Sink-float Test Procedure

Five 55-gallon drums of coal, containing a total sample weight of approximately 2000 - 2150 lb, were used to conduct the washability test for each of the project coals, in accordance with ASTM procedures (ASTM D4371).

The sink-float testing was done on the screened size fractions, namely 4-inch x 1-1/2-inch, 1-1/2-inch x 1/8-inch, 1/8-inch x 28-mesh, 28-mesh x 200-mesh, and 200-mesh x 0; and the results are reported in the format requested by DOE, that is 4-inch x 0, 1-1/2-inch x 0, 1/8-inch x 0, 28-mesh x 0, and 200-mesh x 0. Figure 4.1 summarizes the procedure followed for preparing the gross sample for washability and liberation studies. Table 4.2 gives the crushing topsizes used and indicates the size fractions generated for the washability analyses. A total of 15 size fractions was produced, each of which was subjected to sink-float testing at 5 specific gravity levels, thus generating a total of 90 size-gravity fractions for each coal.

Table 4.2 - Sink-float tests conducted on various size fractions for each coal.

Size Fraction	Original Sample	COAL SIZE REDUCED TO PASS				Total Samples
		1 1/2"	1/8"	28 M	200 M	
+1-1/2"	1	-	-	-	-	1
1-1/2" x 1/8"	1	1	-	-	-	2
1/8" x 28 M	1	1	1	-	-	3
28 x 200 M	1	1	1	1	-	4
200 M x 0	1	1	1	1	1	5
Total	5	4	3	2	1	15

The sink-float tests for particles 1/8-inch in size and coarser were performed using a static method of separation in the organic heavy liquids whereas the size fractions 28-mesh and finer were separated using centrifuging techniques. The organic heavy liquids used for these tests were mixtures of certigrav and perchloroethylene for determinations made at specific gravities between 1.3 and 1.6. For specific gravities above 1.6, dibromomethane was used to obtain the desired density. The typical procedure followed for washability sample preparation and testing is outlined below.

- The original sample (4-inch x 0) weighing approximately 2000 lb, was air dried overnight, and split in half by the long piling method (U.S.B.M. TP133). One half of the sample was used as the original sample for washability testing at the five specific gravities, without any crushing. This sample was screened at 1-1/2 inch, 1/8-inch, 28-mesh, and 200-mesh and the individual fractions were then subjected to sink-float testing. The 1 1/2-inch and 1 1/2 x 1/8-inch fractions were floated in metal containers with 100-mesh wire basket inserts. The specific gravity of the medium was checked and balanced before the sample was floated. The float and sink materials were dried at 105°C after recovery, and weighed.
- The 1/8-inch x 0 portion of the original sample was riffled and one-fourth of the sample screened at 28-mesh and 200-mesh. The sink-float test for the 1/8-inch x 28-mesh material was conducted in 4000-ml glass separating funnels. The 28 x 200-mesh and 200-mesh x 0 fractions were sink-floated by a centrifuge sink-float procedure.
- The remaining original sample was screened at 1-1/2-inch, and all the plus 1-1/2-inch material was then crushed in a jaw crusher and screened. The entire sample was homogenized and riffled into two parts, one half used to obtain the washability data. The procedure for the preparing material for the washability tests with the minus 1 1/2-inch fraction was similar to the one discussed above for the 4-inch x 0 sample.
- About 400 lb of the sample riffled at 1 1/2-inch was used for washability testing for the remaining sizes. The material was screened at 1/8-inch and all plus 1/8-inch material was crushed and homogenized and then riffled into four aliquots. One-quarter was used for conducting the sink-float tests with the 28-mesh and 200-mesh fractions. One of the

1/8-inch sample fractions is screened at 28-mesh and 200-mesh. The 1/8-inch x 28 mesh fraction was subjected to sink-float separation in a glass funnel and the 28 x 200-mesh and 200-mesh x 0 was appropriately centrifuged. Approximately 100 lb of the minus 1/8-inch material was saved for future use.

- Approximately 120 lb coal from the minus 1/8-inch sample was riffled and a fourth (about 30-lb) of the sample was then stage-comminuted to pass 28-mesh. This 28-mesh x 0 sample was then riffled four ways, with one quarter being used for the washability tests at 28-mesh x 0 size. One quarter was used for the next stage of washability testing at 200-mesh x 0 size as described below and another quarter was inerted, packed in plastic bags, and saved for future use.
- One of the quarters from the minus 28-mesh sample was screened at 200-mesh, followed by comminuting the plus 200-mesh material to pass 200 mesh. This material was then riffled into four samples, which were inerted, packed, and stored for use in the centrifuge sink-float testing.
- Following the sink-float separation, a portion of coal in each size-gravity fraction was riffled and its size reduced in stages to prepare samples for analysis. The analytical work consisting of ash, calorific value, total sulfur, and pyritic sulfur analyses was performed using ASTM standard procedures D3174, D2015, D3177 and D2492 respectively.

One means to check whether the heavy-liquid testing procedure has been carried out correctly is to plot the weight of float material at a given density for coals as a function of topsize. As the coal is crushed finer, increased liberation should result in an increase in the amount of material floating at a given specific gravity. The initial washability tests on Illinois No. 6 coal showed a gradual increase in the weight of float material as the particle size was reduced; but with the 28 mesh and 200 mesh material, the amount of float material dropped sharply. This abnormal behavior was attributed to effects resulting from a combination of methodology and the high clay content of Illinois No. 6 coal. Repeat tests gave results more in line with expected behavior. Figure 4.2 presents such plots for the amount of material floating at various topsizes on liquid at specific gravity 1.3. The trend is that which would be expected, namely as the coal is reduced finer in size, the amount of float material increases. Illinois No. 6 coal exhibits somewhat reduced reading of float material at 200 mesh, probably due to the clay problem.

4.2 Washability Results and Data Analysis

The results from the sink-float tests performed on these coals were analyzed to investigate the liberation potential of the coal and are presented in several different ways to illustrate the effect of the degree of comminution on liberation. The ash, total sulfur, pyritic sulfur, and

Figure 4.2 - Fraction of float material at 1.3 specific gravity for the three base coals comminuted to various designated sizes.

percent pyritic sulfur rejection achieved at each topsize have been compared with the percent Btu recoveries. The data analysis also compared the percent pyritic sulfur rejection with the corresponding values of Btu recovery to assess how well the initial project objective of achieving 90% pyritic sulfur rejection at 90% Btu recovery might be met. Also comparisons were made between the pyritic sulfur rejection and corresponding values of the percent Btu recovered. These comparisons enabled us to evaluate the results with respect to the overall project objective of achieving 90% pyritic sulfur rejection at 90% Btu recovery. The percent Btu recovery was computed from the cumulative washability data using the following equation:

$$\text{Percent Btu recovery} = W_i \frac{H_i}{H_t}$$

where W_i = Cumulative weight percent recovered at specific gravity i

H_t = Heating value (Btu/lb) for entire sample

H_i = Heating value (Btu/lb) of float material at specific gravity i

Pyritic sulfur rejection was calculated from the cumulative data using the following relation:

$$\text{Pyritic sulfur rejection, \%} = 100\% - W_i \frac{PS_i}{PS_t}$$

where W_i = Cumulative weight percent recovered at specific gravity i .

PS_t = Pyritic sulfur (percent) in the entire sample,

PS_i = Pyritic sulfur (percent) in the float material at specific gravity i .

The results of these calculations were plotted to enable ready comparison with the overall project objective of achieving 90% pyritic sulfur rejection at 90% Btu recovery.

In the subsections that follow, the results of the washability tests and discussion are provided for each of the base coals tested under this project. The raw data are presented in the Appendix.

4.2.1 Illinois No. 6 Sample Washability Results

Figure 4.3 presents the cumulative weight percent of material floated at various specific gravity levels for the Illinois No. 6 coal that shows that there is a gradual increase in the weight of the float material with increasing specific gravity up to 1/8-inch topsize. As already stated, the weight of float material at 28-mesh and 200-mesh topsize at 1.30 specific gravity decreased drastically in the original washability tests, so only the results of the repeated tests are presented here. Though these results are very consistent in ash and total sulfur values, the weight of the float material at 1.3 specific gravity was still low (22.1% at 28 mesh and 34.5% at 200 mesh). The drop in the weight of float material at 1.3 specific gravity cannot be readily explained. This may be the result of clay contamination of the coal and/or of the separation of this coal at finer sizes. Interestingly, particularly for this coal, a relatively high recovery of float material at 1.4 specific gravity (58.7% for 28 mesh and 65.1% for 200 mesh) was obtained, which tends to confirm the problem of separation at a low specific gravity of 1.3. This will be discussed in greater detail under the QA/QC section.

Figure 4.4 presents the percent Btu recovery as a function of the cumulative percent ash for each size of coal being tested. As can be seen from the plot, the 4-inch x 0 sample exhibits an ash content of approximately 10% at a 90% Btu recovery level, but after grinding the coal to

Figure 4.3 - Cumulative weight percent of float material (yield) at various top sizes of Illinois No. 6 as a fraction of specific gravity.

Figure 4.4 - Btu recovery of the cumulative float product as a function of the ash content for Illinois No. 6 coal at various top sizes.

Figure 4.5 - Btu recovery of the cumulative float product as a function of the pyritic sulfur content for Illinois No. 6 coal at various sizes.

200 mesh, the coal contains only 7.0% ash at the 90% Btu recovery level. Thus, a 30 percent reduction in ash content can be achieved by reducing the tosize from 4-inches to 200 mesh at the corresponding of 90% Btu recovery.

Pyritic sulfur liberation relates to the degree of separation of pyrite that can be achieved from coal species. Sink-float results are a direct indicator of the maximum achievable rejection of pyritic sulfur. Figure 4.5 shows the relation of the cumulative pyritic sulfur content of the various size fractions with the Btu recovery. This provides an estimate of the pyrite liberation potential of this coal. The pyritic sulfur content is reduced from about 1.25% for the 4-inch x 0 sample, to 0.97% for the 1/8-inch sample, and to 0.36% for the 200 mesh tosize sample, all at the 90% Btu recovery level.

Figure 4.6 depicts the percent Btu recovery as a function of the pyritic sulfur rejection for Illinois No. 6 coal comminuted to the designated tosizes. This figure, which contains the information most pertinent to this study, shows the percent pyritic sulfur rejected in relation to the percent Btu recovered. At a 4-inch tosize, only about 60% of the pyritic sulfur was rejected

Figure 4.6 - Btu recovery of the cumulative float product as a function of pyritic sulfur rejection for Illinois No. 6 coal at various sizes.

at 90% Btu recovery. Pyritic sulfur rejection increased to almost 72% by crushing the material to 1/8-inch topsize, and to about 90% by grinding the coal to minus 200 mesh. From this figure, it would appear that Illinois No. 6 seam coal must be comminuted nominally to 200 mesh or finer in order to achieve 90% pyritic sulfur separation if the Btu recovery is to be maintained at 90 percent. Details of the washability results for this coal can be found in Topical Report No. 3 submitted to DOE (1).

4.2.2 Pittsburgh No. 8 Sample Washability Results

Figure 4.7 shows the cumulative weight percent of material floated versus specific gravity for Pittsburgh No. 8 coal. There is a gradual increase in the weight of the float material with increasing specific gravity. The weight of the float material at 1.3 specific gravity increased from 18.3% at 4-inch topsize to 46.4% at 1/8-inch topsize. When the coal is further reduced to minus 200-mesh, the weight of the float material increased to 53.9%. This shows there is a gradual liberation of mineral matter as the size of the coal is decreased, as expected. Apparently no separation problems were encountered with this coal as compared to Illinois No. 6 coal.

Figure 4.7 - Cumulative weight percent float (yield) at various specific gravities for Pittsburgh No. 8 coal at various top sizes.

Figure 4.8 presents the percent Btu recovery as a function of the cumulative percent ash for each top size. The minus 4-inch sample shows an ash content of approximately 6.5% at a 90% Btu recovery level. When the top size is reduced to 1/8-inch, the ash level drops to approximately 5% and to about 3.5% when the top size is further reduced to 200 mesh, all at the 90% Btu recovery level. Thus, an almost 50% reduction of the ash content is achieved by reducing the top size from 4-inch to 200 mesh.

Figure 4.9 depicts the percent Btu recovery as a function of the cumulative pyritic sulfur content of the various size fractions, providing an estimate of the pyrite liberation potential of this coal. The pyritic sulfur content is reduced from about 2.1% at the 4-inch top size to 1.4% for the minus 1/8-inch sample, and to approximately 0.2% when coal had been ground to minus 200-mesh, all at 90% Btu recovery.

Figure 4.10 depicts the percent Btu recovery as a function of pyritic sulfur rejection for Pittsburgh No. 8 coal comminuted to the designated top sizes. At a 4-inch top size, about 40% of the pyritic sulfur was rejected at 90% Btu recovery. Pyritic sulfur rejection can be increased

Figure 4.8 - Btu recovery of the cumulative float product as a function of the ash content for Pittsburgh No. 8 coal.

Figure 4.9 - Btu recovery of the cumulative float product as a function of the pyritic sulfur content for Pittsburgh No. 8 coal at various topsizes.

Figure 4.10 - Btu recovery of the cumulative float product as a function of pyritic sulfur rejection for Pittsburgh No. 8 coal at various topsizes.

to almost 60% by crushing the coal below a 1/8-inch in size, and to over 90% by reducing the topsize to 200 mesh. It appears that, at least ideally, in a true gravity separation the Pittsburgh No. 8 seam coal should be reduced to at least 200 mesh in order to achieve 90% pyritic sulfur rejection while maintaining at least 90% Btu recovery. Details of the washability results for this coal can be found in Topical Report No. 1 submitted to DOE (2).

4.2.3 Upper Freeport PA Sample Washability Results

Figure 4.11 presents the cumulative weight percent of Upper Freeport PA coal that floats as a function of the specific gravity of the liquid. With this coal also there is a gradual increase in the weight of the material floated as the coal is decreased in size. The weight of the float product at 1.3 specific gravity increased from 34.3% at 4-inch topsize to 42.3% at 1/8-inch. By reducing the coal to minus 200 mesh in size, the weight of the float material at 1.3 specific gravity increased to 54.9%. This shows the gradual liberation of mineral matter as the sample is ground to progressively finer size.

Figure 4.11 - Cumulative weight percent float (yield) at various specific gravities for Upper Freeport PA coal.

The overall liberation trend can be seen from the plots given in Figure 4.12, which presents the Btu recovery as a function of the cumulative percent ash for samples at various topsizes. The 4-inch x 0 float material has an ash content of approximately 8% at a 90% Btu recovery level. When crushed to minus 1/8-inch, the ash content of the coal can be reduced to approximately 7.2% and to about 3.8% at 200 mesh, both at the 90% Btu recovery level. Thus, almost a 50% reduction in ash content can be achieved by reducing the coal from 4 inches to 200 mesh in size.

Figure 4.13 shows the relationship between the percent Btu recovery and the pyritic sulfur content of the float product for the various size fractions. The pyritic sulfur content is reduced from 1.54% in the feed at 100% Btu content to about 0.85% at a topsize of 4-inch, to 0.65% at 1/8-inch topsize, and to approximately 0.1% at 200-mesh topsize, all at 90% Btu recovery.

Figure 4.14 presents the relationship between Btu recovery and pyritic sulfur rejection for Upper Freeport PA coal comminuted to the designated topsizes. At a topsize of 4-inches, about 52% of the pyritic sulfur was rejected at 90% Btu recovery. At 90% Btu recovery, pyritic sulfur

Figure 4.12 - Btu recovery of the cumulative float product as a function of the ash content for Upper Freeport PA coal.

Figure 4.13 - Btu recovery of the cumulative float product as a function of the pyritic sulfur content for Upper Freeport PA coal.

Figure 4.14 - Btu recovery of the cumulative float product as a function of pyritic sulfur rejection for Upper Freeport PA coal.

rejection can be increased to almost 65% by crushing the coal to minus 1/8-inch, and to over 94% by reducing the topsize to 200 mesh. The liberation potential of this coal is the highest of the three base coals. Details of the washability results for this coal can be found in Topical Report No. 2 submitted to DOE (3).

4.3 Washability QA/QC

The Quality Assurance/Quality Control program for the washability studies, devised in accordance with the Project Work Plan, was initiated to check the reproducibility and variability in the analytical results and their significance. Since the base washability tests were conducted by four different commercial laboratories, implementation of the QA/QC program was even more relevant to this task.

Upon completion of the original washability tests, a 1-lb sample of the originally prepared minus 200-mesh feed material was sent to a different laboratory for washability cross-checking in a round-robin fashion. The sample was subjected to sink/float analysis using the same centrifuging technique. Each gravity fraction was then analyzed for ash, total sulfur, and pyritic

sulfur. These washability QA/QC results were then compared with the original washability test results on the minus 200-mesh sample for each coal. In cases where there were significant differences between the two sets of results, the tests were repeated.

To conduct a QA/QC analysis on the analytical work, three samples from different size-gravity fractions were sent to a different laboratory for cross-checking. In addition, 10 pulverized samples from each base coal from the minus 28-mesh and minus 200-mesh topsize fractions were sent to the University of California at Berkeley to check the ash, total sulfur, and pyritic sulfur analyses.

Also, the washability data for all the designated topsizes were analyzed for internal self-consistency as a part of the QA/QC program. Check analyses were conducted on selected samples and data analysis carried out in which some discrepancies in the 200 mesh x 0 washabilities were observed with the Illinois No. 6 coal discussed later and the results were discussed with the laboratory where the sink/float centrifuging work was done. Subsequently, the 28-mesh and 200-mesh centrifuge sink-float tests for which the results appeared erroneous were repeated and a revised topical report was issued containing the new data. The method of evaluation and results are discussed further in this section.

4.3.1 Washability Data Evaluation Criteria

A careful review of the results was carried out to locate any errors in experimental techniques, thus identifying the tests which need to be repeated. In this study, the following four evaluation criteria were used.

- (a) Weight percent of 200 mesh x 0 material - The weight of the minus 200-mesh material in the samples that had been crushed to the designated top sizes was compared. An erratic change in the weight percent of the size fraction under consideration is indicative that a loss in fines may have occurred during sample preparation.
- (b) Analysis of the samples - A comparison of the analysis of the total ash, total sulfur, pyritic sulfur and calorific value was conducted, which indicates the accuracy of the sample splitting done during preparation of the washability sample.
- (c) Float products at 1.3 specific gravity - Based on the assumption that size reduction leads to higher liberation, the float products at 1.3 specific gravity should generally show a steady increase or levelling off. Therefore, comparisons of the amount of float material at 1.3 specific gravity were used to evaluate the consistency of the results.

Table 4.3 - Statistical analysis of composite values of ash, total and pyritic sulfur and calorific values for Illinois No. 6 sample.

Composite Size	Ash %	Tot S %	Pyr S %	Btu/lb	200 M fines %
4" x 0	16.4	4.94	2.95	11,850	0.3
1 1/2" x 0	15.9	4.87	2.90	11,800	0.2
1/8" x 0	16.1	4.86	2.97	11,750	4.2
28 M x 0	--	--	--	--	--
200 M x 0	15.7	4.82	3.02	NA	100
200 M x 0*	16.1	4.97	2.79	11,610	
Mean	16.0	4.87	2.96	11,800	
Std. Dev.	0.28	0.04	0.04	50	
SD/Mean	0.017	0.009	0.015	0.004	

*QA/QC sample testing carried out by Geochemical Testing was excluded from the statistical analysis.

- (d) Float products at 1.6 specific gravity - Considering the difficulty of separation at 1.3 specific gravity, comparisons were also made for the float material recovered at 1.6 specific gravity.

Based on this evaluation criteria, the problems observed were limited to the fine coal washability tests where the centrifuge technique was used, and mainly only with Illinois No. 6 coal. A discussion for each coal is provided below.

4.3.2 Data Analysis for Illinois No. 6 Coal

The sink-float test work for the Illinois No. 6 coal was done at Commercial Testing and Engineering Co. at their Henderson, Kentucky laboratory. Preliminary data evaluation indicated internal inconsistencies in the fine coal (minus 28-mesh and 200-mesh) sink-float tests. These tests were repeated twice, once using the procedure normally followed by the laboratory and the second using the DOE-developed procedure. For both sets of results, the weights of float fractions at 1.3 specific gravity were unusually low as compared to the test results run on this coal in other laboratories, as will be discussed later. Consequently, the original washability results on the fine (28-mesh and 200-mesh) Illinois No. 6 samples were discarded and replaced by the new results which are discussed here.

A comparison of the ash and sulfur analyses of the subsamples reduced to pass the designated top sizes can be made from the results summarized in Table 4.3. The ash and total

Table 4.4 - Comparison of the float products at 1.3 and 1.6 specific gravity for the Illinois No. 6 sample.

Size	FLOAT AT 1.3				FLOAT AT 1.6			
	Wt %	Ash %	Tot S %	Btu/lb	Wt %	Ash %	Tot S %	Btu/lb
4" x 0	20.7	4.4	2.98	13,800	87.2	10.7	3.71	12,780
1 1/2" x 0	22.0	4.1	2.92	13,730	87.5	10.4	3.59	12,690
1/8" x 0	34.5	3.3	2.75	13,820	81.5	8.6	3.14	13,010
28 M x 0	--	--	--	--	--	--	--	--
200 M x 0	34.5	2.8	2.38	--	81.2	6.3	2.41	--
200 M x 0*	23.6	2.2	2.71	13,990	76.4	5.4	2.67	13,490

*Results of QA/QC Test done at Geochemical Testing

sulfur values are within an acceptable limit for all the subsamples resulting in a low value for the standard deviation for ash and for the total-sulfur analyses. The values of these parameters for the QA/QC sample run in a different laboratory are also very close to the mean values obtained for the base tests.

The weight of the minus 200-mesh fines given in Table 4.3 increases as the top size is reduced. It is interesting to note that for the minus 4-inch sample, the minus 200-mesh fraction is only 0.3%, indicating low friability of this coal.

A comparison of the float products at 1.3 specific gravity (Table 4.4 and Figure 4.2) indicated that there is a steady increase in the 1.3 specific gravity fraction for sub-samples crushed to 1/8-inch, followed by a drop for the sample that had been comminuted to 28-mesh. However, for the sample comminuted to minus 200 mesh, the float fraction at 1.3 specific gravity increased to 34.5%. The weight of the float material at 1.6 specific gravity exhibits a reasonably smooth trend, that is, observed for the other coals (because of the insensitivity due to high percentage of float material). The results of the QA/QC tests conducted in another laboratory (Geochemical Testing) on the 200-mesh x 0 grind indicated that the weight of the float product at 1.3 specific gravity was 23.6%, which is lower than expected.

It is believed that this problem may be due to the tendency of this coal to agglomerate at fine sizes because of the presence of clays. Therefore, a comparison was made at the next level of specific gravity, namely 1.4. As may be seen in Table 4.5, the weight of float material for the 200-mesh grind at a specific gravity of 1.4 was 65.1%. Considering that the problem of

Table 4.5 - Comparison of float product at 1.4 specific gravity for Illinois No. 6 sample.

<u>Size</u>	<u>Wt %</u>	<u>Ash %</u>	<u>Tot S %</u>	<u>Pyr S %</u>	<u>Btu/lb</u>
4"	75.3	9.10	3.44	1.18	13,052
1-1/2"	73.3	8.40	3.21	0.97	13,023
1/8"	62.6	5.72	2.93	0.65	13,450
28 M x 0	--	--	--	--	NA
200 M x 0	65.1	4.71	2.38	0.19	13,910
200 M x 0*	31.7	2.80	2.66	0.12	

*Results of QA/QC test done at Geochemical Testing

separation of fines is less severe as the specific gravity of separation is increased, a comparison at 1.4 specific gravity appears to be somewhat more meaningful due to the reduced experimental error at this gravity for this coal.

4.3.3 Data Analysis for Pittsburgh No. 8 Coal

The washability of the Pittsburgh No. 8 sample was performed by Geochemical Testing, Somerset, PA. Our data evaluation indicates that the results obtained for the Pittsburgh No. 8 sample are internally consistent. In making this conclusion, composite values of the ash, total sulfur, pyritic sulfur and calorific value were compared for samples reduced to designated top sizes. A statistical analysis of the results given in Table 4.6 indicates that the sub-samples, as prepared from the main sample, are highly consistent as indicated by the low values of standard deviation and variance for all the parameters analyzed. As can be seen, the ash content of all subsamples lies between 11.5% and 12.0%, which is considered excellent. The amount of minus 200-mesh material given in Table 4.6 show a gradual increase in the weight of float fraction as the topline is reduced, which is expected.

The next parameter evaluated was the weight percent of float material at 1.3 specific gravity for each of the subsamples. The 1.3 float data for Pittsburgh No. 8 sample given in Table 4.7 indicate a gradual increase in the weight of the float material. The results, plotted in Figure 4.2 indicate that the weight of 1.3 float products increases somewhat rapidly initially with reduction of top size, but becomes asymptotic at top sizes finer than about 1/8-inch top size.

Table 4.6 - Statistical analysis of the composite values of ash, total and pyritic sulfur and calorific values for Pittsburgh No. 8 sample.

<u>Composite Size</u>	<u>Ash %</u>	<u>Tot S %</u>	<u>Pyr S %</u>	<u>Btu/lb</u>	<u>200 M fines %</u>
4" x 0	11.5	3.90	2.96	13,000	1.8
1-1/2" x 0	12.0	3.91	2.94	12,950	2.8
1/8" x 0	12.0	3.93	2.78	12,950	5.5
28 M x 0	12.0	3.89	2.77	12,850	24.0
200 M x 0	12.0	3.90	2.78	12,750	100.0
200 M x 0*	12.3	4.34	2.80	12,720	100.0
Mean	11.9	3.91	2.85	12,900	
Std. Dev.	0.22	0.01	0.08	91	
SD/Mean	0.018	0.003	0.029	0.007	
95% CI(Mean)	0.27	0.013	0.102	112	

*QA/QC analysis carried out by Core Laboratories Inc.: excluded from standard deviation.

The total sulfur and ash content of the 1.3 float products also decreases with decreasing top size with a corresponding increase in the calorific value. The increase in the calorific value for the fine 200 mesh size may be primarily due to the liberation of hydrogen-rich macerals which tend to concentrate in the low gravity fraction.

While the results for the 1.3 float material appear to be very consistent, the weight of the float material at 1.6 specific gravity showed a minor drop with decreasing size. The same trend is observed for the floats of coals at all other specific gravities tested. The ash of the 1.6 floats decreases with the decreasing top size which in turn results in a corresponding increase in the calorific value.

Table 4.7 - Comparison of the float products at 1.3 and 1.6 specific gravity for Pittsburgh No. 8 sample.

<u>Size</u>	<u>FLOAT AT 1.3</u>				<u>FLOAT AT 1.6</u>			
	<u>Wt %</u>	<u>Ash %</u>	<u>Tot S %</u>	<u>Btu/lb</u>	<u>Wt %</u>	<u>Ash %</u>	<u>Tot S %</u>	<u>Btu/lb</u>
4" x 0	18.3	4.34	2.12	14,200	91.6	7.48	3.33	13,700
1 1/2" x 0	31.8	4.57	2.28	14,250	90.4	7.18	3.24	13,800
1/8" x 0	46.4	3.34	1.85	14,400	88.8	6.28	2.79	13,900
28 M x 0	51.1	2.66	1.59	14,400	87.3	5.29	2.21	14,000
200 M x 0	53.9	2.08	1.35	14,500	86.8	4.26	1.48	14,100
200 M x 0*	70.4	2.21	1.41	14,420	87.8	4.81	1.64	14,010

*Results of QA/QC tests carried out by Core laboratories Inc.

Table 4.8 - Statistical analysis of composite values of ash, total and pyritic sulfur and calorific values for Upper Freeport PA.

Composite Size	Ash %	Tot S %	Pyr S %	Btu/lb	200 M fines %
4" x 0	13.0	2.15	1.54	13,450	1.76
1 1/2" x 0	12.8	2.16	1.44	13,500	1.70
1/8" x 0	12.2	2.25	1.58	13,600	5.12
28 M x 0	12.4	2.19	1.34	13,400	21.36
200 M x 0	11.9	2.35	1.42	13,500	100.00
200 M x 0*	12.6	2.31	1.61	--	100.00
Mean	12.5	2.22	1.46	13,500	
Std. Dev.	0.39	0.08	0.09	71	
95% CI (Mean)	0.48	0.093	0.106	89	
SD/Mean	0.031	0.034	0.059	0.005	

*QA/QC Sample testing carried out by Commercial Testing Laboratory Co.: excluded from standard deviation computation.

4.3.4 Data Analysis for Upper Freeport PA Coal

Table 4.8 gives the statistical analysis of the composite washability samples comminuted to various designated top sizes for this coal. As shown in this table, the ash, total sulfur, pyritic sulfur, and calorific values fall within a close range indicating the consistency of the sample split by the laboratory. For example, the ash content is approximately within 0.5% of the mean value of 12.5% which is indicative of uniformity of splitting the head sample used for conducting the washability tests at each size.

Also, the results reported in Table 4.8 indicate that the increase in the weight of the minus 200-mesh fraction is consistent with the reduction of top size of the sample. The weight of floats at 1.3 specific gravity (Table 4.9 and Figure 4.2) increases steadily as the top size is reduced. The highest value was 54.9%, as obtained for the 200-mesh x 0 sample, with an ash content of 1.87% and a total sulfur content of 0.79%.

The yield at 1.6 specific gravity indicates a drop in the weight of the float fraction with reduction in top size, attributed possibly to the liberation of mineral matter of high specific gravity, which would result in an increase in the weight of material in the 1.7 and 1.8 gravity intervals. These results are nearly identical in trend to those obtained for the Pittsburgh No. 8 sample. Based on the foregoing analysis, it is our conclusion that the Upper Freeport PA washability data are internally consistent.

Table 4.9 - Comparison of the float products at 1.3 and 1.6 specific gravity for the Upper Freeport PA sample.

Size	FLOAT AT 1.3				FLOAT AT 1.6			
	Wt %	Ash %	Tot S %	Btu/lb	Wt %	Ash %	Tot S %	Btu/lb
4" x 0	34.3	3.62	1.23	15,050	91.2	9.16	1.50	14,100
1 1/2" x 0	35.1	3.71	1.20	15,050	91.2	8.98	1.49	14,150
1/8" x 0	42.3	3.29	1.12	15,200	89.9	8.07	1.31	14,350
28 M x 0	44.9	2.32	0.89	15,200	85.4	6.50	1.02	14,500
200 M x 0	54.9	1.87	0.79	15,250	81.0	3.75	0.82	14,600
200 M x 0*	47.7	2.00	0.81	NA	84.9	5.21	0.82	NA

*Results of QA/QC test done at Commercial Testing Laboratory Co.

4.3.5 QA/QC on the Analytical Work

As a part of QA/QC program of the samples used for washability and flotation research were analyzed for the proximate and ultimate analyses and compared. The analytical work was done at UCB on reference (save) samples used for the general research program. Table 4.10 provides comparison of the proximate analysis of a washability sample, and research samples prepared at Berkeley and Utah. The values of volatile matter, carbon ash and total sulfur for the three coals are very consistent in all the samples randomly selected for analysis.

For example, the acceptable reproducibility error for analysis alone, for samples containing 12% ash or higher is 1.0%, and for total sulfur over 2% the acceptable error is 0.2%. This is excluding the allowable variance due to the sample splitting.

Table 4.11 gives comparative results of the ultimate analysis for the randomly picked samples from three sources and analyzed at Berkeley. The results indicate very good consistency in all the cases except for the oxygen contents for the three coals which is higher than the variability of other elements.

A number of specific gravity fractions were randomly selected from the fine coal washability testing for cross checking the analytical work. The results of this work conducted at Berkeley are reported in Table 4.12. As may be seen, the analytical results from the original washability data are very close to the analyses performed under QA/QC at Berkeley.

Table 4.10 - Comparison of proximate and sulfur analyses* of base coals.

<u>Sample/Source</u>	PROXIMATE ANALYSIS (DRY BASIS)				
	Moisture %	V. Matter %	Carbon %	Ash %	Tot S %
<u>Illinois No. 6</u>					
Washability	6.34	36.1	46.0	17.9	5.81
Research (UU)	4.23	36.0	47.4	16.6	5.27
Research (UCB)	4.50	36.2	46.3	17.5	5.73
Mean		36.1	46.6	17.3	5.60
Std. Dev.		0.08	0.60	0.54	0.238
<u>Pittsburgh No. 8</u>					
Washability	1.89	35.1	52.6	12.3	4.15
Research (UU)	2.03	36.1	53.0	10.8	4.19
Research (UCB)	2.32	35.7	52.5	11.8	4.28
Mean		35.6	52.7	11.6	4.21
Std. Dev.		0.41	0.22	0.62	0.0 ^c 4
<u>Upper Freeport PA</u>					
Washability	0.82	25.4	62.1	12.5	2.29
Research (UU)	0.94	26.2	61.4	12.4	2.23
Research (UCB)	1.00	26.2	61.8	12.0	2.38
Mean		25.93	61.97	12.30	2.30
Std. Dev.		0.38	0.287	0.216	0.062

* The analytical work was done at Berkeley in the University of California

4.4 Fine Coal Washability Studies

The results of the washability tests conducted on the three base coals indicated that by gravity separation it is possible to obtain over 90% Btu recovery at 90% pyritic sulfur rejection for samples that had been ground to 200 mesh. The specific gravity of the separation liquid necessary to achieve this result falls between 1.55 and 1.60 for the three base coals. For the sizes investigated in the first set of tests, the liberation of pyritic sulfur is markedly less if the samples were coarser than 28 mesh. Based on those results, an added objective was to investigate the liberation behavior of pyrite from the three base coals at grinds of 100, 325 and 400 mesh and to re-evaluate the 200-mesh results. Therefore, an extensive fine washability study was undertaken.

Table 4.11 - Comparison of the elemental analyses* of the base coals.

<u>Sample/Source</u>	ELEMENTAL ANALYSIS (DRY BASIS)					
	<u>Moisture</u> <u>%</u>	<u>Carbon</u> <u>%</u>	<u>Hydrogen</u> <u>%</u>	<u>Nitrogen</u> <u>%</u>	<u>Sulfur</u> <u>%</u>	<u>Oxygen</u> <u>%</u>
<u>Illinois No. 6</u>						
Washability	6.34	63.9	5.29	1.01	5.81	6.07
Research (UU)	4.23	63.9	5.04	1.39	5.27	7.88
Research (UCB)	4.50	63.8	5.65	1.24	5.73	6.10
Mean		63.9	5.33	1.21	5.60	6.68
Std. Dev.		0.047	0.250	0.156	0.238	0.846
<u>Pittsburgh No. 8</u>						
Washability	1.89	71.0	5.01	1.23	4.15	6.29
Research (UU)	2.03	72.4	5.06	1.47	4.19	6.07
Research (UCB)	2.23	71.0	5.12	1.45	4.28	6.40
Mean		71.5	5.06	1.38	4.21	6.25
Std. Dev.		0.66	0.045	0.109	0.054	0.137
<u>Upper Freeport PA</u>						
Washability	0.82	76.1	4.76	1.34	2.29	3.00
Research (UU)	0.94	76.3	4.66	1.45	2.23	3.02
Research (UCB)	1.00	75.6	4.70	1.45	2.38	3.85
Mean		76.00	4.71	1.41	2.30	3.29
Std. Dev.		0.294	0.041	0.052	0.062	0.396

* The analytical work was done at Berkeley

Table 4.12 - Analysis of sink-float fractions preselected for testing.

<u>Composite</u> <u>Size</u>	<u>Size</u> <u>Fraction</u>	<u>Gravity</u> <u>Fraction</u>	<u>Ash</u> <u>Orig.</u>	<u>Ash</u> <u>QA/QC</u>	<u>Pyr S</u> <u>Orig.</u>	<u>Pyr S</u> <u>QA/QC</u>	<u>Tot S</u> <u>Orig.</u>	<u>Tot S</u> <u>QA/QC</u>
<u>Illinois No. 6</u>								
28 M x 0	28 x 200 M	1.60 x 1.80	35.8	35.8	4.27	5.98	5.78	6.19
28 M x 0	200 M x 0	1.40 x 1.60	11.7	11.7	0.77	0.69	2.54	2.42
28 M x 0	200 M x 0	+ 1.80	60.1	62.9	15.66	15.05	16.50	16.97
<u>Pittsburgh No. 8</u>								
28 M x 0	28 x 200 M	- 1.30	2.8	2.9	0.40	0.33	1.66	1.80
28 M x 0	28 x 200 M	1.40 x 1.60	17.7	17.4	4.66	4.43	5.78	6.60
28 M x 0	200 M x 0	1.35 x 1.40	4.8	4.7	0.36	0.34	1.40	1.66
<u>Upper Freeport Pa</u>								
28 M x 0	28 x 200 M	- 1.30	2.9	3.1	0.61	0.20	0.92	0.96
28 M x 0	200 M x 0	1.35 x 1.40	4.4	4.5	0.48	0.09	0.79	0.83
200 M x 0	200 M x 0	1.35 x 1.40	5.9	4.3	0.14	0.08	0.90	0.85

The coal used for the fine washability and liberation study was a part of the original coal samples collected at the onset of the project and used for the initial washability tests. These samples had been stored at the mine sites in 55-gallon drums which had been purged with argon when the samples were originally collected in November 1988. One 55-gallon drum of each of the three base coals, containing a total sample weight of approximately 450 pounds each, was homogenized and then crushed from 4-inch top size to pass 28 mesh in three stages. The minus 28-mesh was split into sub-samples prior to final comminuting to the four designated top sizes, namely, 95% passing 100 mesh, 200 mesh, 325 mesh and 400 mesh top size. The specific procedure used for preparing samples for a washability test involved the following steps:

1. The gross sample was air dried overnight and crushed to a nominal 3/4-inch top size. The sample was then riffled into four equal lots.
2. One aliquot from the above was crushed to pass 1/4-inch and riffled into four 25-30 lb subsamples.
3. One 30-lb bag was stage-comminuted to 100% passing 28 mesh and split into four 6-lb subsamples.
4. One 6-lb bag from the 28 mesh x 0 sample was then used to prepare one of the final washability samples by pulverizing it to pass 95% of the designated top size. During pulverization, the sample was periodically screened to remove fines and prevent the production of too much very fine undersize material.
5. Each pulverized sample was then split into four subsamples and stored under inert conditions. One sub-sample was used for centrifuge sink-float testing and the remaining samples were saved as reference samples.

4.4.1 Sink-float Test Procedure and Data Compilation

The sink-float tests on the fine coal samples were performed by Geochemical Testing Laboratory in Somerset, Pennsylvania, using centrifuging techniques in accordance with DOE's established procedure. The designated specific gravity levels were 1.3, 1.35, 1.4, 1.6 and 1.8, identical to those used for the original washability tests.

The procedures used to perform the sink-float testing were standardized. This included the make-up of the liquids used for the actual testing and the addition of a dispersing agent as well as the actual separation procedure and product analysis. The procedures used were the following:

1. The sample was oven-dried at 40°C until constant weight was achieved.
2. The gravity of the organic heavy liquid used for the separation was adjusted by mixing Certigrav (specific gravity 1.20) with methylene bromide (specific gravity 2.5).
3. A dispersing agent, Aerosol OT-100, was added to each of the solutions (10 grams/liter). The solution was then mixed thoroughly and its gravity checked. The gravity was then readjusted if necessary.
4. The prepared solution was then placed in a 500-ml glass flask and the coal sample added such that the solid-liquid ratio did not exceed 1:2.
5. Each glass flask was placed in a sonic cleaner and was treated ultrasonically for 90 seconds.
6. The sample was then centrifuged for at least 20 minutes at 1500 rpm.
7. The sink fraction was then dried at 40°C before proceeding with the next higher gravity, and the entire procedure from step 4 was repeated until all gravity separations were completed. The float products were removed, filtered, dried and weighed.
8. All the sink-float gravity fractions corresponding to each gravity were analyzed for ash, calorific value, total sulfur and pyritic sulfur following procedures specified in ASTM D3174, D2015, D3177 D2492.

Following the completion of the analytical work, the results were tabulated as elementary data (Appendix I). Computations were also made to convert data to cumulative form for increasing specific gravity. The pounds of SO₂ in each cumulative float product per million Btu were calculated. Also computations were made for the percentage of the calorific value (Btu's) recovered and the percentage pyritic sulfur rejected for the cumulative float material at each specific gravity. Both the elementary and calculated data for all three base coals are included in Appendix I.

The results of these washability tests were also analyzed in several different ways to illustrate the effect of comminuting coal to the various designated sizes on the degree of liberation of the contained mineral matter and pyritic sulfur.

4.4.2 Illinois No. 6 Sample Washability Results

Results of the additional washability testing of Illinois No. 6 coal were completed and are presented here. However, considerable problems were encountered in achieving good separation

at low specific gravities for both the 325-mesh and 400-mesh sizes. Slight problems were also encountered at 1.3 specific gravity for the other two coals, but for Illinois No. 6 the problem was more severe, probably due to the higher clay content of this coal. The heavy-liquid separation tests with these two sizes were repeated and only the results of the second set of tests are reported here.

Because prior experience showed difficulty of separation at 325 mesh, for the 400 mesh sink-float tests, an alternative procedure was followed where separation was first performed at 1.8 specific gravity in order to reject the clay. The float material at 1.8 gravity was then subjected to sink-float separation at 1.3 specific gravity. However, this method yielded strange results and it was decided to revert back to the original procedure. The results for the 400-mesh sink-float tests presented here are those from tests conducted with the procedure outlined in Section 4.1.

Figure 4.15 shows the yield-vs-specific gravity results for Illinois No. 6 coal. As expected, there is a gradual increase in the amount of float material with increasing specific gravity for a given topline, with the limiting yield being about 85% at high specific gravity. The coal yield at 1.3 specific gravity for 100-mesh topline coal is 22.6% compared to 23.6% for the 200-mesh coal. For the 325-mesh and 400-mesh grinds, however, lower yields of float were obtained at 1.3 specific gravity, presumably due to difficulty of separation at finer sizes. Problems of lower yields at 1.3 specific gravity may be either due to complications with the separation of clay matter from the coal particles or due to agglomeration of the clay particles. Also, the yields at various higher specific gravities are very close for all size fractions, indicating general liberation of mineral matter at about 100 mesh.

The separation of the curves at low Btu recoveries indicates that some fine ash minerals are present in the coal particles. The overall liberation trend is given in Figure 4.16 which presents the percent Btu recovery vs. cumulative percent ash for each grind. The minus 100-mesh sample shows an ash content of approximately 5.7% at 90% Btu recovery. When comminuted to a topline of 200 mesh, only about 5.5% ash is present at the same Btu recovery. At a 400-mesh grind, the ash at 90% Btu recovery level was 5.6%. Figure 4.17 plots the calorific value of the cumulative float product of Illinois No. 6 as a function of the ash content for the four grind sizes. It can be seen from this figure that a roughly linear relationship was found between

Figure 4.15 - Cumulative weight percent float at various specific gravities for Illinois No. 6 coal.

Figure 4.16 - Btu recovery vs. ash percent for Illinois No. 6 coal.

Figure 4.17 - Calorific value of the cumulative float product as a function of the ash content for Illinois No. 6 coal.

the calorific value and the ash content of Illinois No. 6 coal for 100, 200 and 325 mesh grinds. For the 400-mesh grind, however, the relationship is different from that for the other three grinds. On the other hand, for Upper Freeport PA and Pittsburgh No. 8 coals, the same linear relationship between the calorific value and ash content for the 100, 200, 325 and 400 mesh grinds was observed. This suggests that sink-float separation and/or the analyses of the ultra-fine (minus 400 mesh) Illinois No. 6 coal are inaccurate, possibly due to the higher clay content of this coal.

For the 100-mesh sample, the total sulfur content is approximately 2.69% at 90% Btu recovery. By grinding the coal to minus 200-mesh, the total sulfur content can be reduced to 2.67% at 90% Btu recovery, to 2.54% by grinding to 325 mesh, and to 2.4% at 400 mesh. This indicates that by grinding the material finer, that is, from 200-mesh topsize down to 400-mesh topsize, the total sulfur can be reduced only by 0.27%.

Pyritic sulfur liberation refers to the ability to physically separate pyrite from the carbonaceous coal species. Sink-float results should be a good indicator of the maximum

Figure 4.18 - Btu recovery of the cumulative float product as a function of pyritic sulfur rejection for Illinois No. 6 coal.

achievable rejection by gravity separation methods of pyritic sulfur at a given grind size.

Figure 4.18 gives the Btu recovery as a function of the pyritic sulfur rejection for the different designated topsizes. This figure, which contains the information most pertinent to the scope of this study, shows the percent of pyritic sulfur rejected as a function of the percentage of the Btu's recovered. At a topsize of 100 mesh, approximately 92% of the pyritic sulfur can be rejected at 90% Btu recovery. The observed pyritic sulfur rejection is about 91% for the 200-mesh sample and 93% for the 325-mesh and the 400-mesh samples.

A comparison of the ash, sulfur and calorific values was made for the samples comminuted to different sizes as well as the samples used for the Task 2 washability work. The cumulative composite data for the ash, total and pyritic sulfur, and calorific value for various designated grind sizes were compared to the average values for the composite data of the original washability test sample used for the Task 2 work. The latter test work was conducted at a different laboratory (Commercial Testing & Engineering, Henderson, Kentucky). The results summarized in Table 4.13 show that the subsamples used for the different washability tests are consistent with

Table 4.13 - Comparison of ash, sulfur, and Btu values of the Illinois No. 6 coal samples used in the additional washability samples with those used for coarse coal washability.

Grind Size	Ash %	Tot S %	Pyr S %	Btu/lb
100 mesh	16.03	4.76	2.68	11,620
200 mesh	16.11	4.97	2.79	11,608
325 mesh	16.20	4.72	2.70	11,405
400 mesh	15.60	4.44	2.34	10,979
Mean	15.99	4.72	2.63	11,403
SD	0.230	0.189	0.171	259
Original Washability Sample (mean)	16.00	4.87	2.94	11,751

each other and also with the samples used for the washability tests under Task 2. Also the ash, total sulfur and calorific values of the samples that had been comminuted to various designated sizes are consistent, as can be seen by low values of standard deviation.

Samples of the Illinois No. 6 coal used in the additional washability testing were prepared and characterized following the procedure described for the Upper Freeport PA sample. The results, given in Table 4.14, show that the coal used in the additional washability tests was nearly identical in composition to that used in the original study.

Table 4.15 compares the results for the 1.30 and 1.60 gravity float fractions for the 100, 200, 325 and 400-mesh samples of the additional washability tests conducted under the program. Also included are the results for the 28 and 200-mesh samples from the original washability study.

Table 4.14 - Proximate, total sulfur and pyritic sulfur analyses for the additional fine washability test samples for Illinois No. 6 coal.

Grind Size	Moisture %	Vol Mat %	Ash %	Fix C %	Tot S %	Pyr S %
100 mesh	4.22	36.14	16.89	46.97	4.92	2.78
200 mesh	3.77	36.03	16.84	47.13	5.09	2.72
400 mesh	4.12	35.77	16.40	47.83	4.85	2.75
Original Washability Sample			16.00		4.87	2.94

Table 4.15 - Comparison of float products at 1.3 and 1.6 specific gravity for Illinois No. 6 coal.

Grind Size	FLOAT AT 1.30				FLOAT AT 1.60			
	Wt %	Ash %	Tot S %	Btu/lb —	Wt %	Ash %	Tot S %	Btu/lb —
100 mesh	22.6	1.97	2.69	14,030	78.1	5.65	2.69	13,348
200 mesh	23.6	2.18	2.71	13,990	76.4	5.45	2.67	13,490
325 mesh	8.9	1.86	2.62	13,950	76.9	5.80	2.53	13,170
400 mesh	7.0	2.05	2.81	13,770	76.0	5.54	2.41	12,620
28 mesh (Task 2)	6.1	1.64	2.52	14,000	84.9	7.76	3.03	13,050
200 mesh (Task 2)	1.7	1.59	2.63	14,150	79.5	7.74	2.75	13,050

As can be seen, all the analyzed values are consistent within the acceptable analytical error and the separation achieved for the additional washability testing at low specific gravities is more satisfactory as compared to the earlier work.

The particle size distributions of the 100-mesh, 200-mesh and 400-mesh grinds of Illinois No. 6 coal used for the additional washability test work were determined using a L&N Microtrac Particle Size Analyzer. In the case of the minus 100-mesh grinds, the sample was first wet-sieved using a 200-mesh screen to remove the plus 200-mesh material before Microtrac analysis. The results, presented in Figure 4.19, indicate that the particle size distributions of the samples are as expected with the minus 200- and 400-mesh grinds being about 95% minus 75 μ m and 37 μ m, respectively, and the 100-mesh sample 96% minus 150 μ m. Details of the washability results for this coal can be found in Topical Report No. 9 submitted to DOE (4).

4.4.3 Pittsburgh No. 8 Sample Washability Results

The feed coal (reconstituted by taking the weighted average of the ash and Btu contents of the various sink-float fractions) had an ash content of 12.1% and a calorific value of 12,600 Btu/lb, both on a dry basis. Total sulfur analyzed at 4.0%, which corresponds to a total sulfur dioxide (SO₂) emission of nearly 6.4 lb SO₂/million Btu. The pyritic sulfur content of the feed analyzed at 2.6%, which represents 65% of the total sulfur in the coal.

Figure 4.20 presents the cumulative weight percent of material floated versus specific gravity of separation for Pittsburgh No. 8 coal. As expected, there is a gradual increase in the

Figure 4.19 - Particle size distribution for Illinois No. 6 coal washability test samples ground to 100, 200 and 400 mesh top size fraction.

Figure 4.20 - Cumulative weight percent float at various specific gravities for Pittsburgh No. 8 coal.

Figure 4.21 - Btu recovery of the cumulative float product as a function of the ash content for Pittsburgh No. 8 coal.

weight of the float product with increasing gravity for a given top size. The coal yield at 1.3 specific gravity for the 100-mesh sample is 60.4% compared to 61.7% for the 200-mesh coal. This suggests that no significant additional liberation of coal and mineral matter occurs between 100 and 200 mesh. For the 325- and 400-mesh grinds, however, a drop in float product yield was observed at 1.3 specific gravity, possibly due to the difficulty in carrying out the separation at extremely fine sizes. This drop has an impact on the weight of the float products at other specific gravities and ultimately on the cumulative results. For example, the weight of the float product at 1.35 x 1.40 specific gravity for the 325-mesh and 400-mesh samples is considerably higher than that for the 200-mesh sample. This effect carried over to higher gravity intervals as well. The limiting yield at high specific gravity appears to be about 90%, as expected if the ash minerals are liberated at the grind size.

Aspects of the liberation of mineral matter from Pittsburgh No. 8 coal can be seen from the results plotted in Figure 4.21 which shows the Btu recovery as a function of the cumulative percent ash for each top size. The 100-mesh sample exhibits an ash content of about 4% at a

Figure 4.22 - Btu recovery of the cumulative float product as a function of pyritic sulfur rejection for Pittsburgh No. 8 coal.

90% Btu recovery level. When crushed to a top size of 200 mesh, only about 3.9% ash is present at the same Btu recovery. The difference in the ash levels at 90% Btu recovery is very small between the two sizes, suggesting that ash mineral liberation at 100 mesh is comparable to that at 200 mesh. This can also be seen from other results to be discussed later. The ash at 90% Btu recovery decreases to around 3.5% for the 325- and 400-mesh grinds.

For the minus 100-mesh sample, the total-sulfur content of the float product is approximately 1.7% at 90% Btu recovery, while it is about 1.6% for the 200-mesh grind, and approximately 1.5% for the next two finer grinds. The pyritic sulfur content of the Pittsburgh No. 8 sample is reduced from 2.67% to about 0.30% for the 100-mesh sample, 0.27% for the 200-mesh sample, 0.15% at 325 mesh and 0.15% for the minus 400-mesh sample at 90% Btu recovery.

Figure 4.22 presents the Btu recovery as a function of pyritic sulfur rejection for Pittsburgh No. 8 coal at the designated sizes. At 100-mesh top size, more than 90% of the pyritic sulfur can be rejected by heavy-liquid separation at 90% Btu recovery. Pyritic sulfur rejection can

Table 4.16 - Comparison of ash, sulfur and Btu values of the Pittsburgh No. 8 coal samples used in the fine and coarse coal washability tests.

<u>Grind Size</u>	<u>Ash %</u>	<u>Tot S %</u>	<u>Pyr S %</u>	<u>Btu/lb —</u>
100 mesh	11.91	4.09	2.67	12,750
200 mesh	12.14	4.08	2.75	12,600
325 mesh	11.57	3.76	2.42	12,750
400 mesh	12.70	4.16	2.40	12,250
Mean	12.07	4.02	2.56	12,600
SD	0.41	0.155	0.153	200
Original Washability Sample (Mean)	11.90	3.91	2.85	12,900

be increased to almost 93% by reducing the top size to 200 mesh and further reduction of the grind size to 325 mesh increases the pyritic sulfur rejection to over 95%. The test results indicate that a 400-mesh grind does not appear to achieve any better results over a 325-mesh grind, possibly due to the difficulty of obtaining a good separation at these fine sizes.

The cumulative composite data for the samples comminuted to various sizes were compared to the average values of the composite data from the original washability sample. The results, given in Table 4.16, show that the additional washability subsamples are consistent with each other and also with the samples used for the washability tests conducted as part of Task 2.

As part of the QA/QC program, samples of the Pittsburgh No. 8 coal used in the additional washability testing were prepared, and proximate and sulfur forms analyses were completed following the procedure described for the Upper Freeport PA sample. The results, given in Table 4.17, show that the coal used in the additional washability tests was nearly identical in composition to that used in the original study Task 2 investigation.

Table 4.18 summarizes the analyses of the float products at 1.30 and 1.60 specific gravity for the various grind sizes tested. The analytical values generally appear to be consistent within the allowable analytical error for ash, total sulfur and calorific values except at 1.3 where the decrease in the float product yield is greater than expected. This again is probably due to the difficulty of separation of ultrafine coal.

The particle size distributions of the 100-mesh, 200-mesh and 400-mesh grinds of the Pittsburgh No. 8 coal used for the additional washability test work were determined using a

Table 4.17 - Proximate, total sulfur and pyritic sulfur analyses for the additional fine washability test samples for Pittsburgh No. 8 coal.

Grind Size	Moisture %	Vol Mat %	Ash %	Fix C %	Tot S %	Pyr S %
100 mesh	2.49	35.45	12.21	52.34	4.03	2.65
200 mesh	2.57	34.86	12.52	52.62	4.26	2.72
400 mesh	2.35	34.49	12.32	53.19	3.93	2.64
Original Washability sample			11.90		3.90	2.84

L&N Microtrac Particle Size Analyzer. In the case of the minus 100-mesh grinds, the sample was first wet-sieved using a 200-mesh screen and the minus 200-mesh fraction was then used for Microtrac analysis. The results, presented in Figure 4.23, indicate that the particle size distributions of the samples were as expected with the minus 200 and 400-mesh grinds, being about 95% minus 75 and 37 μ m, respectively, and the 100-mesh sample being 96% minus 150 μ m. Details of the washability results for this coal can be found in Topical Report No. 7 submitted to DOE (5).

4.4.4 Upper Freeport PA Sample Washability Results

The feed coal (reconstituted by taking the weighted average of the ash and Btu contents of the various sink-float fractions) had an ash content of 12.5% and a calorific value of 13,500 Btu/lb, both on a dry basis. Total sulfur analyzed at 2.2%, which corresponds to a total sulfur

Table 4.18 - Comparison of floats at 1.3 and 1.6 specific gravity for Pittsburgh No. 8 seam samples tested.

Size	FLOAT AT 1.3				FLOAT AT 1.6			
	Wt %	Ash %	Tot S %	Btu/lb	Wt %	Ash %	Tot S %	Btu/lb
100 Mesh	60.4	2.73	1.57	14,400	86.2	4.44	1.78	14,100
200 Mesh	61.7	2.45	1.45	14,050	84.7	4.21	1.64	14,050
325 Mesh	50.6	1.86	1.43	14,550	85.5	3.94	1.54	14,100
400 Mesh	39.4	1.73	1.43	14,400	77.3	3.82	1.50	13,900
28 mesh (Task 2)	51.1	2.66	1.59	14,400	87.3	5.29	2.21	14,000
200 mesh (Task 2)	53.9	2.08	1.35	14,500	86.8	4.26	1.48	14,050

Figure 4.23 - Particle size distribution for Pittsburgh No. 8 coal washability test samples ground to 100, 200 and 400 mesh top size fraction.

dioxide (SO_2) emission of nearly 3.3 lb SO_2 /million Btu. The pyritic sulfur content of the feed analyzed at 1.5%, which represents 66% of the total sulfur in the coal.

Figure 4.24 presents a plot of yield vs specific gravity for Upper Freeport PA coal at the four different grinds. As expected, there is a gradual increase in the amount of float material with increasing specific gravity for a given top size. The float yield at 1.3 specific gravity for the 100-mesh top size coal is 51.0% compared to 54.7% for the 200-mesh coal. This suggests that most of the practical liberation of coal and mineral matter has taken place at 100 mesh and there is no significant additional liberation at 200 mesh. For the 325-mesh and 400-mesh grinds, however, slightly lower float yields were obtained at 1.3 specific gravity, presumably due to the increasing difficulty of separation at finer sizes. The yields at other specific gravities are very close for all four grinds. The limiting yield at high specific gravity tends towards 92 percent.

The overall liberation trend of the ash minerals can be observed from the graphs shown in Figure 4.25, which presents the percent Btu recovery vs. cumulative percent ash for each sample. The minus 100-mesh sample has an ash content of approximately 5.5% at a 90%

Figure 4.24 - Cumulative weight percent float at various specific gravities for Upper Freeport PA coal.

Figure 4.25 - Btu recovery of the cumulative float product as a function of the ash content for Upper Freeport PA coal.

Figure 4.26 - Calorific value of the cumulative float product as a function of the ash content for Upper Freeport PA washability samples comminuted to various top sizes.

Btu recovery level. When comminuted to a topsize of 200 mesh, only about 4.5% ash is present at the same Btu recovery. The difference of the ash level at 90% Btu recovery is small between the two sizes, indicating that only a small amount of additional liberation has taken place between 100 and 200 mesh. At 400 mesh, the ash decreases only slightly over that of the two coarser sizes at 90% Btu recovery. Figure 4.26 plots the calorific value of the cumulative float product of Upper Freeport PA coal as a function of ash content using the results for different grind sizes from the original washability and fine washability tests. As expected, this figure shows that a

linear relationship exists between the calorific value and the ash content of Upper Freeport PA samples. This is because the combustible material content in the samples decreases linearly with increasing ash content, and for the same coal, the calorific value of the carbonaceous material should be constant. By extrapolating these linear relationships to zero ash content, the calorific value of the coal macerals in the base coals were determined to be in the following order:

Upper Freeport PA (15,700 Btu/lb) > Pittsburgh No. 8 (15,600) > Illinois No. 6 (14,300)

This order is the same as that of the dmmf carbon content of the base coals.

The total-sulfur content in the Upper Freeport PA feed sample was 2.2%. At 90% Btu recovery, the total-sulfur content of this coal was reduced to approximately 0.95% for the 100-mesh sample, at 90% Btu recovery was about 0.92% for the 200-mesh sample 0.85% for the 400-mesh grind. While there is a major drop in the total-sulfur content from the head sample of 2.2% to 0.95% in the float products at 90% Btu recovery for the 100-mesh grind, grinding the coal to top sizes of 200, 325, and 400 mesh resulted in only a slight additional drop in total sulfur at corresponding Btu recoveries.

Pyritic sulfur liberation refers to the capability of being able to physically separate pyrite from the carbonaceous coal species. Sink-float results are a good indicator of the maximum achievable rejection of pyritic sulfur by gravity separation at a given grind size. Starting with a pyritic sulfur content of 1.46% (average value) in the Upper Freeport PA test sample, the pyritic sulfur content of the coal sample decreased to about 0.14% for the 100-mesh grind, 0.09% for the 200-mesh grind, and 0.07% for the 400-mesh grind at 90% Btu recovery.

Figure 4.27 depicts the Btu recovery as a function of the pyritic sulfur rejection for Upper Freeport PA coal comminuted to four different top sizes. This figure, containing the information most pertinent to the scope of this study, gives the percent of pyritic sulfur rejected in relation to the percentage of the Btu's recovered. At 100-mesh top size, approximately 92% of the pyritic sulfur can be rejected at 90% Btu recovery. At this same level of Btu recovery in the float product, the pyritic sulfur rejection increased to almost 95% by further reducing the top size to

Figure 4.27 - Btu recovery of the cumulative float product as a function of pyritic sulfur rejection for Upper Freeport PA coal.

200 mesh. Further reduction of the top size to 325 mesh and 400 mesh appears to produce only marginally higher pyritic sulfur rejection.

A comparison of the ash, sulfur and calorific values was made for the samples comminuted to different sizes as well as the samples used for the Task 2 washability work. The cumulative composite data for the ash, total and pyritic sulfur, and calorific value for various designated grind sizes were compared to the average values for the composite data of the original washability sample (mean) sample used for the Task 2 work, (the latter test work was conducted at a different laboratory, Gould Energy/Warner Laboratories Division, Cresson, Pennsylvania). The results, summarized in Table 4.19, show that the subsamples used for various designated grinds in the washability program are consistent with each other and with the mean values of the samples used for the original washability program under Task 2. Also, the standard deviation in ash, total sulfur and calorific values of the samples comminuted to various designated sizes is small.

Table 4.19 - Comparison of ash, sulfur, and Btu values of the Upper Freeport PA coal samples used in the fine and coarse washability testing.

Grind Size	Ash %	Tot S %	Pyr S %	Btu/lb —
100 mesh	12.54	2.20	1.42	13,400
200 Mesh	12.46	2.32	1.54	13,300
325 Mesh	12.36	2.18	1.47	13,400
400 Mesh	12.46	2.18	1.42	13,150
Mean	12.46	2.22	1.46	13,300
SD	0.064	0.058	0.049	98.6
Original Washability Sample (Mean)	12.46	2.22	1.46	13,470

The coal samples used in the fine washability study (minus 100, 200 and 400 mesh) were further characterized at Berkeley by proximate, total-sulfur and pyritic-sulfur analyses to ensure that the coal samples used in the additional washability tests were the same as those used in the original study and as part of the QA/QC program. These samples were prepared for washability tests at the Geochemical Laboratory for each of the three base coals and a portion of each sample was sent to Berkeley for various characterization tests.

Once the samples were received at Berkeley, they were homogenized and split in a rotating splitter. Half of each sample was inerted, packed and sent to Columbia University for size analysis and other QA/QC tests. The remainder of each sample was further homogenized and split in order to perform characterization studies. Approximately 10 grams of each sample was used for proximate, total-sulfur, and pyritic-sulfur analyses and the remaining sample was used for particle-size analysis.

Table 4.20 presents the Berkeley results for the proximate, total-sulfur and pyritic-sulfur analyses for all the washability test samples except the minus 325-mesh sample. These results show that the samples used in the washability tests were consistent in composition and comparable to the samples used in the original washability study. This indicates that the Upper Freeport PA coal did not degrade while stored in the drums and that the results can be compared with the original washability data to yield information on both coarse and fine liberation behavior.

Table 4.20 - Proximate, total sulfur and pyritic sulfur analyses for the additional fine washability test samples for Upper Freeport PA coal.

Grind Size	Moisture %	Vol Mat %	Ash %	Fix C %	Tot S %	Pyr S %
100 mesh	1.54	25.51	12.32	62.22	2.44	1.55
200 mesh	1.22	25.39	12.83	61.78	2.31	1.53
400 mesh	0.98	24.95	12.97	62.08	2.25	1.53
Original Washability Sample			12.46	2.22	1.46	

Table 4.21 compares the results for the float products at 1.30 and 1.60 gravity for the 100-mesh, 200-mesh, 325-mesh and 400-mesh samples of the additional washability tests conducted under the program. In the original coarse washability tests (4-inch, 1 1/2-inch, and 1/8-inch) the float products at 1.3 specific gravity increased gradually with decreasing top size. However, in the case of the additional washability test results of the fine sizes presented here, the weight of the float product at 1.3 increases only marginally and then drops off slightly for the very fine 325-mesh and 400-mesh sizes. This may be the result of difficulty in the separation of fine particles at fine sizes. The weight of the float products at 1.6 specific gravity remains nearly constant for all four grind sizes.

The particle size distributions of the 100 mesh, 200 mesh and 400 mesh grinds of the Upper Freeport PA coal used for the additional washability and test work were determined using a L&N Microtrac Particle Size Analyzer. Because of the particle size limitation on the

Table 4.21 - Comparison of floats at 1.3 and 1.6 specific gravity for Upper Freeport PA coal samples tested.

Size	FLOAT AT 1.3				FLOAT AT 1.6			
	Wt %	Ash %	Tot S %	Btu/lb	Wt %	Ash %	Tot S %	Btu/lb
100 Mesh	51.0	1.88	0.86	15,300	86.9	6.04	0.96	14,600
200 Mesh	54.7	1.81	0.83	15,300	85.7	5.03	0.93	14,700
325 Mesh	46.8	1.81	0.79	15,450	86.6	5.40	0.86	14,700
400 Mesh	47.1	1.94	0.80	15,200	85.5	4.91	0.86	14,550
28 mesh (Task 2)	44.9	2.32	0.89	15,200	85.4	6.50	1.02	14,500
200 mesh (Task 2)	54.9	1.87	0.79	15,250	81.0	3.75	0.82	15,000

Figure 4.28 - Particle size distribution for Upper Freeport PA coal washability test samples ground to 100, 200 and 400-mesh top size fraction.

Microtrac, samples of minus 100-mesh grinds were first wet-sieved using a 200-mesh screen and the minus 200-mesh fraction was used for Microtrac analysis. The results, presented in Figure 4.28, indicate that the particle size distributions are as expected. We were concerned that the size distributions of each of the fine washability grinds did in fact represent minus 100-, 200- and 400-mesh grinds. As can be seen from the plotted size distributions, the minus 200 and 400-mesh grinds are indeed about 95% minus 75 and 37 μm , respectively, while the 100-mesh grinds appear to be about 96 - 98% minus 150 μm . The deviation of the particle size distributions from a straight line at extremely fine particles sizes is probably due to the measuring device since 5 μm is near the lower limit of detection by the measuring method. Details of the washability results for this coal can be found in Topical Report No. 8 submitted to DOE (6).

4.4.5 Fine Coal Washability Summary

The results of the fine washability test work show that for all the base coals, liberation is sufficient to achieve project objective (90% pyritic sulfur rejection at 90% combustible matter

Table 4.22 - Ash, total sulfur, pyritic sulfur content and calorific value of the additional coals.

<u>Coal</u>	<u>Ash, %</u>	<u>Tot. S, %</u>	<u>Pyt. S, %</u>	<u>Btu/lb</u>
Upper Freeport WV	14.73	3.01	2.19	13,066
Kentucky No. 9	14.34	4.50	2.98	12,284
Wyodak	7.30	0.49	0.08	11,788

recovery) by gravity separation (heavy liquid) when the sample is pulverized to 95% passing 100 mesh. Further size reduction results in only slightly better gravity separations. However, since the process under consideration in this project is flotation, the effect of particle size on the surface properties of the particles and on bubble/particle interaction must be considered. Because the efficiency and frequency of collisions between bubbles and particles decrease with decreasing particle size, and the energy consumption in grinding increases with decreasing particle size, it is desirable to keep the particle size as large as possible. Further, gravity separations by sink-float are probably as close to a perfect separation as can be achieved based on the difference in the specific gravity of individual particles, whereas flotation processes using conventional flotation machines, can not achieve a perfect separation based on the difference in hydrophobicity of particles. Thus, the pyritic sulfur rejections obtained by sink-float separation at fine sizes may not be achievable by flotation processes.

4.5 Washability Tests on Additional Coal Samples

As part of the program, samples of coal were obtained of Upper Freeport, Grant County, West Virginia; Kentucky No. 9, Hopkins County, Kentucky; and Wyodak Seam, Campbell County, Wyoming. Table 4.22 shows the coal composition and calorific value for each of these coals. Sample collection, handling, washability testing, and data analysis were carried out in the same manner as described in Sections 4.1 and 4.2 of this report.

4.5.1 Washability of Upper Freeport WV Coal

Several figures were constructed from the results of the sink-float tests on Upper Freeport WV coal in order to compare the percent ash, total sulfur, and pyritic sulfur reduction in the float fractions achieved for progressively finer sizes of the coal sample with the corresponding Btu recovery values. The percent pyritic sulfur rejection is also compared with the

Figure 4.29 - Cumulative weight percent (yield) float at various specific gravities for Upper Freeport WV coal.

corresponding values of percent Btu recovery in the float fraction. The results for each of these parameters are discussed below.

Figure 4.29 presents the cumulative weight percent material floated vs. specific gravity of the test liquid for the Upper Freeport WV coal. As expected, there is a gradual increase in the weight of the floats with increasing specific gravity for a given top size. In terms of coal yield at a given specific gravity as particle size is reduced, the coal yield at 1.3 specific gravity for the 4-inch top size is 33.8% compared to 42.2% for the 28-mesh coal. This suggests that some of the liberation of mineral matter from the coal takes place when the size is reduced to 28 mesh. For the 200-mesh grind, however, the yield decrease at all specific gravities suggests difficulties of separation at the fine size during the test procedure.

Figure 4.30 presents the Btu recovery as a function of cumulative ash for this coal. The 4-inch and 1-1/2-inch top size products exhibit an ash content of approximately 7.5% at 90% Btu recovery level. When crushed to a top size of 1/8 inch, about 6.5% ash is present in the float material at the same Btu recovery, dropping to 5.5% when the coal top size is reduced to

Figure 4.30 - Btu recovery of the cumulative float product as a function of the ash content for Upper Freeport WV coal.

28 mesh. Since the ash decreases to about 4.5% for samples comminuted to 200 mesh, at 90% Btu recovery level, over 40% reduction in the ash content can be achieved by reducing the top size from 4 inches to 200 mesh.

The total sulfur content of this coal, which is nearly 3.0%, can be reduced to 1.4% at 90% Btu recovery at a top size of 4-inches. Both of these values are identical to those of the Upper Freeport PA seam. In addition, plots of the total sulfur and Btu recovered in the float fractions follow essentially the same trend as the ash. As may be seen from the plots given in Figure 4.31, a major reduction occurs when the sample top size is reduced to 1/8-inch, where the total sulfur in the float fraction is nearly 1.0% at 90% Btu recovery. The total sulfur of the 28-mesh product is about 0.8% at 90% Btu recovery. Surprisingly, no additional reduction in total sulfur at the 90% Btu recovery level occurs by comminuting this coal to 200-mesh top size. This trend is somewhat different from that observed in the sample of this seam obtained from Indiana County, Pennsylvania.

Figure 4.31 - Btu recovery of the cumulative float product as a function of the total sulfur content for Upper Freeport WV coal.

Total pyritic sulfur content of this coal averages 2.19% for the five samples. Pyritic-sulfur liberation is directly related to the top size of the feed. In the sink-float separation, the pyritic-sulfur content of the float material is nearly 1.0% at 90% Btu recovery for a sample at 4-inch top size (Figure 4.32). Significant liberation takes place when the top size is reduced to 1/8 inch, when the pyritic sulfur content of the float product is only about 0.35% at about 90% Btu recovery. In contrast to the ash and total sulfur levels, the pyritic sulfur rejection increases further at 200 mesh.

Figure 4.33 depicts the percent Btu recovery as a function of pyritic sulfur rejection for this coal crushed to pass various top sizes. As expected from the results given in Figure 4.33, there is an increase in the pyritic sulfur rejection value as the coal is ground finer. At the 90% Btu recovery level, pyritic sulfur rejection is about 92% for the 28-mesh grind and 95% for the 200-mesh grind. These results indicate that high levels of pyritic sulfur rejection are potentially achievable for this coal. These results are almost identical to those obtained for the Upper Freeport sample collected from Indiana County, Pennsylvania.

Figure 4.32 - Btu recovery of the cumulative float product as a function of the pyritic sulfur content for Upper Freeport WV coal.

Figure 4.33 - Btu recovery of the cumulative float product as a function of pyritic sulfur rejection of Upper Freeport WV coal.

As a part of the project QA/QC procedures, a statistical analysis was performed on the cumulative composite data for all five size fractions, that is, 4-inch x 0, 1-1/2-inch x 0, 1/8-inch x 0, 28 mesh x 0, and 200 mesh x 0.

The mean and standard deviations for ash are 14.73 and 0.31%, respectively. The corresponding values for total sulfur are 3.01 and 0.10% and those for pyritic sulfur are 2.19 and 0.25%, respectively. The average ash and total sulfur values for the Upper Freeport WV sample are 14.0% and 2.85%, respectively. The ash content of the project sample may appear to be lower than is commonly reported in the literature for this seam. The difference in this value is because the coal sample procured for this project was "clean," that is, it was sampled from the mine face to represent a channel sample, and high ash contamination was excluded by carefully removing the overburden material. Details of the washability results for this coal can be found in Topical Report No. 5 submitted to DOE (7).

4.5.2 Washability of Kentucky No. 9 coal

As part of this research project, a sample of Kentucky No. 9 coal from Hopkins County, Kentucky was also collected and submitted for standard washability testing at Geochemical Testing Lab, Somerset, Pennsylvania.

This coal contains 4.50% total sulfur, 2.98% pyritic sulfur and 14.34% ash. Figure 4.34 plots the Btu recovery as a function of cumulative ash in the float product. Figures 4.35 and 4.36 are plots of the Btu recovery as a function of total sulfur and pyritic sulfur, respectively, in the float products for sink-float separation of material crushed to the various top sizes. Figure 4.37 presents the Btu recovery as a function of the percentage of pyritic sulfur rejected for the various degrees of comminution. As can be seen, only by comminuting the coal to minus 200-mesh, does the washability analysis show the possibility of 90% Btu recovery at 90% pyritic sulfur rejection. Crushing the coal to minus 28-mesh leaves the coal much coarser than the apparent liberation size. Details of the washability results for this coal can be found in Topical Report No. 4 submitted to DOE (8).

Figure 4.34 - Btu recovery plotted as a function of ash for various topsizes of Kentucky No. 9 coal.

Figure 4.35 - Btu recovery plotted as a function of total sulfur for various topsizes of Kentucky No. 9 coal.

Figure 4.36 - Btu recovery plotted as a function of cumulative pyritic sulfur for various topsizes of Kentucky No. 9 coal.

Figure 4.37 - Btu recovery plotted as a function of pyritic sulfur rejection for various top sizes of Kentucky No. 9 coal.

Figure 4.38 - Cumulative weight percent yield (float) at various specific gravities for the Wyodak seam coal from Campbell County, Wyoming.

4.5.3 Washability of Wyodak Coal

A sample of Wyodak seam coal was obtained from Campbell County, Wyoming, and the coal was submitted to standard washability testing at Core Laboratories, Casper, Wyoming. This coal analyzed 0.49% total sulfur, 0.08% pyritic sulfur and 7.3% ash. Because of the low sulfur and low ash, the washability curves yield no useful information and will not be presented here. Figure 4.38, which is a plot of the yield as a function of separation specific gravity, indicates that essentially everything floats at all specific gravities except for minus 200-mesh data point at specific gravity 1.3. This point is probably meaningless. Details of the washability results for this coal can be found in the Topical Report No. 6 submitted to DOE (9).

4.6 References

1. Topical Report No. 3, "Coal Washability Data Analysis - Illinois No. 6 Seam Coal, Randolph County, Illinois," Coal Surface Control for Advanced Fine Coal Flotation, DOE Contract No. DE-AC22-88PC88878, March 1989.

2. Topical Report No. 1, "Coal Washability Data Analysis - Pittsburgh No. 8 Seam Coal, Belmont County, Ohio," Coal Surface Control for Advanced Fine Coal Flotation, DOE Contract No. DE-AC22-88PC88878, January 1989.
3. Topical Report No. 2, "Coal Washability Data Analysis - Upper Freeport Seam Coal, Indiana County, Pennsylvania," Coal Surface Control for Advanced Fine Coal Flotation, DOE Contract No. DE-AC22-88PC88878, July 1989.
4. Topical Report No. 9, "Fine Coal Washability Data Analysis - Illinois No. 6 Seam Coal, Randolph County, Illinois", Coal Surface Control for Advanced Fine Coal Flotation, DOE Contract No. DE-AC22-88PC88878, May 1990.
5. Topical Report No. 7, "Fine Coal Washability Data Analysis - Pittsburgh No. 8 Seam Coal, Belmont County, Ohio", Coal Surface Control for Advanced Fine Coal Flotation, DOE Contract No. DE-AC22-88PC88878, March 1990.
6. Topical Report No. 8, "Additional Coal Washability Data Analysis - Upper Freeport Coal, Indiana County, Pennsylvania", Coal Surface Control for Advanced Fine Coal Flotation, DOE Contract No. DE-AC22-88PC88878, March 1990.
7. Topical Report No. 5, "Coal Washability Data Analysis - Upper Freeport, Grant County, West Virginia," Coal Surface Control for Advanced Fine Coal Flotation, DOE Contract No. DE-AC22-88PC88878, March 1991.
8. Topical Report No. 4, "Coal Washability Data Analysis - Kentucky No. 9, Hopkins County, Kentucky," Coal Surface Control for Advanced Fine Coal Flotation, DOE Contract No. DE-AC22-88PC88878, September 1989.
9. Topical Report No. 6, "Coal Washability Data Analysis - Wyodak Seam, Campbell County, Wyoming", Coal Surface Control for Advanced Fine Coal Flotation, DOE Contract No. DE-AC22-88PC88878, May 1990.

5.0 SURFACE CHARACTERIZATION STUDIES

As stated in Chapter 3, characterization of coal can be concerned with either bulk or surface properties. Because flotation is a surface-based separation process, the surface properties will ultimately control the behavior of the particles during flotation. Often, the surface properties of a solid are very much different than bulk properties.

Surface characterization studies conducted as part of this investigation include analysis of both the macroscopic and microscopic properties. Macroscopic properties evaluated included analysis of the wettability behavior by means of film flotation, contact angle measurements and induction time measurements, and surface charge characteristics by zeta potential measurements. Analysis of surface elemental composition by ESCA (electron spectroscopy for chemical analysis) and determination of organic functional groups by DRIFT constituted the microscopic characterization of the coal.

5.1 Surface Area Determination

The specific surface areas of representative samples of the three base coals were determined by gas adsorption using a Micromeritics BET (Brunauer, Emmett, Teller) Surface Area and Pore Size Analyzer Model 2100. The adsorbate gas was research grade CO₂ (minimum purity of 99.998%). For each coal, two samples of different particle size were used, namely minus 28-mesh and minus 200-mesh. The samples were dried and degassed in a vacuum at room temperature for at least 24 hours before the measurements were performed. The specific surface area was calculated using the Dubinin-Polanyi equation (1,2) and the results given in Table 5.1.

Table 5.1 - Determination of the specific surface areas of the three base coals by CO₂ adsorption at room temperature.

<u>Sample</u>	<u>Grind size</u>	<u>m²/g</u>
Upper Freeport PA	-28 mesh	157
	-200 mesh	136
Illinois No. 6	-28 mesh	180
	-200 mesh	164
Pittsburgh No. 8	-28 mesh	144
	-200 mesh	155

These results show that Upper Freeport PA and Pittsburgh coals each have a specific surface area of around $150 \text{ m}^2/\text{g}$. On the other hand, Illinois No. 6 coal exhibits a higher specific surface area ($172 \text{ m}^2/\text{g}$), although slight, considering the accuracy of the instrument (about 10 to $20 \text{ m}^2/\text{g}$). In all cases, the particle size of the coal samples did not change the specific surface area significantly. This is typical of high surface area materials since any change in external surface due to size is small compared with the large surface area in the pores.

5.2 Wettability Behavior Studies

5.2.1 Film Flotation

Wetting characteristics of the three base coals (Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA) were assessed using the film flotation technique developed by Fuerstenau and coworkers (3,4). Representative samples were ground and sieved in a glove box filled with argon gas to prevent oxidation of the coal. The sieved samples were also stored inside a glove box prior to performing the film flotation tests. Specifically, a 100 x 150 mesh size fraction of each of the base coal samples was used for determination of the wetting tension distribution of the coal particles. Briefly, the experimental procedure consisted of carefully placing a monolayer of particles onto the surface of a liquid of given surface tension. Particles that separate into hydrophobic (float) and hydrophilic (sink) fractions were then filtered, dried and weighed.

The wettability of the three coals was assessed using aqueous methanol solutions of different concentrations to vary the surface tension. The critical wetting tension distribution diagrams obtained for the three samples are presented in Figure 5.1. It can be seen from this figure that the partition curves of Pittsburgh No. 8 and Upper Freeport PA coals are similar to each other whereas that of the Illinois No. 6 coal is shifted to the more hydrophilic region (higher surface tension). This indicates that both the Pittsburgh No. 8 and the Upper Freeport PA coals have similar wetting behavior and are more hydrophobic than Illinois No. 6 coal. The greater hydrophilicity of Illinois No. 6 coal, as discussed earlier, is also reflected in its higher ash and moisture contents (see Table 4.1).

Figure 5.1 - Film flotation partition curves for the three base coals.

5.2.2 Contact Angle Measurements

Contact angles of the three base coals were measured using sessile-drop and captive-bubble methods on polished surfaces of coal specimens. The contact angle is defined as the angle measured across the liquid at the three-phase line of contact. The measured contact angles were compared with the values calculated from the critical wetting surface tension measured by film flotation using a model developed at Berkeley (4).

For contact angle measurement, a hand-picked coal lump was cut to produce a flat surface using a clean sharp chisel. The specimen was dry-polished with polishing papers ranging from 240 to 600 grit in an argon-filled glove box. In this manner the oxidation of the coal surface during polishing was minimized. Argon gas was used to blow the debris from over the polished surface. The dry polished surface was further polished on a polishing wheel with 0.3 micron alumina powder suspended in distilled water. The polished specimen was then washed with triple-distilled water several times to remove traces of the polishing powder. For some of the measurements, 100 x 150 mesh coal particles were pressed into a pellet in a 0.5 inch diameter mold at 10,000 psi

Table 5.2 - Air/water advancing contact angles (in degrees) measured on the three base coals by the sessile-drop method on surfaces prepared by different methods.

<u>Coal Sample</u>	<u>Dry-polished in Argon</u>	<u>Wet-polished in Air</u>	<u>Pellet</u>
Illinois No. 6	70	44	41
Pittsburgh No. 8	82	60	66
Upper Freeport PA	81	62	69

for 10 minutes and contact angles were measured with the pellets in the same manner as was used with the polished specimen.

"Equilibrium" contact angles were measured after a drop or a bubble had made perfect contact with the sample surface. The receding contact angle was determined after adding air to a small bubble that was already attached to the coal surface and the advancing contact angle was determined after removing some air from the same bubble that had been used for measuring the receding contact angle. Each reading is an average of the angles measured on both sides of the drop or the bubble in order to account for any slight specimen tilt or surface roughness. The reported contact angles are the average of at least ten such readings on two specimens for each coal. The contact angles were measured with a Rame-Hart contact angle goniometer.

Initially, dry polished samples were used for measuring advancing contact angles by the sessile drop method. However, the angles were found to be much larger than those obtained with either the wet-polished samples or coal pellets, as shown in Table 5.2. This behavior may be due to "smearing" of hydrophobic material over the dry polished surface, resulting in larger and somewhat misleading contact angles.

The advancing, receding and equilibrium contact angles measured on a wet-polished surface by the captive-bubble method are given in Table 5.3. The results obtained show that the receding contact angles are always smaller than the advancing angles and are closer to the equilibrium contact angles. The difference between the receding and the advancing angles is generally observed when measuring contact angles and is often attributed to surface roughness or other heterogeneities, which are generally too fine to see without high-powered microscopes. The difference in advancing and receding angles contributes to bubble/particle attachment/detachment

Table 5.3 - Air/water contact angles (in degrees) measured on the three base coals by the captive-bubble method on surfaces wet-polished in air.

<u>Coal Sample</u>	<u>Advancing</u>	<u>Receding</u>	<u>Equilibrium</u>
Illinois No. 6	42	23	25
Pittsburgh No. 8	61	45	49
Upper Freeport PA	61	42	50

processes in flotation. In all cases, Illinois No. 6 is much more hydrophilic than Pittsburgh No. 8 and Upper Freeport PA coals which exhibit essentially equal hydrophobicity.

Because the advancing and receding contact angles measured by the captive-bubble technique can vary widely, care must be taken to choose the most meaningful one when comparing these results to those obtained using other techniques or physical processes. Since the sessile drop method involves the addition of a liquid drop on the surface the resulting value is that for a liquid-advancing contact angle. Similarly, the physical process involved in film flotation is also dependent on advance of the liquid on the coal particles. Table 5.4 shows the results of the contact angles measured from the sessile-drop method and those calculated from film flotation data. The results compare quite well with those of the advancing captive-bubble measurements.

Sessile drops sitting on the coal surface may be absorbed into the pores of the coal or may solubilize components present at the surface. The effect of time on the contact angle of a sessile drop on the surface of the three base coals is given in Table 5.5. As before, Illinois No. 6 appears to be the most hydrophilic, with the bubble being completely absorbed by the coal within two minutes. This is indicative of the fact that the pores of Illinois No. 6 are hydrophilic and are filled rapidly with the liquid in the droplet. The other two coals again behaved similarly, but

Table 5.4 - Air/water contact angle measurements on the three base coals calculated from film flotation results and advancing contact angles measured by the sessile-drop and the captive-bubble methods on surfaces wet-polished in air.

<u>Coal Sample</u>	<u>CONTACT ANGLE, DEGREES</u>		
	<u>Film Flotation</u>	<u>Sessile-Drop</u>	<u>Captive-Bubble</u>
Illinois No. 6	52	44	42
Pittsburgh No. 8	63	60	61
Upper Freeport PA	63	62	61

Table 5.5 - Contact angle measurements of the three base coals as a function of the time after placing a water drop on a pellet surface (using the sessile-drop method).

Coal Sample	ELAPSED TIME, MINUTES								
	0	0.5	1	2	5	10	15	25	40
Illinois No. 6	46	32	24	7	0				
Pittsburgh No. 8	68	--	66	65	62	59	49	41	15
Upper Freeport PA	71	69	--	68	65	55	47	32	10

showing less decrease in contact angle for 40 minutes, after which the contact angles were about 10 degrees indicating that the pores of these two coals have a much lower affinity for water than do those of Illinois No. 6. It should be noted that as the water absorbs into the coal, the contact angle becomes a receding angle since the liquid/vapor interface is contracting.

In summary, a detailed contact angle study of the three base coals was observed to help to characterize the wettability of the coal. Good agreement was obtained among the angles obtained using various direct measurement techniques and those calculated from film flotation results. In all tests, Illinois No. 6 was consistently more hydrophilic than either Pittsburgh No. 8 or Upper Freeport PA coals. These results are consistent with all the other characterization studies conducted thus far, and with the flotation response.

5.2.3 Induction Time Measurements

Induction time measurements characterize bubble-particle attachment by measuring the minimum time required for the thinning of the liquid film to a point where the film ruptures and recedes, leading to a stable bubble-particle interface. This phenomenon is one of the most important in flotation-based separations as it is a measure of the successful contacts made for a preset contact time and hence a measure of the hydrophobicity of coal.

The instrument used for measuring the induction time is a Yordan-Yoon electronic induction timer. This apparatus provides a measure of the induction time by moving a captive bubble down against a bed of particles for a pre-selected period of time, that can range from 30 microseconds to 2.2 seconds. These times should be considered relative, rather than exact. After each contact, the bubble is visually examined to determine if any coal particles have become attached to the bubble. The power drive for the linear movement of the bubble holder is an

electromechanical device which receives a signal from an electronic pulse generator. Some important device parameters that can affect the measured induction time are the voltage (which controls speed of vertical movement), bubble size (fixed at about 2 mm in diameter), the distance (0.2 to 0.3 mm) travelled by the bubble depending on the voltage selected and the distance between the bubble and the bed of particles (fixed at about 0.1 mm).

In performing a test, a bubble is first formed, then contact with the bed is made and observed through a microscope to determine if a fruitful contact between the bubble and the particles had occurred. A fruitful contact is defined as a contact resulting in at least one particle remaining attached to the bubble after it has been withdrawn from the bed of particles.

The induction time curve is obtained by computing the percentage of fruitful contacts from a minimum of twenty measurements for each selected contact time, as a function of the contact time selected. Figure 5.2 shows a typical induction time curve for 100 x 150 mesh (natural fines) prepared from the Pittsburgh No. 8 research sample. Taking the "induction time" as the contact time that results in fruitful contacts 50 percent of the time, the induction time obtained from these results is 600 microseconds for the Pittsburgh No. 8 coal. Figure 5.3 presents for comparison, the induction time curves for natural fines of Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA research samples. Clearly, the hydrophobicity (induction time decreases as the hydrophobicity increases) of the coals is in the following order:

Illinois No. 6 < Pittsburgh No. 8 < Upper Freeport PA.

In order to complete the surface characterization studies of the three base coals, induction time measurements were performed on freshly-ground material from the research samples. The objective of these tests was to assess the hydrophobicity of the new surface generated after grinding. Because 100 x 150 mesh particles are needed for induction time measurements, samples of Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA coal were prepared by reducing the standard grinding time to 15 minutes in order to obtain a sufficient quantity of material for carrying out these measurements. Induction times were measured for samples that had been deslimed as well as on freshly ground samples.

Figure 5.2 - Induction time curve indicating the percentage of fruitful contacts as a function of contact time in microseconds for a 100 x 150 mesh sample of Pittsburgh No. 8 coal.

Figure 5.3 - Induction time curve indicating the percentage of fruitful contacts as a function of contact time in microseconds for 100 x 150 mesh samples of the three base coals.

The results of the induction time measurements for freshly ground Illinois No. 6 are presented in Figure 5.4. As was observed for the weathered and natural coal fines, the deslimed sample shows a shorter induction time (470 microseconds) than that of the undeslimed sample (500 microseconds), indicating a slight increase in the ability of deslimed particles to attach to bubbles in comparison with the undeslimed sample. Slime-coatings seem to be responsible for the larger induction times of the undeslimed material. However, in the case of freshly-ground samples this difference is smaller than that observed for the natural fines of the research and weathered samples, possibly due to larger amount of slime presented in the weathered samples.

Figure 5.5 presents the percentage of fruitful contacts as a function of contact time for fresh-ground Pittsburgh No. 8 coal, for both the undeslimed and deslimed samples. In the case of Pittsburgh No. 8 coal, the induction time for the deslimed sample was 180 microseconds and that of the undeslimed sample was 265 microseconds. The difference between the induction times of the deslimed and the undeslimed samples is larger than that of Illinois No. 6 coal, probably due to the nature of the coals and the ash constituents. In the case of Upper Freeport PA coal (Figure 5.6), similar behavior to that of Illinois No. 6 and Pittsburgh No. 8 was observed. However, induction times for the deslimed sample (75 microseconds) and the undeslimed sample (105 microseconds) are significantly lower than those of Illinois No. 6 and Pittsburgh No. 8 confirming that Upper Freeport PA coal is by far the most hydrophobic of the three coals.

Advancing contact angles on the three base coals exhibited the following order:

Illinois No. 6 (41 deg.) < Pittsburgh No. 8 (61 deg.) < Upper Freeport PA (63 deg.)

and the average induction times for deslimed 100 x 150 mesh particles of the same coals:

Illinois No. 6 (475 μ sec.) > Pittsburgh No. 8 (180 μ sec.) > Upper Freeport PA (75 μ sec.)

Induction-time measurements of ground coal clearly show that the freshly-ground base coals are more hydrophobic than the natural fines of the unground research coals and the weathered samples, probably due to the fresh surfaces generated during comminution. Again, it seems that the slime-coating phenomenon is responsible for a decrease in the hydrophobicity of the freshly-ground material and the magnitude of this decrease is coal specific. The extent of the

Figure 5.4 - Induction time curves indicating the percentage of fruitful contacts as a function of contact time for Illinois No. 6 coal both undeslimed and deslimed fresh ground samples.

Figure 5.5 - Induction time curves indicating the percentage of fruitful contacts as a function of contact time for Pittsburgh No. 8 coal both undeslimed and deslimed fresh ground samples.

Figure 5.6 - Induction time curves indicating the percentage of fruitful contacts as a function of contact time for Upper Freeport PA coal both undeslimed and deslimed fresh ground samples.

slime coatings has a major role in controlling the time of attachment of particles to bubbles. In agreement with all previous results, the hydrophobicity of the three base coals has the following order for both the undeslimed and deslimed samples tested:

Upper Freeport PA > Pittsburgh No. 8 > Illinois No. 6

5.3 Surface Charge Characterization

The surface charge of the three base coals was studied in terms of their electrokinetic behavior which is related to zeta potential. Zeta or electrokinetic potential is the potential at the shear plane where slip occurs when the particle moves relative to the fluid and is related to the charge density at the shear plane (not the surface charge density). The zeta potential is not only easily determinable experimentally but also is the one that governs the kinetics of interaction of charged droplets with the particles and the attachment of bubbles to particles, both of which govern the flotation performance to a large extent.

Zeta potential measurements were carried out using a Penkem Model 501 Laser Zee Meter. A brief description of the instrument and the measurement technique is given to illustrate the advantage of using this device over Zeta Meter used in the past for the measurement of electrokinetic mobility of the major component in a suspension containing a mixture of heterogeneous particles such as coal and ash components. The Laser Zee meter basically consists of an electrode chamber that is filled with the suspension. The chamber is attached to a stage consisting of a microscope and a control module. The potential is applied between a platinum plated cathode and a molybdenum anode and the movement of the charged particles, illuminated by a laser beam, is monitored by a microscope. A prism, the movement of which is controlled by a knob, is mounted between the objective and the eye piece of the microscope. As the charged particles move towards either the anode or the cathode (depending on the sign of the charge) at a particular velocity (which depends on the magnitude of the charge), the movement of the prism is so adjusted that it moves at the same velocity as the particles. The particles appear stationary when the rate of movement of the prism and the particles are the same. The zeta potential is calculated from the mobility of the particle and their direction of motion. In a mixture of heterogeneous particles, the zeta potential of the major component can be measured by adjusting the rate of prism movement such that majority of the particles remain stationary when viewed through the microscope. In the Zeta Meter the mobility of an individual particle is monitored and the overall mobility is calculated by measuring the mobility of a large number of particles and computing the average mobility. Thus, in the latter method, the mobility of the ash particles will also be considered and if the ash content of the coal is high the average zeta potential will have a substantial contribution from ash components.

The suspension for zeta potential measurements was prepared by stirring 0.075 grams of minus 200-mesh coal in 75 ml of triply distilled water or 2×10^{-3} KNO_3 solution in a glove box maintained under argon atmosphere. Since it was found that pH of the suspension was constantly changing with time, the suspensions were initially adjusted to different pH values, varying in the range 2 to 12, equilibrated for 9 hours (time required to attain a constant pH) and the final pH noted. The zeta potential of the three base coal samples was measured as a function of pH, both in distilled water and in 2×10^{-3} M KNO_3 solution, and the results obtained are presented

Figure 5.7 - Zeta Potential versus pH curve for Illinois No. 6 coal.

in Figures 5.7, 5.8 and 5.9 for the Illinois No. 6, Pittsburgh No. 8, and Upper Freeport PA coals, respectively. These measurements show that the isoelectric point (IEP), that is the point at which zeta potential is reversed, of the three base coals decreases in the following order:

Upper Freeport (pH 5.5) > Pittsburgh No. 8 (pH 3.5) > Illinois No. 6 (pH 2.5).

These results also indicate that for the three coals examined, there is no significant difference between the zeta potentials measured in distilled water and in 2×10^{-3} M potassium nitrate solution. The absence of ionic strength effect, due to KNO_3 addition, on the magnitude of the zeta potential is probably due to the presence of dissolved ions from the mineral matter in coal that can mask the effect of the added electrolyte. An estimate of ionic strength (KNO_3 equivalent) from measured specific conductances of supernatants from the three coal suspensions, both in triply distilled water (TDW) and 2×10^{-3} M KNO_3 solution is shown in Table 5.6. The percentage differences between the zeta potential values in TDW and in potassium nitrate solution are also given in the table for the three coals and it can be seen that these differences are only about 15 percent. The instrument used, the Laser Zee Meter, is not sensitive enough

Figure 5.8 - Zeta Potential versus pH Curve for Pittsburgh No. 8 coal.

Figure 5.9 - Zeta Potential versus pH Curve for Upper Freeport PA coal.

Table 5.6 - Calculated difference between zeta potential values in triply distilled water and 2×10^{-3} M KNO_3 solution for the three base coals.

<u>Coal</u>	<u>Est. Ionic Strength</u> <u>$\times 10^3 \text{M}$ (KNO_3 equiv.)</u>		<u>% Change in</u> <u>zeta potential</u>
	<u>TDW</u>	<u>2×10^{-3} M KNO_3</u>	
Illinois No. 6	0.65	4.94	16
Pittsburgh No. 8	6.87	17.20	17
U Freeport PA	0.13	3.13	15

to differentiate such small changes, according to the manufacturer. The relationship between the zeta potential and the ionic strength is well established and can be found in standard books on colloid science.

Surface charge characteristics of coal in an aqueous environment are determined by surface functional groups such as $-\text{COOH}$ (carboxylic) and ArOH (phenolic) because these groups dissociate and become negatively charged (5). The extent of ionization depends on the solution pH which is determined by hydronium and hydroxyl ions and therefore these are considered to be the potential-determining ions. When coal is oxidized, carboxyl and phenolic groups form and therefore the surface of coal can be expected to become increasingly negative with increase in the degree of oxidation. Thus, the IEP can be expected to decrease with increasing degree of oxidation as experimentally observed by Wen (6) and Fuerstenau and co-workers (7). As discussed later in the chapter, our ESCA study did suggest that the surface of the base coals are in an oxidized state. This observation along with that from the zeta potential results (the trend in IEP values in particular) leads to the inference that the Illinois No. 6 sample has undergone the highest degree of oxidation, followed by the Pittsburgh No. 8 and Upper Freeport PA samples. This inference will in turn lead to the conclusion that the hydrophobicity of Upper Freeport PA coal will be the highest followed by that of Pittsburgh No. 8 and Illinois No. 6 coals, which is supported by induction time measurements, contact angle determination and film flotation studies. However, it should be noted that zeta potential results by themselves do not give conclusive evidence about the relative states of oxidation of the three coals, especially due to the fact that the dissolved ion concentration in the supernatants of these coals are high. This is due to the fact that the adsorption of ions and precipitation of charged, complex hydroxides

of metal ions such as Fe, Al and Mg from solution (which also depend on pH) onto the coal surface will also contribute to the surface charge (8,9). Therefore, interpretation of results in terms of the origin of surface charge in a complex system such as the coal-water suspension is not straight forward. Nevertheless, the information obtained from zeta potential measurements can be taken as supporting evidence for the fact, established from other studies such as film flotation, induction time measurement and contact angle determination, that the hydrophobicity of Upper Freeport PA sample is the highest followed by that of Pittsburgh No. 8 and Illinois No. 6 coal.

An apparent discrepancy to the inference that the surface of Illinois No. 6 sample is more oxidized than the Pittsburgh No. 8 sample, which in turn is in a higher state of oxidation than the Upper Freeport PA sample, is the fact that above pH 8 the zeta potential of Upper Freeport PA coal is most negative, followed by Pittsburgh No. 8 and Illinois No. 6 coal. Similar observations can also be seen from the results of Wen and Sun (9) and Fuerstenau and coworkers (7) who found that above pH 8 coal that has been heated for longer periods has a relatively lower negative zeta potential. Wen and Sun attribute this phenomenon to the solubility of humic acid for which hydronium and hydroxyl ions are not the potential-determining ions (6). They argue that humic acids are the major constituents of organic oxidation products and are formed on coal surfaces, having various molecular weights depending upon the oxidation conditions. They are insoluble in acidic solutions but become soluble in alkaline solutions with the solubility varying as a function of molecular weight. The foregoing discussion suggests the possibility that the surface of Illinois No. 6 coal contains more humic acids which dissolve in alkaline solutions, thereby exposing fresh unoxidized surfaces that are relatively less negative.

5.4 Analysis of Surface Elemental Composition by ESCA

Ultimate analysis of the three base coals indicate that in terms of the bulk carbon-to-oxygen ratio (shown in parentheses) the three base coals follow the order:

Upper Freeport PA (19.8) > Pittsburgh No. 8 (11.5) > Illinois No. 6 (10.5)

Although contact angles, measured using sessile drop and captive bubble techniques as well as calculated from film flotation results, also follow the same order, in terms of size of the angles,

Pittsburgh No. 8 and Upper Freeport PA samples are closer to each other than that of Illinois No. 6 sample. On the other hand, carbon-to-oxygen ratio (which is also a measure of hydrophobicity) of Pittsburgh No. 8 and Illinois No. 6 samples, as shown above, are closer to each other while that of the Upper Freeport PA sample is much higher than that of the other two. One reason for this discrepancy may be that the bulk carbon-to-oxygen ratio is different from that of their surface ratio which is a more relevant parameter for correlation with wettability (contact angle) and flotability. Therefore, the surface carbon-to-oxygen ratio was determined using ESCA (electron spectroscopy for chemical analysis). This technique gives information on the first 50 - 100 Å thick layer on the surface. Samples for ESCA analysis were prepared by grinding a 10-gram batch of minus 28-mesh coal (details of the comminution of the minus 1/4-inch sample to minus 28-mesh can be seen in the section on SEM/EDXRF analysis). Further reduction to minus 400 mesh was carried out with an agate mortar, inside a glove box maintained under an argon atmosphere. About 0.1 gram of this sample was then mounted on a molybdenum mask and loaded into the sample chamber of the photoelectron spectrophotometer. The sample was then bombarded with aluminum x-rays and the spectra recorded. A semi-quantitative analysis of the carbon and the oxygen contents, made using peak areas and the sensitivity factors of the respective elements obtained from the ESCA handbook, indicated that the surface carbon-to-oxygen ratio (wt/wt) decreased in the following order:

Upper Freeport PA (5.2) > Illinois No. 6 (3.6) > Pittsburgh No. 8 (2.6)

Although the hydrophobicity of these coals is expected to vary in the same order, film flotation results and contact angle measurements showed that Pittsburgh No. 8 sample is more hydrophobic than the Illinois No. 6 sample. This discrepancy is possibly due to the differences in the carbon and oxygen contents contributed by the surface inorganic species. Further, carbon-to-oxygen ratios obtained using ESCA are much smaller than those calculated from the results of ultimate analysis. This may result from the fact that the surface of these coals are highly oxidized.

The above discussions indicate that the carbon-to-oxygen ratio could not be used as a measure of the hydrophobicity of these coals, especially when the ash content is high, since the mineral matter is also a source of oxygen.

5.5 Analysis of Surface Functional Groups by DRIFT

Diffuse reflectance infrared Fourier transform (DRIFT) spectroscopic studies were conducted to determine possible differences in the concentration of functional groups responsible for the observed differences in the wettability of the three base coals. Infrared spectra on coal are well documented in the literature (10,11). The diffuse reflectance technique, in particular, has become very popular for handling solid samples since it does not involve any complicated sample preparation procedure as in the preparation of KBr pellet. The only restriction is that the sample must be powdered. Minus 400-mesh samples for the analysis were prepared similarly to the procedure used for ESCA analysis. Other details are already described in the section on mineralogical analysis.

Analysis of surface functional groups of the three base coals by DRIFT spectroscopy showed (Figure 5.10) the presence of major peaks around 2920 cm^{-1} (aliphatic C-H stretching), 1600 cm^{-1} (aromatic C=C), 1460 cm^{-1} (aliphatic C-H bending), and 1040 cm^{-1} (Si-O stretching from clays). A semi-quantitative analysis using peak height measurements indicated that the peak intensities of each of the three organic groups identified are similar in all three coals (Figures 5.11 and 5.12). However, the intensity of the clay mineral peak (Si-O stretching; 1040 cm^{-1}) of Illinois No. 6 coal was ~50 percent more than that of the corresponding peaks of Pittsburgh No. 8 and the Upper Freeport PA samples. Considering that there are no differences in either the nature or the intensity of the organic functional groups identified by the DRIFT analysis, the differences in the hydrophobicity of the three base coals is likely to have arisen from variations in the composition with respect to carbonyl and carboxyl containing functional groups such as ketones ($\text{RC}=\text{O}$), aldehydes (RCHO), carboxylic acids (RCOOH) and ethers (R-O-R).

It might be mentioned here that no oxygen containing organic functional groups were identified in any of the three base coals in the DRIFT analysis although ESCA analysis did indicate considerable surface oxidation of the base coals. It appears that the concentration of the oxygen functional groups in the base coals is not high enough to be detected by infrared analysis. The other possibility is that infrared penetrates deeper than the x-rays used in ESCA so that signals from surface oxygen functional groups are diluted by signals from the bulk. In fact, in

Figure 5.10 - Diffuse reflectance spectra of the three base coals.

Figure 5.11 - Comparison of intensities of aliphatic CH stretching and bending peaks in the three base coals.

Figure 5.12 - Comparison of intensities of aromatic C=C peak of coal and Si-O peak of clay in the three base coals.

comparing the sensitivity of different techniques for characterizing the low temperature oxidation of coal, Huffman and coworkers (12) reported that the carbonyl peaks were clearly observed only after heating the freshly mined Pittsburgh No. 8 coal in air at 50°C and 65 percent relative humidity for 167 days or longer. In this study, it was found that heating the Pittsburgh No. 8 coal in air at 200°C for 30 minutes did result in the appearance of a carbonyl peak (1710 cm⁻¹) of significant intensity which increased with heating time (Figure 5.13). Also, as a function of time, the (C=O) shifted gradually towards the region of the ether group (R-O-R or Ar-O-Ar; 1770 cm⁻¹). Another interesting observation was that an increase in the intensity of the carbonyl peak was accompanied by a decrease in the intensity of the aliphatic CH peak (Figure 5.14). However, there was no measurable change in the intensity of the aromatic C=C peak, suggesting that only aliphatic groups are oxidized at 200°C. Also, it can be seen from Figure 5.14 that beyond 24 hours of heating no further reduction in the intensity of aliphatic group occurred although the intensity of oxygen functional groups continued to increase. This indicates possible further oxidation of primary oxidation product/s beyond 24 hours.

Figure 5.13 - Effect of heating time on the intensity of C=O stretching peak of Pittsburgh No. 8 coal.

Figure 5.14 - Effect of heating time on the intensity of C=C stretching peak (negative peak signifying reduction) from Pittsburgh No. 8 coal.

In summary, the current study showed that DRIFT spectroscopy cannot explain the differences in hydrophobicity among three coals and can detect changes in functional groups only when the coal is subjected to severe oxidation. A comparison of DRIFT results with ESCA analysis points to the possibility that the DRIFT technique is not sufficiently surface selective. However, surface sensitivity of DRIFT can be studied by comparing the changes in carbonyl peaks subjected to oxidation, at say 200°C, with the corresponding changes in carbon-to-oxygen ratio as obtained from ESCA (surface technique) and the ultimate analysis (purely a bulk technique).

5.6 References

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6.0 DEVELOPMENT OF STANDARD GRINDING TEST

The evaluation of various surface control techniques requires a standard test that will enable flotation results to be evaluated and positive effects identified. For advanced coal flotation, this standard test includes the preparation of the sample, comminution of the test feed, and flotation. Without a standardized procedure, the results from the test cannot be statistically evaluated to assess the effectiveness of a given treatment in enhancing flotation behavior.

The advanced flotation of coal is limited by the degree of liberation of pyrite and ash from the coal matrix. The degree of liberation in turn is a function of the particle size distribution. The reduction of particle size of a solid (comminution) results in the production of a set of particles of varying sizes. It is impossible to obtain monosized fine particles by grinding and instead, one obtains a distribution of particle sizes. During grinding, it is the top size and, to some extent, the particle size distribution that can be controlled. Both of these parameters can greatly influence down-stream processing of the coal. The effects of flotation are a result of changes in hydrodynamics and in the surface area of the material. Additionally, reduction of the particle size requires substantial inputs of energy. These factors combined justify an extensive study on the grinding characteristics of coal.

6.1 Development of Standard Grinding Test

The main objective of this part of the investigation was to develop a comminution procedure in the laboratory that would reproduce a meaningful ground product to be used for further characterization and testing. According to the Project Work Plan, the flotation tests were to be conducted using nominal minus 28-mesh and minus 200-mesh material. More specifically, the comminuted material should have a particle size distribution such that 95 percent of the ground product passes through the stated mesh size. Until recently, grinding of coal to very fine sizes was considered to be undesirable and efforts were focused on reducing the total amount of coal fines, minus 28 mesh (600 microns), generated during coal mining and handling. However, with emphasis on the deep cleaning of coal at fine sizes, it is necessary to study the liberation characteristics so that the most effective comminution circuit can be designed for downstream preparation plant separation processes.

Grinding is one of the most important unit operations in mineral processing because size reduction is necessary to liberate valuable minerals from the waste material. Without adequate liberation, it is impossible to effectively separate minerals using physical methods. Although many methods have been tried to improve grinding efficiency and to decrease energy consumption in the grinding of industrial minerals, little fundamental research historically has been reported on the grinding of coal as related to its beneficiation. The main reason is that specific gravity separation processes used for cleaning coarse coals are generally very efficient and economical, if finely disseminated pyrite need not be removed from the coal. In the past, flotation was used to recover only fine particles generated during mining and handling operations, which usually constitute less than 15 percent of the preparation plant feed. Thus, historically there was no need to study the grinding of coal in relation to coal cleaning. However, the grinding of coal is currently gaining increased industrial significance due to a number of factors:

- i) environmental constraints requiring the partial liberation and removal of inorganic sulfur from coal prior to combustion,
- ii) the use of superior quality coals to replace oil and natural gas in combustion equipment, and
- iii) the development of coal gasification/liquefaction processes that require fine clean coal as feed.

Current research objectives in fine coal grinding include:

- i) selectively liberating pyrite and other mineral matter from coals,
- ii) minimizing oxidation of coal surfaces during grinding, and
- iii) increasing the efficiency of grinding.

Apart from the process variables and other external factors, such as adding grinding aids, fine grinding of coal is also affected by the inherent nature of coal formation and metamorphosis. The grindability of coal is a function of its rank and the amount and type of minerals associated with the coal. In general, the grindability of coal increases with rank up to low-volatile bituminous coals and then decreases as one approaches anthracite (1).

This section summarizes detailed tests carried out to establish conditions for both wet and dry grinding of each of the three base coals. Since coal is both chemically and physically heterogeneous, grindability can also be expected to vary over a range, even for coals belonging

to the same rank. The grinding studies discussed here included the selection of a batch laboratory grinding mill and the determination of the time and procedures for dry and wet grinding of the three base coals that would ensure a reproducible product with the proper size distribution.

The coal samples used in the grinding test work were prepared by crushing the new coal in a laboratory jaw mill to a top size of 3/4-inch followed by a secondary crushing to 1/4-inch nominal top size in a laboratory roll crusher. The 1/4-inch coal was then coned and quartered into 30-lb lots and placed in plastic bags. The 30-lb bag was further split into 500 gram lots and stored in plastic bags to be used as needed for feed to the grinding mill. This entire process was conducted in an argon atmosphere to prevent oxidation of the freshly-generated coal surfaces.

6.2 Selection of a Laboratory Grinding Mill

Because of the ability to reproduce the comminuted product, laboratory grinding studies are often carried out in tumbling mills. The grinding media can either be rods (rod mill) or balls (ball mill). Both are suitable for wet as well as dry grinding. Because of the difference in the grinding action in the rod mill as compared to that of the ball mill, coarser particles in the feed create a wedging action between the rods and, hence, are preferentially ground. Such selectivity in grinding yields a product which is relatively narrow in size distribution. That is, with less production of both oversize and ultra fine material. If a ball mill is used for the same task, the size distribution of the product exhibits a long tail at the coarse size. To eliminate this, the product has to be classified to remove the fines. Such a scheme of grinding, called closed-circuit grinding, is generally applied in commercial plants using ball mills. For the purpose of selecting a suitable grinding mill to be used in this project, experiments were carried out using both rod and ball mills.

A comparison of the size distribution of products obtained by grinding Illinois No. 6 coal for four minutes in the mill charged with balls and with rods as grinding media is presented in Figure 6.1, together with the size distribution of the feed material. From these curves, it can be seen that the product from the rod mill indeed contains relatively less coarse particles and has a narrower size distribution than the product from the ball mill even for grind times as short as

Figure 6.1 - Comparison of the size distribution of the products obtained by grinding Illinois No. 6 coal in ball and rod mills for four minutes.

4 minutes. Minimizing the production of very fine particles is advantageous since large amounts of energy are consumed by over grinding and the resulting high surface area associated with fines leads to problems in flotation. For these reasons, and because the size-distribution of the rod mill product is more like the product from a closed ball mill-classifier circuit, rod milling was deemed to be the most suitable method for grinding coal for the laboratory-scale batch flotation studies to be performed for this project.

The laboratory grinding mill, manufactured by Sepor Inc. of Wilmington, California, is constructed of 316 stainless steel. It is 9 inches in diameter and 10 inches in length, and charged with 9 1/2-inch long rods made from the same steel. The mill is driven on parallel rolls at fixed speed. The diameter and number of rods chosen were based on a number of systematic experimental tests carried out early on in this project and are discussed further in this chapter.

Figure 6.2 - Particle size distribution of Illinois No. 6 coal ground in the rod mill for different grind times.

6.3 Effect of Rod Charge on Grinding Time and Particle Size Distribution

Initial experiments were carried out to determine the size distribution of the ground product as a function of grinding time. Experimental operating conditions included charging the mill with 24 rods (9 rods of 3/4-inch, 8 rods of 5/8-inch, and 7 rods of 1/2-inch diameter) and operating it at 60 percent of the critical speed (56 rpm). A representative sample (500 grams) of the minus 1/4-inch secondary crushed material from the roll crusher was used as feed to the rod mill. As an example, the grinding characteristics of Illinois No. 6 coal were studied by grinding for periods ranging from 1 minute to 256 minutes, doubling the grinding time between each successive experiment. The size distribution of the ground product was determined by wet-sieving the ground product on either a 400 or a 200-mesh sieve, drying the oversize and then sieving that material for 15 minutes through a set of Tyler sieves on a Ro-Tap sieve shaker. The results obtained for Illinois No. 6 coal are presented in Figure 6.2. These tests were done in a carbon steel mill (referred to as the old mill) that is identical in size to the mill procured for the project. To prepare a dry-ground product with 95 percent passing through 28 mesh, a grinding

Table 6.1- Number of rods tested and rod mix used for various grinding tests.

<u>Rod Charge</u>	<u>ROD DIAMETER</u>		
	<u>3/4-inch</u>	<u>5/8-inch</u>	<u>1/2-inch</u>
24 rods	9	8	7
36 rods	14	12	10
42 rods	16	14	12
48 rods	18	16	14

time of 8 minutes is required, whereas to obtain a dry-ground product with 95 percent passing through 200 mesh a grinding time of 256 minutes is necessary. Similar experiments were conducted using Pittsburgh No. 8 and Upper Freeport PA coals and the results are presented in Figures 6.3 and 6.4, respectively. By comparison, it can be seen that the time required for similar grinds is the shortest for Upper Freeport PA, followed by Pittsburgh No. 8 and finally Illinois No. 6 coal. Even though all three coals belong to the bituminous category, their grindabilities differ considerably from one another, justifying the extensive experimentation needed for establishing the grinding conditions for these coals.

The grinding time of 256 minutes required to prepare a 200-mesh product of all three base coals is prohibitively high in respect to the duration of the experiment and more importantly, in view of coal surface control. Therefore, steps were taken to reduce the grinding times, especially for the 200-mesh grind. The most obvious option was to increase the number of rods used in the mill. In order to study the effect of the number of rods on the grinding time, dry grinding experiments were carried out with Illinois No. 6 coal using 24, 36, 42, and 48 rods. The grinding time was determined for each of the rod charges such that the end product was 95 percent passing 28 mesh. The rod composition (number of rods) for each charge is given in Table 6.1 and the resulting particle size distributions for Illinois No. 6 coal are presented in Figure 6.5 for nominal 28-mesh grinds. When only 24 rods are used, unusual grinding behavior results, in that less coarse material is broken but a greater proportion of fine material is produced. However, with 36 rods or more, the particle size distribution remains virtually unchanged. Due to the extremely short grinding times required with 36 or more rods, and the associated lack of control

Figure 6.3 - Particle size distribution of Pittsburgh No. 8 coal ground in the rod mill for different grind times.

Figure 6.4 - Particle size distribution of Upper Freeport PA coal ground in the rod mill for different grind times.

Figure 6.5 - Effect of the number of rods on the particle size distribution of the ground product of Illinois No. 6 coal conforming to 95 percent passing through 28-mesh.

on the production of fine particles, 24 rods were considered to be an appropriate choice for the preparation of the 28-mesh feed material.

However, the long grinding time required to produce minus 200-mesh material necessitated the use of more rods in the mill and tests were also carried out to determine the effect of the rod charge on the production of minus 200-mesh material, using Illinois No. 6 coal. The grinding experiments were performed under both wet and dry conditions. Test results for the 8-minute grinds under dry and wet conditions are presented in Figure 6.6. As can be seen from these plots, the production of minus 200-mesh material increases as the number of rods is increased and stabilizes near 42 rods. The calculated struck volume (excluding coal charge) increases from about 19 to 43 percent as the number of rods is increased from 24 to 48 as shown in Table 6.2. The levelling off of fines production with rod charges greater than 42 rods is due to the decrease in the mill torque as the total filled volume of the load approaches and exceeds half of the total mill volume. Since the particle size distributions produced by 42 and 48 rods for both dry and wet grinding are nearly identical, and because the grind times are roughly equal, a charge of 42 rods

Figure 6.6 - Effect of the number of rods on the production of minus 200-mesh Illinois No. 6 coal for a grind time of 8 minutes.

was considered suitable for both wet and dry grinding of coal to 95% passing 200-mesh. The results presented in Figures 6.7 and 6.8 show how the grind time effects the percent passing 28 mesh and 200 mesh for two different rod charges. From these graphs the standard grinding times were chosen.

Grinding experiments were conducted with the other two base coals (Pittsburgh No. 8 and Upper Freeport PA) which also established similar conclusions. The results for Upper Freeport PA coal are given in Figures 6.9 and 6.10 for the 200-mesh dry and wet grind conditions, respectively.

Table 6.2 - Measured struck volume for the various rod charges used.

<u>Rod Charge</u>	<u>Struck Volume, % of Mill</u>
24	19.1
42	37.3
48	43.3

Figure 6.7 - Effect of the number of rods on 28-mesh material produced during the dry grinding of Illinois No. 6 coal as a function of grinding time.

Figure 6.8 - Effect of the number of rods on 200-mesh material produced during the dry grinding of Illinois No. 6 coal as a function of grinding time.

Figure 6.9 - Effect of the number of rods on 200-mesh material produced during the dry grinding of Upper Freeport PA coal as a function of grinding time.

Figure 6.10 - Effect of the number of rods on 200-mesh material produced during the wet grinding of Upper Freeport PA coal as a function of grinding time.

Table 6.3 - Standard wet and dry grinding conditions for the three base coals.

<u>PARAMETER</u>	<u>28-MESH GRIND</u>	<u>200-MESH GRIND</u>
No. of rods	24	42
Rod mix 3/4", 5/8", 1/2", resp.	9, 8, 7	16, 14, 12
Water charge (wet grinding)	700 ml	700 ml
Coal charge	500 g	500 g
<u>Grind Times</u>		
Illinois No. 6		
Dry	8 min.	48 min.
Wet	3 min. 20 sec.	15 min.
Pittsburgh No. 8		
Dry	4 min. 50 sec.	30 min.
Wet	3 min.	16 min. 30 sec.
Upper Freeport PA		
Dry	2 min. 50 sec.	21 min. 30 sec.
Wet	1 min. 45 sec.	11 min. 20 sec.

In conclusion, a charge consisting of 24 rods is sufficient to obtain a ground product having 95 percent passing 28 mesh while 42 rods are needed to produce 200-mesh material. The results also show that when using 42 rods there is considerable reduction in the grinding time required when the coal is ground dry. A 24-rod charge was used to prepared the 28-mesh feed because the grinding time was too short if 42 rods were used for this purpose. These standard grinding conditions that were developed were then used to prepare the feed for the flotation experiments.

6.4 Standard Grinding Procedure

The experimental work presented in the previous sections led to the development of standard grinding procedures to be used to produce and reproduce a coal sample of an indicated top size and similar particle size distribution to be used as feed material to the flotation cell. The average grinding times were established for the three base coals using 24 rods for the 28-mesh grind and 42 rods for the 200-mesh grind. The mill charge consisted of 500 grams of coal for both wet and dry conditions. For wet grinding, 700 ml of water was also charged to the mill. The mill was operated at 56 rpm (60% critical speed). The grinding time and rod charges are summarized in Table 6.3. Repeatability of the grind time was checked periodically at each location as well as the reproducibility at other locations.

Table 6.4 - Size distribution and ash analysis of Upper Freeport PA 28-mesh standard ground material.

Size	WET GROUND			DRY GROUND		
	Cum. Wt. %			Cum. Wt. %		
	Weight %	Passing	Ash %	Weight %	Passing	Ash %
+ 28 M	4.0	100.0	16.5	5.0	100.0	20.3
28 x 48 M	31.7	96.0	12.4	23.6	95.0	14.7
48 x 65 M	16.4	64.3	11.7	14.4	71.4	12.5
65 x 100 M	11.1	47.9	11.7	15.4	57.0	12.0
100 x 150 M	10.8	36.8	11.1	10.1	41.6	9.6
150 x 200 M	4.3	26.0	13.7	7.3	31.5	12.4
- 200 M	21.7	21.7	14.4	24.2	24.2	12.8
TOTAL	100.0		12.7	100.0		

Detailed compositional analyses of different separate size fractions of Upper Freeport PA coal produced by the standard grinding procedure have also been performed. The results, which are summarized in Tables 6.4 and 6.5 show that the ash content of the coarser material is slightly higher than that of the finer material, indicating that the high-ash material does not grind as easily as the carbonaceous material.

An analysis of the liberation potential of the coals must consider the particle size distributions of the washability samples with that produced by the standard grinding procedure. The size distributions of the Illinois No. 6 coal from the washability testing, and the dry- and wet-ground products are shown in Figure 6.11. The particle size distribution of the minus 200-mesh washability sample is nearly linear with no tail of fines or particles larger than 200 mesh, indicating that the sample had been pulverized and then sieved repeatedly to reduce the coal to

Table 6.5 - Size distribution and ash analysis of Upper Freeport PA 200-mesh standard ground material.

Size	WET GROUND			DRY GROUND		
	Cum. Wt. %			Cum. Wt. %		
	Weight %	Passing	Ash %	Weight %	Passing	Ash %
+ 200 M	5.8	100.0	13.8	5.7	100.0	13.9
200 x 270 M	15.1	94.2	11.1	19.8	94.3	13.0
270 x 325 M	8.0	79.1	9.9	5.7	74.5	9.8
325 x 400 M	5.3	71.1	14.6	5.1	68.8	15.2
- 400 M	65.8	65.8	12.9	63.7	63.7	12.0
Total	100.0		12.6	100.0		12.3

Figure 6.11 - Size distributions of Illinois No. 6 coal prepared for washability testing and 200-mesh wet and dry-ground products prepared by the standard grinding procedure.

the required top size (closed-circuit size reduction). Both the standard wet and dry-ground samples have particle size distributions that are close to that of the washability sample with the dry-ground product slightly finer and the wet-ground product slightly coarser. This suggests that the pyritic sulfur rejection predicted by the washability data is theoretically attainable with a perfect flotation separation.

6.5 Relative grindabilities of base coal samples

Taking as a reference the time required for the dry grinding of Illinois No. 6 coal to obtain a product 95% finer than 28 mesh or 200 mesh, the relative grindabilities (in mass/unit energy) of the three base coals were calculated, and are tabulated in Table 6.6. In all cases, Upper Freeport PA is the easiest to grind. Illinois No. 6 tends to require the most energy to achieve a given size reduction, either by dry or wet grinding. However, the wet 200-mesh grind for Pittsburgh No. 8 appears to require more energy than Illinois No. 6 for the same grind size.

Table 6.6 - Relative grindabilities* of the three base coals.

	DRY GRIND		WET GRIND	
	28 mesh	200 mesh	28 mesh	200 mesh
Illinois No. 6	1.0	1.0	2.4	3.2
Pittsburgh No. 8	1.7	1.6	2.7	2.9
Upper Freeport PA	2.8	2.7	4.6	4.2

*Grindability in mass/unit energy relative to dry grinding Illinois No. 6 coal to $95 \pm 1\%$ minus 28 mesh or minus 200 mesh.

6.6 Cleanability of Standard Ground Coals

The washability analyses presented in Chapter 4 of this report were made on samples that had been stage-ground to the desired top size. As a result, the particle size distribution tends to be much narrower than that of samples ground to the same top size in either rod or ball mill in a single stage. Therefore, it was of interest to determine the cleanability of the base coal that had been prepared by the standard grinding procedure. However, instead of performing the actual washability analysis of the ground coal samples, a new technique using scanning electron microscopy with automated image analysis techniques (SEM-AIA) was used to predict the washability of the ground coal samples. The SEM-AIA study was completed at Ames Laboratory by W.E. Straszheim et al. (2) and the results for the three base coals dry ground to 200 mesh, following the standard grinding procedure, are presented in Figures 6.12 - 6.14.

The SEM-AIA results for Illinois No. 6 (Figure 6.12) predict that the pyritic sulfur in the standard ground coal is not liberated as well as that in the samples used in the washability study. However, the SEM-AIA results suggest that a gravity separation of close to the 90-90 goal should be obtainable with the rod-mill ground product. The difference between these two curves may be due to a variety of reasons, the most obvious being the different methods of size reduction and the different techniques used for assessing the cleanability. In spite of the difference, the results are surprisingly close. The predicted cleanability of the ash-forming minerals (also shown in Figure 6.12) by the SEM-AIA is greater than the actual washability results at all CMR values. Again, these differences are not surprisingly due to the differences in the sample preparation and the methods of assessing the cleanability.

The comparison of washability and SEM-AIA results for Pittsburgh No. 8 and Upper Freeport PA coals (Figures 6.13 and 6.14) are analogous to those of Illinois No. 6. The predicted

Figure 6.12 - Measured and SEM-AIA predicted washability results for 200-mesh Illinois No. 6 coal.

Figure 6.13 - Measured and SEM-AIA predicted washability results for 200-mesh Pittsburgh No. 8 coal.

Figure 6.14 - Measured and SEM-AIA predicted washability results for 200-mesh Upper Freeport PA coal.

pyritic sulfur rejection is slightly lower than the actual washability analysis with the SEM-AIA results falling just short of the 90-90 goal. The predicted ash washability for both coals is greater than the actual washability results at all CMR values.

In summary, the SEM-AIA analyses predicts that cleanability of the standard dry-grinding procedure for 200 mesh coal is only slightly different from that of the washability. In general, for all three coals the pyritic sulfur rejection is slightly less than the washability results and the ash rejection is slightly better. The difference may be due to differences in the particle size distribution of the various samples as well as differences in the two techniques used to assess the cleanability of the ground coals.

6.7 QA/QC Grinding Tests

Coal feed samples for flotation studies were comminuted using a rod mill following the standard grinding test procedures to obtain a fraction $95 \pm 1\%$ passing 28 mesh or 200 mesh. To fulfill the QA/QC plan, a comparison of grinding data obtained at Berkeley, Columbia and

Table 6.7 - Results of QA/QC tests of the standard grinding procedure showing the percent passing the desired grind size for grinding conducted at various sites for the three base coals.

	28 MESH		200 MESH	
	<u>Dry</u>	<u>Wet</u>	<u>Dry</u>	<u>Wet</u>
Pittsburgh No. 8				
Berkeley	95.8	96.1	96.0	97.5
Columbia	95.0	96.0	94.0	96.0
Utah	--	--	--	--
Illinois No. 6				
Berkeley	95.8	95.0	95.9	95.5
Columbia	--	--	--	--
Utah	--	--	--	--
Upper Freeport PA				
Berkeley	97.3	97.7	92.9	95.5
Columbia	96.7	95.4	92.8	92.0
Utah	95.0	95.0	95.0	94.9

Utah was made to check the percent of material passing a given size after comminuting the three base coals at the three different sites for the standard grinding times. These QA/QC Standard Grinding Test results are presented in Table 6.7 and show that the three research sites produced a ground product that contained about 95% passing the desired top size.

6.8 References

- (1) R.R. Klimpel, "Fine Coal Grinding in Fine Coal Processing", S.K. Mishra and R.R. Klimpel (eds), Noyes Publications, Park Ridge, New Jersey, pp. 19-58 (1987),
- (2) W.E. Straszheim, R. Markuszewski and G.M. Orem, "Prediction and Evaluation of Coal Cleanability," Fossil Energy Quarterly Report for January 1, 1990 through March 31, 1990, DOE Contract W-7405-Eng-82.

7.0 DEVELOPMENT OF THE STANDARD FLOTATION TEST

One of the most important goals at the onset of the research project was to develop a standard flotation procedure that would ensure repeatable and reproducible tests. Further, such a standard test had to enable the evaluation of the effects of various grinding and flotation reagent schemes, which was part of the overall objective of this research. The same standard flotation test was also used to evaluate the effect of coal weathering on its flotation response.

7.1 Standard Flotation Test Procedure

Standard flotation test procedures were developed using operating parameters within reasonable ranges so that the influence of grinding and surface modifying reagents could be studied. The standard test is not an optimal test with respect to any single parameter, but rather a test conducted at conditions generally suitable for coal flotation and pyrite rejection. Preliminary tests confirmed that a number of flotation conditions should be made coal-specific rather than kept constant for all coals. For example, since the hydrophobicity of the coals differs significantly, the collector and frother dosages are coal-specific and hence were selected to suit the flotation of each coal. Also, both collector and frother additions are somewhat dependent on the flotation feed size of a given coal. Therefore, frother and collector dosages were selected individually for the two feed sizes (28 mesh and 200 mesh). Tests were also conducted to establish if different dosages were required for dry grinding and wet grinding for a given grind size. While a number of operating parameters were set at ranges considered suitable from past experience, others were determined empirically with the three base coals. These included the pulping time and the collector and frother conditioning times.

In coal flotation, a combination of collector and frother is generally used. Coal flotation collectors are usually nonpolar oils obtained from petroleum distillation or coke oven plants and, therefore, have low solubility in water. Typical nonpolar collectors are kerosene, fuel oil, crude petroleum, and coal-tar distillates. As the molecular weight of these reagents increases, considerable agitation is required to disperse the reagent and to ensure its spreading on the solid surfaces. Kerosene (with a boiling range of 170-270°C), a widely used collector in coal flotation, is a mixture of saturated paraffinic hydrocarbons whose exact composition depends on the source

of the crude and the distillation operation. In order to ensure a consistent quality of the collector during the course of this research, dodecane, with a molecular weight close to that of kerosene, (boiling point 216°C) was chosen as the standard collector.

Frothers are heteropolar reagents that strongly absorb at the air-water interface and thereby act as strong foaming agents. MIBC (methylisobutylcarbinol or 4-methyl-2-pentanol), and pine oil (a terpeneol) are common frothers used in coal flotation. The choice of the frother depends on the consistency of performance, availability and price. MIBC has been found to be the most commonly used frother in flotation practice, not only for the foregoing reasons, but also because it produces a fine-textured froth that is not very persistent. Therefore, MIBC was selected as the standard frother for the purpose of the present work.

The operating parameters for flotation were separated into two parts (a) those which are common for the three base coals and (b) those which are coal specific. The common or fixed set of parameters includes rotor speed, froth paddle speed, pulp density, air rate, pulp level in the cell, and conditioning and flotation times for the standard tests. In order to minimize the effect of regional water quality, distilled water was used at all three locations. These operating conditions were the same for the two grinds (wet and dry) tested under this project.

Because the contract required the use of a standard flotation machine for the standard flotation test, a Denver Laboratory Model flotation machine, which is commonly used and is available at all three research locations, was selected. However, the flotation tank or cell used is of special design which allows for mechanical froth removal and air flow control. Each of the three universities is equipped with identical flotation cells. The flotation cell was made of plexiglass following the design developed by the DOE PETC⁽¹⁾. Figure 7.1 shows the two-paddle design of the cell which enables the removal of froth from two sides of the cell. Typically, for low-grade ores, a 4-liter cell is used for rougher flotation while a 2-liter cell is used for cleaner flotation. In coal flotation, the 2-liter cell was selected for the standard single-stage batch flotation tests. Figure 7.2 shows the automatic flotation apparatus which has been used at DOE PETC as well as at the University of Utah for coal flotation. The entire experimental set up for the Standard Flotation Test is similar to that shown with the exception of pulp level control which is maintained manually to a predetermined level marked on the plexiglass cell. Our

Figure 7.1 - Two-paddle flotation cell used in The Standard Flotation Test.

Figure 7.2 - Standard flotation apparatus showing the flotation cell and Denver Laboratory Model Flotation Machine.

conclusions were that it would be simpler to maintain pulp level manually because of the long flotation times used in the standard tests.

The mechanical scrapers, attached to a small motor, can be adjusted by a rheostat to a speed between 0 and 40 rpm. The air flow rate is controlled by a diaphragm pump connected to a 3-way valve and flow meter assembly, allowing for easy control of the flotation gas composition. This automated flotation machine is reported to provide more reproducible data than can be obtained with a standard Denver laboratory flotation machine without automatic froth removal (1).

The standard flotation test conditions are given in Table 7.1 for all three base coals. As can be seen from this table, there are numerous variables associated with the flotation that could affect the final clean coal product. Fortunately, previous investigations have made some of the choices much less complicated than if the general behavior of coal flotation were not already known. For instance, the collector (dodecane), frother (MIBC) and flotation pulp density were all chosen from previous experimental work (2).

Even though the selection of the collector and frother were based on previous flotation work, the dosages are particularly sensitive to the type of coal and the extent of comminution, and therefore necessitated empirical testing to determine reasonable dosage levels. Accordingly, the dosages selected as standard are different for each of the three base coals and were determined by following a statistical design. Levels of flotation reagents were chosen so that a reasonable yield was obtained, but not necessarily optimized. In general, each coal was floated first with no reagents and then with increasing amounts of frother and collector. Flotation tests were conducted with both 28-mesh and 200-mesh standard grinds to establish the collector and the frother dosages.

The standard frother and collector dosages were determined for the three base coals by systematically varying either the frother or collector and observing the effect on the flotation product. The standard dosages were chosen at the start of a plateau region in the dosage/recovery curves so that when the effects of surface modifiers and grinding conditions were being evaluated, both beneficial and detrimental effects could be observed. The results of some of these tests for wet-ground Pittsburgh No. 8 coal are shown in Figure 7.3.

Table 7.1 - Standard flotation test conditions.

<u>PARAMETER</u>	<u>28 M GRIND</u>	<u>200 M GRIND</u>
<u>Feed Sample</u>		
Grinding sample weight	500 g	500 g
Flotation test feed	125 g $\pm$ 5 g	125 g $\pm$ 5 g
Method of splitting	riffle	riffle
Max. time before flotation	2 hr	2 hr
Flotation time	5 minutes	5 minutes
<u>Flotation Equipment and Operating Conditions</u>		
machine type	Denver machine with 2 liter DOE cell	
machine rotor speed	1200 rpm	1200 rpm
froth paddle speed	36 rpm	36 rpm
H ₂ O	1000 ml	1000 ml
Level make up method	manual	manual
Cell water level below lip	20 mm	20 mm
Aeration rate	4 lit/min	4 lit/min
<u>Conditioning Times</u>		
Pulping time (minimum)	2 min	4 min
Pulp level adj. and pH meas. time	1 min	1 min
Collector conditioning time	1 min	1 min
Frother conditioning time	3 min	3 min
Total conditioning time	7 min	7 min
<u>Collector dosage</u> (100 μ l = 1.20 lb/T)		
Illinois No. 6		
dry grind	4.80 lb/T (400 μ l)	5.76 lb/T (480 μ l)
wet grind	4.80 lb/T (400 μ l)	5.76 lb/T (480 μ l)
Pittsburgh No. 8		
dry grind	1.92 lb/T (160 μ l)	2.16 lb/T (180 μ l)
wet grind	2.02 lb/T (168 μ l)	1.92 lb/T (160 μ l)
Upper Freeport PA		
dry grind	0.24 lb/T (20 μ l)	0.48 lb/T (40 μ l)
wet grind	0.24 lb/T (20 μ l)	0.48 lb/T (40 μ l)
<u>Frother dosage</u> (100 μ l = 1.30 lb/T)		
Illinois No. 6		
dry grind	1.17 lb/T (90 μ l)	1.17 lb/T (90 μ l)
wet grind	1.17 lb/T (90 μ l)	1.17 lb/T (90 μ l)
Pittsburgh No. 8		
dry grind	0.39 lb/T (30 μ l)	0.39 lb/T (30 μ l)
wet grind	0.39 lb/T (30 μ l)	0.30 lb/T (23 μ l)
Upper Freeport PA		
dry grind	0.26 lb/T (20 μ l)	0.21 lb/T (16 μ l)
wet grind	0.26 lb/T (20 μ l)	0.21 lb/T (16 μ l)

Flotation results with Illinois No. 6 showed that this coal was the most difficult to float and required large dosages of collector due to its hydrophilicity. Since the collector is somewhat nonselective, the pyrite rejection decreases with increasing collector dosage. The net effect is a poor flotation separation. The results of the standard flotation results for Illinois No. 6 are given

Figure 7.3 - Effect of collector and frother dosage on the flotation yield of 200-mesh wet-ground Pittsburgh No. 8 coal.

in Table 7.2 and are characterized by low flotation efficiencies for the 200-mesh grinds as compared to the other two base coals.

Initial experiments with Pittsburgh No. 8 indicated that this coal was fast floating and yields of 75-80% were achieved easily. Therefore, separate collector and frother dosages for both sizes, ground dry and wet, were selected in order to keep recoveries in this range. The results of tests using the selected frother and collector dosages are given in Table 7.3.

Table 7.2 - Results of standard flotation tests using Illinois No. 6 coal.

Grind Size	Grinding Mode	FLOTATION PRODUCT ANAL				PERCENT REJ		El _H
		Yield %	CMR %	Ash %	Pyr S %	Ash %	Pyr S %	
28 M	Dry	74.6	79.8	9.6	1.76	53.8	48.9	28.7
28 M	Wet	88.0	92.6	11.0	1.82	37.6	37.5	30.1
200 M	Dry	60.4	64.8	9.3	1.36	63.7	68.0	32.8
200 M	Wet	77.0	83.3	8.4	1.57	57.6	52.7	36.0

Table 7.3 - Results of the standard flotation tests using Pittsburgh No. 8 coal.

Grind Size	Grinding Mode	FLOTATION PRODUCT ANAL				REJECTION		EI _H
		Yield %	CMR %	Ash %	Pyr S %	Ash %	Pyr S %	
28 M	Dry	82.0	87.3	6.9	1.99	55.0	42.1	29.4
28 M	Wet	86.0	91.0	7.5	2.09	48.7	36.3	27.3
200 M	Dry	77.5	83.3	6.0	1.37	63.0	62.3	45.6
200 M	Wet	75.7	82.4	4.9	1.20	70.5	67.8	50.2

The results of tests for 200-mesh wet-ground Upper Freeport PA coal indicated that this coal floated the easiest of the base coals, and that reasonable yields could be obtained with no collector at all. Three combinations of frother and collector, each giving equivalent efficiencies, were considered for 200-mesh wet-ground Upper Freeport PA coal, the most readily floatable of the three base coals.

- (a) Frother only (MIBC),
- (b) Balanced addition of frother and collector, and
- (c) Higher collector and lower frother dosage.

The first option produced a high quality concentrate, but the flotation yield was very low. On the other hand, the third option produced a high yield, but the quality of the concentrate was compromised. Thus, the second option with a balanced collector and frother addition was selected as the most suitable reagent conditions for the standard flotation test. By a similar procedure, the reagent dosages for the other three grinds were determined and are given in Table 7.4.

Table 7.4 - Results of standard flotation tests for Upper Freeport PA coal.

Grind Size	Grinding Mode	FLOTATION PRODUCT ANAL				REJECTION		EI _H
		Yield %	CMR %	Ash %	Pyr S %	Ash %	Pyr S %	
28 M	Dry	81.1	86.5	6.7	0.69	56.6	65.5	52.0
28 M	Wet	75.5	80.8	6.4	0.63	61.4	70.6	51.4
200 M	Wet	59.1	63.3	6.3	0.67	70.2	75.6	38.9
200 M	Dry	68.2	73.8	5.3	0.51	71.1	78.5	52.3

Figure 7.4 - Flotation yields of the three base coals as a function of flotation time using the standard collector and frother dosages tabulated in Table 7.1.

Just as the collector and frother dosages affect the clean coal product so too can the flotation time, which is controlled by the kinetics of the process. A balance must be struck between the quality of the product and the recovery. Figure 7.4 shows the flotation yield-vs-time relationship for the three base coals with the standard collector and frother additions. Yields close to eighty percent were desired so that a ninety percent Btu recovery could be achieved yet any improvements in the selectivity of the flotation process could be detected. The results presented in Figure 7.4 show that at flotation times of five minutes and the standard collector and frother additions for each coal, this criterion is met. This flotation time was used for all three coals, both 28-mesh and 200-mesh, dry- and wet-ground material.

These test conditions and procedures developed here were used consistently throughout this project and changed only as needed for experiments carried out with other reagents in the testing of surface modifying reagents. The test conditions summarized in Table 7.1 comprise the standard flotation test procedures that were used in this project for the three base coals.

7.2 QA/QC for Standard Flotation Tests and Coal Analyses

In order to check the reproducibility and repeatability of the standard flotation tests and coal analyses, an extensive QA/QC program was undertaken. In these studies, 500-gram samples (minus 1/4-inch in size) of Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA coals were wet-ground to 95% minus 200 mesh using our standard grinding procedures. The ground samples were then split into four parts using a stainless steel riffle (8-1/4 inch x 1 inch in size with 32-1/4 inch chutes) and three of the four splits were used as feed for the standard flotation tests. The fourth split was reserved for analysis.

Subsequently, each sample of flotation feed concentrate, and tailings (from floated samples) was analyzed for ash, total sulfur, and pyritic sulfur. In order to provide enough material for QA/QC analyses, the three flotation concentrates and tailings for each coal were combined and homogenized. The feed and the combined concentrate and tailing for each coal were then split into four parts. One part was sent to Columbia University and one to the University of Utah to conduct proximate, total-sulfur, and pyritic-sulfur analyses while the remaining samples were kept at Berkeley for further characterization studies.

Tables 7.5 to 7.7 give the flotation yield, ash and total-sulfur contents of the flotation products of the three base coals. The pyritic-sulfur content of the flotation products of Illinois No. 6 coal and the efficiency index for each separation are also presented in Table 7.7. The results given in these tables clearly show that the standard flotation tests are quite reproducible. The yields obtained for each coal differ by no more than 4.4 percentage points at most (Upper Freeport PA) and only 3.2 percentage points for Illinois No. 6 coal. The differences in the efficiency index for Illinois No. 6 coal is less than 2 points. The ash and total sulfur contents of the flotation products are also quite consistent in the different tests. Furthermore, the average flotation yields obtained in this study, 76.8, 75.9 and 75.3% for Illinois No. 6, Pittsburgh No. 8, and Upper Freeport PA coals, respectively, agree very well with the average values reported in our First Annual Report (77.4% for Illinois No. 6, 76.2% for Pittsburgh No. 8 and 73.0% for Upper Freeport PA coals).

Table 7.8 compares the weight and composition of the split samples from the 200 mesh wet grinds of the three base coals. The ash, total-sulfur, and pyritic-sulfur contents of parts A, B and

Table 7.5 - Reproducibility of the standard flotation tests (dodecane 5.75 lb/T, MIBC 1.16 lb/T, pH 6.6) of 200-mesh, wet-ground Illinois No. 6.

<u>Split Sample</u>	<u>Product</u>	<u>Yield, %</u>	<u>Ash, %</u>	<u>Tot S, %</u>	<u>Pvt. S %</u>	<u>El_H</u>
Part A	Conc.	78.3	8.4	3.54	1.15	47.7
	Tail	21.7	38.2	8.72	7.20	
Part B	Conc.	77.0	8.1	3.51	1.04	49.7
	Tail	23.0	37.0	8.50	6.96	
Part C	Conc.	75.1	8.0	3.50	1.10	49.4
	Tail	24.9	35.7	8.53	7.12	
Part D	Feed		14.9	4.66	2.37	

Table 7.6 - Reproducibility of the standard flotation tests (dodecane 1.92 lb/T, MIBC 0.30 lb/T, pH 3.8) of 200-mesh, wet-ground Pittsburgh No. 8 coal.

<u>Split Sample</u>	<u>Product</u>	<u>Yield, %</u>	<u>Ash, %</u>	<u>Tot S, %</u>
Part A	Conc.	73.8	5.2	2.73
	Tail	26.2	29.1	8.61
Part B	Conc.	76.4	5.5	2.79
	Tail	23.6	31.3	9.13
Part C	Conc.	77.7	5.4	2.72
	Tail	22.3	31.9	9.02
Part D	Feed		11.6	4.29

Table 7.7 - Reproducibility of standard flotation tests (dodecane 0.48 lb/T, MIBC 0.21 lb/T, pH 3.3) of 200-mesh, wet-ground Upper Freeport PA coal.

<u>Split Sample</u>	<u>Product</u>	<u>Yield, %</u>	<u>Ash, %</u>	<u>Tot S, %</u>
Part A	Conc.	73.7	6.3	1.27
	Tail	26.3	34.0	4.61
Part B	Conc.	74.1	6.4	1.26
	Tail	25.9	34.3	4.78
Part C	Conc.	78.1	6.8	1.29
	Tail	21.9	38.5	5.57
Part D	Feed		13.9	2.28

Table 7.8 - Comparison of split samples from the 200-mesh wet grind of the three base coals.

Coal	Part	Weight grams	Ash %	Tot S %	Pyr S %
Illinois No. 6	A*	111.4	14.8	4.66	2.46
	B	112.3	14.7	4.66	2.40
	C	104.2	14.9	4.75	2.60
	D	115.6	14.9	4.66	2.37
Pittsburgh No. 8	A	121.9	11.5	4.27	
	B	122.1	11.6	4.29	
	C	115.1	11.3	4.12	
	D	117.3	11.6	4.29	
* Upper Freeport PA	A	120.6	13.6	2.15	
	B	118.4	13.7	2.17	
	C	114.4	13.7	2.23	
	D	123.0	13.9	2.21	

*Results of splits A, B and C were calculated from the analyses of the flotation concentrates and tailings.

and C in this table were calculated from the analyses of the flotation concentrate and tailings. The results show that the split samples are quite uniform with respect to the ash content, total-sulfur, and pyritic-sulfur contents. The maximum difference in the weight of the split samples was 12 grams, which did not significantly effect the flotation behavior as can be seen from the results given in Tables 7.5 to 7.7.

Table 7.9 compares the results of ash, total-sulfur and pyritic-sulfur analyses conducted at the various laboratories on the flotation feed, concentrate and tailings of the three base coals. The results in this table show that the values are consistent and reproducible at each of the three test locations, especially the ash and total-sulfur contents. The difference in the pyritic-sulfur contents is also within the range of experimental error.

Table 7.10 shows the combustible material recovery (CMR), ash and pyritic-sulfur rejection, and the efficiency index resulting from standard flotation as calculated from the analyses obtained by the different research groups using two different methods. In the first method, the results were calculated from the analyses of the flotation feed and concentrate. In the second method, the results were calculated from the analyses of the flotation concentrate and tailing. It can be seen from this table that the results obtained using these two calculation methods using the experimental results from three different test locations are quite consistent. The maximum

Table 7.9 - Comparison of ash, total-sulfur, and pyritic-sulfur analyses conducted at different universities.

<u>Coal Sample</u>	<u>Product</u>	<u>Location</u>	<u>Yield, %</u>	<u>Ash, %</u>	<u>Tot S, %</u>	<u>Pvt S, %</u>
Illinois No. 6	D Feed	Berkeley		14.9	4.61	2.25
		Columbia		14.5		2.16
		Utah		14.9	4.73	2.48
	A, B, C Con	Berkeley	76.8	7.9	3.50	1.11
		Columbia		8.1		0.96
		Utah		8.1	3.57	1.01
	A, B, C Tail	Berkeley	23.2	36.5	8.39	6.92
		Columbia		35.8		7.23
		Utah		36.7	8.86	7.64
Pittsburgh No. 8	D Feed	Berkeley		11.5	4.27	2.91
		Columbia		11.3		2.59
		Utah		11.4	4.10	2.82
	A, B, C Con	Berkeley	75.9	5.5	2.70	1.38
		Columbia		5.5		1.18
		Utah		5.4	2.68	1.26
	A, B, C Tail	Berkeley	24.1	30.7	8.97	8.18
		Columbia		30.0		7.88
		Utah		30.5	9.33	9.01
Upper Freeport PA	D Feed	Berkeley		13.9	2.30	1.68
		Columbia		13.9		1.37
		Utah		13.9	2.21	1.56
	A, B, C Con	Berkeley	75.3	6.5	1.27	0.59
		Columbia		6.7		0.50
		Utah		6.5	1.25	0.51
	A, B, C Tail	Berkeley	24.7	35.4	4.88	4.85
		Columbia		35.2		5.10
		Utah		35.3	5.08	4.86

difference in the efficiency index is only 4.3 points between the two different methods and 6.4 points when the data obtained from different test locations are used. The difference in the efficiency index is mainly due to variation in pyritic-sulfur rejections.

7.3 Efficiency Index Comparison

Because the Standard Flotation Test is the basis for judging the effect of various coal preparation and treatment procedures, data handling becomes an important issue. The efficiency index used to rank the grinding environments and coal surface modification treatments is an important tool in identifying positive effects and the index that is most appropriate depends on

Table 7.10 - Comparison of CMR, ash rejection, pyritic-sulfur rejection, and efficiency index of three base coal calculated using different methods based on the results obtained by the different research groups.

Coal Sample	Location	CMR %		Ash Rej %		Pyr S Rej %		EI _H	
		M1	M2	M1	M2	M1	M2	M1	M2
Illinois No. 6	Berkeley	83.1	82.8	59.2	58.2	62.1	65.3	45.2	48.1
	Columbia	82.5	82.6	57.1	57.2	65.9	69.5	48.4	52.1
	Utah	82.9	82.8	58.2	57.8	68.7	69.6	51.6	52.4
Pittsburgh No. 8	Berkeley	81.8	81.1	63.8	64.0	64.0	65.3	45.1	46.4
	Columbia	80.9	81.0	63.1	63.4	65.4	68.0	46.3	49.0
	Utah	81.1	81.1	64.1	64.2	66.1	69.4	47.2	50.5
Upper Freeport PA	Berkeley	81.7	81.5	64.6	64.0	73.6	72.9	55.3	54.4
	Columbia	81.6	81.4	63.7	63.3	72.5	77.0	54.1	58.4
	Utah	81.7	81.5	64.5	64.0	75.4	75.8	57.1	57.3

M1 - Results based on the analyses of the flotation feed and concentrate.

M2 - Results based on the analyses of the flotation concentrate and tailings.

the recovery and rejection values. Extensive work has been directed at delineating the most appropriate index for our purposes and these findings are discussed in this section.

An analysis was carried out to compare two definitions of efficiency, namely the Department of Energy's efficiency index (EI_{DOE}):

$$EI_{DOE} = \frac{\% \text{ Pyritic Sulfur in tail}}{\% \text{ Pyritic Sulfur in clean coal}} \times \text{Yield}$$

and the well-known Hancock separation efficiency (EI_H)(3):

$$EI_H = (100\% \times \frac{\text{wt conc}}{\text{wt feed}}) \times (\frac{\% \text{ mineral in conc}}{\% \text{ mineral in feed}} - \frac{\% \text{ gangue in conc}}{\% \text{ gangue in feed}})$$

For the purposes of this illustrative discussion, undesirable or gangue material will be equated with pyritic sulfur and the rest of the desirable material will be equated with the Btu¹, in accordance with the efficiencies being defined for a binary system of valuable and waste products.

The Hancock efficiency varies between -100% and +100% where negative numbers correspond to unfavorable separation processes and positive numbers to desirable separations.

¹The Efficiency Index reported throughout this report is the Hancock efficiency and is based on the combustible material recovery (CMR) instead of Btu Recovery. The relationship between CMR and Btu recovery will be discussed in the next section.

Further, EI_H is linear with respect to both gangue rejection with constant yield and to yield with constant gangue rejection. A perfect separation, that is, all valuable mineral being recovered and all gangue being rejected, will give $EI_H = 100$; a sampling operation where no concentration of mineral or gangue takes place gives $EI_H = 0$; and a truly imperfect separation (where all the mineral is rejected and all the gangue is recovered) gives $EI_H = -100$. This method of measuring efficiency is, perhaps, more easily visualized than the EI_{DOE} method because EI_{DOE} is non-linear with respect to both gangue rejection with constant yield and to yield at constant gangue rejection; further the EI_{DOE} can vary between zero and infinity. Both these aspects lead to difficult visualization of the real effectiveness of a separation corresponding to a number given by the EI_{DOE} .

The standard flotation test results on Illinois No. 6 coal, for 200-mesh dry grind gave an average yield of 60.4% and a pyritic sulfur rejection of 68.0%. For purposes of this illustration of how the efficiency indices vary with recovery and rejection, a standard value of 60% yield and 80% pyritic sulfur rejection will be used. The pyritic sulfur in feed is taken to be 2.70%. For a perfect separation all the pyritic sulfur reports to the tailings, and the denominator of the Department of Energy's EI equation goes to zero, and EI_{DOE} goes to infinity. At the other extreme, if the separation is perfectly deleterious all the pyritic sulfur reports to the concentrate, the numerator of the above equation goes to zero, as does the efficiency index EI_{DOE} . All values in between these extremes are possible.

However, for the standard test result as used in this illustration, $EI_{DOE} = 360$. Figure 7.5 shows how keeping the CMR constant at 60% and allowing the pyritic sulfur rejected to vary, affects the indices. EI_{DOE} asymptotically approaches each axis at the extremities, which means at low pyritic sulfur rejection a small perturbation hardly changes EI_{DOE} yet at high pyritic sulfur rejection an equally small perturbation of the pyritic sulfur rejection gives a large change in EI_{DOE} . The Hancock efficiency varies linearly with pyritic sulfur rejection, 100% pyritic sulfur rejection coinciding with $EI_H = 60$. No matter how good the pyritic sulfur rejection is, the Hancock efficiency cannot give a value of 100% and cannot indicate a perfect separation because the recovery of the valuable component is less than 100%. EI_{DOE} on the other hand tends to infinity as pyritic sulfur rejection tends to 100% irrespective of the yield and irrespective of the

Figure 7.5 - Comparison of the Hancock and DOE efficiency indexes as a function of pyritic sulfur rejection at a constant CMR of 60 percent.

recovery (except for the case when yield = 0%) thus a perfect separation gives $EI_{DOE} = \text{infinity}$. Both this dichotomy of the asymptote, and the non-linearity of the EI_{DOE} graph may be regarded as counter intuitive and is difficult to visualize as a method of grading a separation.

Many of the same comments regarding the Department of Energy efficiency index and the Hancock efficiency apply equally well when pyritic sulfur is kept constant and the yield is allowed to vary. EI_{DOE} asymptotically tends to zero and infinity as the yield tends to zero and 100% respectively, as can be seen from Figure 7.6. Again EI_H is seen to vary linearly while EI_{DOE} undergoes an exponential increase. Again EI_H cannot indicate a perfect separation, that is it cannot take a value of 100 (not all the pyrite is rejected) whereas EI_{DOE} tends to infinity, even though the separation is not perfect.

Finally, the standard flotation test results give $EI_{DOE} = 360$. Figure 7.7 shows the locus of points of pyritic sulfur rejected vs. yield that also gives the value $EI_{DOE} = 360$. It should be noted that for a pyritic sulfur rejection of 100% and a yield of zero, EI_{DOE} is indeterminate and not 360, there is a discontinuity at that point. However, the main point is that in the region

Figure 7.6 - Comparison of the Hancock and DOE efficiency indexes as a function of yield at a constant pyritic sulfur rejection of 80 percent.

Figure 7.7 - Comparison of the pyritic sulfur rejection as a function of yield at constant efficiency for Hancock and DOE indexes.

of standard test results the value $El_{DOE} = 360$ can mean many different things in regards to how much pyritic sulfur is rejected and how much of the combustible matter is recovered. The value of El_{DOE} has little intrinsic meaning.

A similar criticism of the Hancock efficiency can be made, that is, one value of El_H does not correspond to one separation result. However, El_H does correspond to how much material has gone through the ideal separator as defined by Schulz (4).

Schulz proposed defining the efficiency of a separation with the aid of his ideal separator. The ideal separator, as the name suggests, is a notional machine that recovers all desirable material and rejects all undesirable material. Any separation process can be regarded as a system in which some of the material goes through the ideal separator the rest of the material bypasses the machine and is separated randomly between recovery and rejection. The Hancock efficiency is such a measure. For the standard test results, $El_{DOE} = 360$ and $El_H = 41.1$ which could be interpreted as 41.1% of the material passing through the ideal separator. The rest of the material may report to either tailings or concentrate, so even for the Hancock separation efficiency, a single value can be ambiguous.

In summary, then, it would seem that Hancock has produced a measure of efficiency that corresponds to some notion commensurate with intuition, whereas the Department of Energy's efficiency is more difficult to interpret. Both methods, though, have their shortcomings as a measure of efficiency because one value can correspond to a number of different states. Each different separation scenario is not defined by an efficiency unique to itself, but may share the same efficiency as any number of different separations. Maybe the main conclusion to be drawn is that one number alone cannot properly describe the efficiency of a separation process. At least El_H is positive for favorable separations, negative for unfavorable separations, in each of these regions varies from 0% to 100%, and returns a value of zero if nothing happens. Therefore, the Hancock efficiency was used throughout this project to assess the degree of flotation separation for all flotation tests.

7.4 Release Analysis

The objective of this research project was to control the surface properties of coal and pyrite to improve their separation efficiency by flotation. In order to evaluate the best obtainable separation with the standard flotation test conditions, it was necessary to establish a reference curve, based on surface properties, analogous to gravity-based washability tests. The liberation analysis obtained from the washability data is based on a bulk property, that is, the density of the particles, whereas froth flotation is controlled by surface properties which may differ from the bulk properties. The best separation efficiency achievable under the standard flotation conditions was determined by using the Release Analysis Procedure of Dell (5). Analogous to the use of washability curves for assessing the potential for cleaning coal by gravity methods, the flotability curve determines the flotation-cleaning potential of a coal. However, the latter results are limited to the reagents used to obtain the flotability curve. This procedure, using our standard reagents, gave a range of products comprised of the cleanest concentrate (naturally hydrophobic material) to the cleanest tailings (composed mainly of ash and pyrite) that can be achieved with MIBC as frother and dodecane as collector.

7.4.1 Illinois No. 6

The experimental procedure used for the release analysis of Illinois No. 6 coal is shown in Figure 7.8. The flotation time for each flotation step and the number of cleaning stages were selected in order to minimize mechanical carryover or entrapment of non-desirable mineral matter in the concentrates and to minimize coal content in the final tailing. Following the same criteria, for the first stage (4 runs), the standard reagent dosage for Illinois No. 6 coal was used. For the release analysis test, dodecane was added to each of the flotation steps, whereas MIBC was added only to the first, third and fourth. For the second and third cleaning stages, smaller amounts of both dodecane and MIBC were added only to the last flotation step of each of these successive cleaning stages. No reagents were added to the fourth cleaning stage. Similar procedures were also used in the case of Pittsburgh No. 8 and Upper Freeport PA coals. The results of the release analysis are plotted in Figure 7.9 and show that there is a large difference between the washability and release analysis, indicating that surface-based separation processes are different

Figure 7.8 - Release analysis flotation scheme for Illinois No. 6 coal.

Figure 7.9 - Release analysis, washability data, and SEM-AIA data showing CMR (or BTU) recovery as a function of pyritic sulfur rejection for Illinois No. 6 coal.

from those based on bulk properties, such as gravity (washability test). This is particularly critical when locked particles are present. Figure 7.9 also shows SEM-AIA predicted flotability data for dry-ground 200 mesh Illinois No. 6 coal. These data, taken from a study by W. Straszheim et al. (6), were obtained by considering the fractional coverage of particle surface by coal and minerals. The SEM-AIA flotability curve slightly under predicts the pyritic sulfur rejection at CMR values greater than 80 percent and over predicts at values less than 80 percent. The hydrophobicity of pyrite, either inherent or induced, is probably the cause of this difference.

The release analysis procedure produces a concentrate that has been cleaned numerous times, whereas the standard flotation test does not reclean the reject. This difference makes it difficult to compare results. To alleviate this problem, one-step flotation kinetic tests were conducted, using the Time Release Analysis Procedure described by Cavallaro et al. (7). In this procedure, a set of concentrates of varying flotation rates is obtained. It is expected that the most hydrophobic clean coal will float at the fastest rate but with the lowest yield, and as the yield increases, mineral matter reporting to the concentrate will also increase, thus, all the concentrates will lie over the same separation line. The froth was collected at 0.5, 1, 3, and 5 minutes.

The results of these kinetic standard tests are shown in Figures 7.10 and 7.11. The pyritic sulfur and ash rejection for one-step flotation is substantially less than for the release analysis. Pyritic sulfur rejection decreases by about 20 percent at yields of 80 percent. This indicates that the efficiency of the flotation tests reported here will understate the maximum separation efficiency obtainable if the products had been cleaned.

7.4.2 Pittsburgh No. 8

Figure 7.12 shows the experimental procedure used for Pittsburgh No. 8. Flotation time and the number of flotation stages were selected in order to evaluate the best possible separation obtainable with the standard flotation reagent dosages. The first stage was run four times and the concentrates combined in order to obtain enough material in each subsequent stage and to minimize the effects of pulp dilution during cleaning. The pH was the natural pH of the slurry.

The flotation results are presented for Pittsburgh No. 8 coal in Figure 7.13. Even with extensive cleaning and recleaning, the flotation separation does not coincide with the washability

Figure 7.10 - Ash rejection as a function of (100-Yield) for Illinois No. 6 coal for the kinetic flotation tests, release analysis and washability studies.

Figure 7.11 - Pyritic sulfur rejection as a function of (100-Yield) for Illinois No. 6 for the kinetic flotation tests, release analysis and washability studies.

Figure 7.12 - Release analysis flotation scheme for Pittsburgh No. 8 coal.

Figure 7.13 - Release analysis, washability data, and SEM-AIA data showing CMR (or Btu) recovery as a function of pyritic sulfur rejection for Pittsburgh No. 8 coal.

results. This highlights the difference between gravity and surface-based separations where a particle primarily of pyrite may be quite hydrophobic, depending on the amount of coal present on the surface. In order to overcome this phenomenon, a number of methods including the addition of reagents that will selectively alter the hydrophobicity of these coal/pyrite particles or regrinding the middlings may be necessary.

Also included in Figure 7.13 are the SEM-AIA predicted flotability data for dry-ground 200 mesh Pittsburgh No. 8 coal. As can be seen from this figure, the SEM-AIA technique predicts substantially better pyritic sulfur rejection at CMR values below 85%. In fact, the predicted values are close to those obtained in the washability study. Again, this difference may be due to the pyrite being partially hydrophobic. At CMR values above 85% the release analysis and the SEM-AIA predicted curves are almost identical.

Several tests were performed with varying amounts of frother added to the cell at constant collector addition to directly compare with the standard flotation results. Since the same frother and collector were used for these tests, the results lie on a single separation curve for one-step flotation tests. Figures 7.14 and 7.15 present the pyritic sulfur and ash rejection for Pittsburgh No. 8 coal as a function of the recovery taken from the washability, Release Analysis and one-step flotation tests.

These curves show that at yields of 80% and less, the pyritic sulfur rejection associated with one-step flotation about 10 percent less than that of the release analysis at equivalent CMR values. This difference is expected to be machine dependent and should decrease if a cleaning step is added.

7.4.3 Upper Freeport PA

In the case of Upper Freeport PA, the release analysis was performed according to the experimental procedure shown in Figure 7.16 and the results are plotted in Figure 7.17. As in the case of the other two coals there is a substantial difference between the release analysis results and the washability results. The SEM-AIA predicted results are like those for Pittsburgh No. 8 coal in that they slightly over predict the pyritic sulfur rejection at CMR values greater than 90 percent and are almost identical to the release analysis at CMR's above this value.

Figure 7.14 - Ash rejection as a function of (100-Yield) for Pittsburgh No. 8 coal for the one-step flotation tests, release analysis and washability studies.

Figure 7.15 - Pyritic sulfur rejection as a function of (100-Yield) for Pittsburgh No. 8 coal for the one-step flotation tests, release analysis and washability studies.

Figure 7.16 - Release analysis flotation scheme for Upper Freeport PA coal.

Figure 7.17 - Release analysis, washability data, and SEM-AIA data showing CMR (or BTU) recovery as a function of pyritic sulfur rejection for Upper Freeport PA coal.

7.4.4 Summary

The release analysis curves obtained for the three base coals predict that the best flotation separations with only dodecane and MIBC will be substantially less than that obtained during the washability studies. The inferior separation made by the surface-based process, flotation, may be due to a number of different factors, including the smearing of ash and pyrite particles making them hydrophobic, liberation problems and some inherent or induced hydrophobicity of pyrite. The inefficiencies of the one-step flotation process compared to the release analysis is probably due to mechanical entrainment and entrapment of pyrite and ash particles.

In general, the SEM-AIA predicted flotability curves agree quite well with the release analysis data at high CMR values (80% - 90%). At low CMR values, the SEM-AIA data fall almost on top of the data obtained in the gravity based washability studies, which for all three coals always gives better pyritic sulfur rejection than the release analysis. The over prediction at low CMR values may be due to a natural or induced hydrophobic surface on pyrite. This may be particularly critical with particles that contain semi-liberated pyrite.

7.5 Correlation Between Combustible Material Recovery (CMR) and Btu Recovery

Throughout this project, the yield and CMR have been used to evaluate process efficiency in order to limit the need for more elaborate and costly heating-value (Btu) analysis. Therefore, the correlation between the yield, CMR, and Btu recovery needs to be established empirically.

The calorific value of the feed samples and selected flotation products of the three base coals were determined, and the data were treated by regression and correlation analysis. The relationship between CMR and Btu recovery is shown in Figures 7.18, 7.19 and 7.20 for Illinois No. 6, Pittsburgh No. 8, and Upper Freeport PA coals, respectively. It can be seen that a good linear relationship was obtained for each coal. These results concur with Appendix A-2 of our First Annual Report that asserts that CMR values slightly underestimate Btu recovery, and therefore, Efficiency Indices based on CMR values under report the flotation efficiency based on Btu recovery.

Figure 7.18 - Relation between Combustible Material Recovery and Btu Recovery for Illinois No. 6 coal.

Figure 7.19 - Relation between Combustible Material Recovery and Btu Recovery for Pittsburgh No. 8 coal.

Figure 7.20 - Relation between Combustible Material Recovery and Btu Recovery for Upper Freeport PA coal.

7.6 Relation Between Total Sulfur and Pyritic Sulfur

Desulfurization of coal prior to combustion is becoming important for the effective utilization of this important energy resource without polluting the environment. A significant portion of the total sulfur present in many raw coals is inorganic in nature, occurring mostly as pyrite and sometimes with small amounts of sulfate, with the remainder being organic sulfur intimately bound to the organic coal matrix (8). Because the degree to which sulfur can be reduced in coal by physical methods, such as flotation or gravity separation, is directly related to the portion of pyritic sulfur in the coal, determination of the pyritic sulfur and ash content must be routinely carried out for large numbers of products in order to evaluate the separation efficiency of the process.

The commonly accepted method for determining sulfur forms in coal is that designated in the United States as the ASTM D2492 Method (9). In this procedure, the sulfate sulfur is first analyzed by extracting minus 60-mesh coal with hot dilute HCl and determining the sulfate sulfur gravimetrically as BaSO_4 . Pyritic sulfur is then obtained by extracting the HCl-treated coal with

hot 2M HNO₃ for 30 minutes and determining the iron present in the extract by atomic absorption spectrophotometry. The amount of pyritic sulfur in the coal is calculated from the iron content, assuming that all the extracted iron came from FeS₂ in the coal. Organic sulfur is then calculated as the difference between the total sulfur and the sum of the sulfate and pyritic sulfur.

Several alternative methods for the determination of pyritic sulfur content of coal have been proposed (10-14). Many of these methods involve the reduction of the inorganic sulfur forms to hydrogen sulfide, allowing for the direct determination of pyritic sulfur. Hyman and Rowe (14) proposed a different method that combines the techniques of thermogravimetry and magnetometry to determine the pyritic sulfur in coal and lignite by reducing the pyrite to metallic iron. However, among these methods, the ASTM procedure is still the most straightforward, and many tests have confirmed its utility on raw coals. However, because a large number of concentrates and tailings can be produced in coal flotation experimentation or in coal cleaning operations, determination of the pyritic sulfur content of large numbers of samples by the ASTM procedure is prohibitively time-consuming. In this report, we present a way to estimate the pyritic sulfur content of the coal samples from their total sulfur and proximate analysis.

In order to evaluate the separation efficiency for the physical processing of a given coal, the pyritic sulfur and ash contents of the desired and waste products must be determined. From the pyritic sulfur and ash contents of the concentrates and tailings (waste) produced by the separation process, the pyritic sulfur rejection, ash rejection and combustible material recovery can be calculated. The unique feature of these various products is that they come from the same coal and, therefore, the organic coal matrix and the mineral matter components present in the products are the same except that the percentage of each component in these products is different. Since physical separation methods cannot reduce the organic sulfur in the coal matrix, the organic sulfur content contained in the combustible material in these process streams can be considered to be constant, that is

$$(\% \text{ Tot. S} - \% \text{ Pyr. S} - \% \text{ Sul. S}) / (100 - \% \text{ Ash}) = \text{constant.} \quad (1)$$

Therefore, if the sulfate sulfur constant is small, the pyritic sulfur content of the coal process sample should be a linear function of its total sulfur and ash content and can be represented by the following equation:

$$\% \text{ Pyt. S} = A (\% \text{ Tot. S}) + B (\% \text{ Ash}) + C \quad (2)$$

Theoretically, A should be equal to one, B should be related to the organic sulfur content and C should be a function of both the organic and sulfatic sulfur contents. However, empirically the values of A, B and C can be determined by statistical analysis, by measuring the pyritic sulfur content using the ASTM procedure and also the total sulfur and ash contents of several flotation products of the same coal. After the parameters in the equation have been established for a particular coal, the pyritic sulfur content of other flotation products of the same coal can be calculated based on their total sulfur and ash content using the equation. Because determination of pyritic sulfur of coal using the ASTM procedure requires considerable time (at least two hours for each sample) and determination of total sulfur and ash content of coal using microprocessor-controlled machines is rapid and reliable, significant time and effort can be saved with our proposed procedure.

7.6.1 Testing the Proposed Procedure with Flotation Samples

This hypothesis was tested with three bituminous coals, Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA. The flotation products were obtained by floating minus 200-mesh coal with dodecane and MIBC at natural pH. The pyritic sulfur content of these flotation products were determined using ASTM Procedure D2492. The total sulfur and ash contents were determined with a LECO SC-132 Sulfur Analyzer and a LECO MAC-400 Proximate Analyzer. Both machines are controlled by a microprocessor. Typically, determination of the total sulfur of one coal sample required less than five minutes using this sulfur analyzer. Duplicate tests were conducted for each sample in order to obtain more accurate results and the average value was used in the regression analysis. Using the proximate analyzer, 19 coal samples can be analyzed for their ash content at one time.

For Illinois No. 6 coal, 20 flotation products were analyzed and the corresponding regression equation is

$$\% \text{ Pyr. S} = 0.9751 (\% \text{ Tot. S}) + 0.0128 (\% \text{ Ash}) - 2.6064 \quad (3)$$

with a correlation factor R square equal to 0.9934. For Pittsburgh No. 8 coal, 55 flotation products were analyzed and the regression equation is

$$\% \text{ Pyr. S} = 0.6213 (\% \text{ Tot. S}) + 0.0935 (\% \text{ Ash}) - 0.9203 \quad (4)$$

with a correlation factor R square of 0.9958. For Upper Freeport PA coal, 16 flotation products were analyzed and the regression equation is

$$\% \text{ Pyr. S} = 0.9413 (\% \text{ Tot. S}) + 0.0108 (\% \text{ Ash}) - 0.7820 \quad (5)$$

with a correlation factor R square of 0.9983.

Figures 7.21, 7.22 and 7.23 are plots of the determined pyritic sulfur content following the ASTM procedure versus the values calculated from the total sulfur and ash contents using Equations 3, 4 and 5 for the three coals. These figures confirm that a very good linear relationship does exist between the three parameters. Also the difference in the pyritic sulfur rejection by flotation calculated using the estimated pyritic sulfur and the determined values are within an accuracy of one percentage point.

To test the relationship further, an additional six samples of Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA coals were analyzed for their pyritic sulfur, total sulfur and ash contents. Tables 7.11, 7.12, and 7.13 compare the pyritic sulfur content determined experimentally using

Table 7.11 - Comparison of the pyritic sulfur content determined experimentally using the ASTM procedure and calculated using Eq. 3 for flotation samples of 200-mesh Illinois No. 6 coal.

Sample Specification	Ash %	Tot. S %	PYRITIC SULFUR, %		
			Exp.	Calc.	Differ.
0-0.25 min. Conc.					
+ 400 mesh	6.74	3.46	0.94	0.85	0.09
- 400 mesh	10.12	4.10	1.52	1.52	0.00
0.25-0.5 min. Conc.					
+ 400 mesh	6.26	3.38	0.80	0.77	0.03
- 400 mesh	9.09	3.96	1.38	1.37	0.01
0.5-1 min. Conc.					
+ 400 mesh	6.34	3.34	0.79	0.73	0.06
- 400 mesh	9.43	4.04	1.47	1.45	0.02

Figure 7.21 - Correlation between the pyritic sulfur content determined experimentally using the ASTM Procedure and the value calculated using Equation 3 for flotation samples of Illinois No. 6 coal.

Figure 7.22 - Correlation between the pyritic sulfur content determined experimentally using the ASTM Procedure and the value calculated using Equation 4 for flotation samples of Pittsburgh No. 8 coal.

Figure 7.23 - Correlation between the pyritic sulfur content determined experimentally using the ASTM Procedure and the value calculated using Equation 5 for flotation samples of Upper Freeport PA coal.

the ASTM procedure with the value calculated using Eqs. 3, 4 and 5 for Illinois No. 6 Pittsburgh No. 8 and Upper Freeport PA coals, respectively. These results show clearly that the calculated pyritic sulfur contents of these coal samples agree well with the determined values and

Table 7.12 - Comparison of the pyritic sulfur content determined experimentally using the ASTM Procedure and calculated using Eq. 4 for Flotation samples of 200-mesh Pittsburgh No. 8 coal.

Sample Specification	Ash %	Tot. S %	PYRITIC SULFUR, %		
			Exp.	Calc.	Differ.
-0.25 min. Conc.					
+ 400 mesh	4.75	2.42	1.04	1.03	0.01
- 400 mesh	6.07	2.72	1.29	1.34	-0.05
0.25-0.5 min. Conc.					
+ 400 mesh	4.45	2.28	0.97	0.91	0.06
- 400 mesh	5.48	2.62	1.18	1.22	-0.04
0.5-1 min. Conc.					
+ 400 mesh	4.21	2.30	0.93	0.90	0.03
- 400 mesh	5.32	2.63	1.16	1.21	0.05

Table 7.13 - Comparison of the pyritic sulfur content determined experimentally using the ASTM Procedure and calculated using Eq. 5 for Flotation samples of 200-mesh Upper Freeport PA coal.

<u>Sample Specification</u>	<u>Ash %</u>	<u>Tot S %</u>	<u>PYRITIC SULFUR, %</u>		
			<u>Exp</u>	<u>Calc</u>	<u>Differ</u>
0-0.25 min. Conc.					
+ 400 mesh	5.52	1.14	0.37	0.35	0.02
- 400 mesh	6.27	1.44	0.64	0.64	0.00
0.25-0.5 min. Conc.					
+400 mesh	5.20	1.13	0.35	0.34	0.01
- 400 mesh	6.08	1.41	0.62	0.61	0.01
0.5-1 min. Conc.					
+ 400 mesh	4.95	1.03	0.31	0.24	0.07
- 400 mesh	6.04	1.34	0.58	0.54	0.04

further confirms the linear relationship between the pyritic sulfur, total sulfur and ash contents. The difference between the experimental pyritic sulfur and the calculated value is within the precision of the ASTM procedure (9). Therefore, the method described here can be safely used to calculate the pyritic sulfur content of coal flotation products from their total sulfur and ash contents once the constants in the linear relationship have been evaluated.

7.6.2 Testing the Proposed Procedure with Washability Samples

The proposed procedure for estimating the pyritic sulfur content from the total sulfur and ash content was also tested with washability samples of Pittsburgh No. 8 coal. Both washability tests and the analysis of the different density fractions of the coal reported in this section were conducted by Geochemical Laboratory in Somerset, Pennsylvania. In these tests, the coal samples were first crushed to different top size namely 4 inch, 1.5 inch, 1/8 inch and 28 mesh. The sink-float tests were conducted according to ASTM procedure (D4371) using heavy liquids with the specific gravity of 1.30, 1.35, 1.40, 1.60 and 1.80, respectively. The different density fractions of the coal obtained from the sink-float tests were analyzed using the ASTM procedures for their calorific value, ash, total sulfur and pyritic sulfur contents.

Figure 7.24 - Correlation between the pyritic sulfur content determined experimentally using the ASTM Procedure and the value calculated using Equation 6 for washability samples of Pittsburgh No. 8 coal.

The regression equation for Pittsburgh No. 8 coal obtained from 20 washability samples is the following:

$$\% \text{ Pyr. S} = 1.023 (\% \text{ Tot. S}) + 0.0120 (\% \text{ Ash}) - 1.252 \quad (6)$$

with a correlation factor R square of 0.9922. Figure 7.24 plots the determined pyritic sulfur content versus the values calculated from the total sulfur and ash content using Equation 6 for washability samples of Pittsburgh No. 8 coal. This figure confirms that a very good linear relationship does also exist between the three parameters for coal samples obtained from the washability tests. To test Equation 6 further, the washability results of three size fractions of Pittsburgh No. 8 coal were used to calculate the pyritic sulfur content of the various density fractions of the coal. Tables 7.14, 7.15, and 7.16 compare the determined pyritic sulfur content using ASTM procedure and the calculated values based on the ash and total sulfur content using Equation 6 along with the pyritic sulfur rejection calculated using both the determined and the calculated pyritic sulfur content. It can be seen from Tables 7.14 through 7.16 that the

Table 7.14 - Comparison of the pyritic sulfur content determined experimentally using the ASTM Procedure and calculated using Eq. 6 for washability samples of 200x28 mesh size fraction of Pittsburgh No. 8 coal (1.5 inch top size).

Density	Yield %	Ash %	Tot S %	PYR S, %		PYR S REJ, %		
				Exp	Calc	Exp	Calc	Differ
1.30	50.2	3.04	1.73	0.51	0.55	92.97	92.50	0.47
1.35	66.1	3.89	1.98	0.78	0.82	85.84	85.38	0.46
1.40	72.3	4.43	2.15	0.97	1.00	80.73	80.49	0.24
1.60	81.9	5.79	2.51	1.38	1.39	68.95	69.41	-0.46
1.80	85.4	6.73	2.69	1.58	1.58	62.93	63.60	-0.67
Total	100.0	15.30	4.67	3.64	3.71			

calculated values agree well with the determined value for all samples in the three size fractions. The maximum difference in the pyritic sulfur content and the pyritic sulfur rejection is 0.07% and 1.1% respectively, which is within the precision of the ASTM procedure (11). These results show that the proposed procedure can be safely used to calculate the pyritic sulfur content of coal washability products from their total sulfur and ash contents. The main advantages of this method is that it is reliable and rapid because the total sulfur content of a coal sample can be analyzed within five minutes and multiple analyses for the same sample can be done easily.

However, it is necessary to point out that the values of the regression constants in Equation 4 for flotation samples differ from their values in Equation 6 for washability samples even though both samples are Pittsburgh No. 8 coal. Tables 7.17 and 7.18 compare the pyritic sulfur content of Pittsburgh No. 8 coal calculated using the flotation equation and washability equation for both flotation samples and washability samples. Clearly, the error involved in

Table 7.15 - Comparison of the pyritic sulfur content determined experimentally using the ASTM Procedure and calculated using Eq. 6 for washability samples of 28 mesh x 1/8 inch size fraction of Pittsburgh No. 8 coal (1.5 inch top size).

Density	Yield %	Ash %	Tot S %	PYR S, %		PYR S REJ, %		
				Exp	Calc	Exp	Calc	Differ
1.30	47.8	3.66	1.79	0.74	0.73	87.18	87.48	-0.29
1.35	74.9	4.68	2.32	1.20	1.18	67.43	68.15	-0.72
1.40	82.6	5.21	2.54	1.44	1.41	56.90	57.97	-1.07
1.60	89.8	6.21	2.89	1.79	1.78	41.76	42.31	-0.55
1.80	92.5	6.96	3.05	1.95	1.95	34.65	34.81	-0.16
Total	100.0	11.28	3.80	2.76	2.77			

Table 7.16 - Comparison of the pyritic sulfur content determined experimentally using the ASTM Procedure and calculated using Eq. 6 for washability samples of 1.5 x 1/8-inch size fraction of Pittsburgh No. 8 coal (1.5 inch top size).

Density	Yield %	Ash %	Tot S %	PYR S, %		PYR S REJ, %		Differ
				Exp	Calc	Exp	Calc	
1.30	27.2	5.22	2.54	1.44	1.41	85.86	85.84	0.02
1.35	72.2	5.84	2.86	1.78	1.74	53.60	53.49	0.11
1.40	84.8	6.44	3.13	2.09	2.03	36.02	36.50	-0.48
1.60	92.7	7.53	3.41	2.40	2.33	19.68	20.33	-0.65
1.80	95.1	8.28	3.46	2.46	2.39	15.54	16.16	-0.61
Total	100.0	11.15	3.74	2.77	2.71			

calculating the pyritic sulfur content of flotation samples using the washability equation is much larger than that using the flotation equation. Similarly, the error involved in calculating the pyritic sulfur content of washability samples using the flotation equation is much larger than that using the washability equation. Furthermore, the pyritic sulfur content calculated using Eq. 6 (the washability equation) is consistently higher than that calculated using Eq. 4 (the flotation equation). This difference may be due to a number of reasons. For example, pyrite in Pittsburgh No. 8 coal begins to oxidize within a short time period. The washability tests were conducted early in the program and the flotation campaign was begun after eighteen months had elapsed. Furthermore, preliminary flotation results obtained early in the investigation were between the two. The parameters in the equations should be obtained for similar conditions, for example, coal samples of equal age. Also, since the washability samples were separated in nonaqueous media whereas the flotation samples were tested in aqueous

Table 7.17 - Comparison of the pyritic sulfur content determined experimentally using the ASTM Procedure and calculated using Eqs. 4 and 6 for washability samples of 28 x 200 mesh size fraction of Pittsburgh No. 8 coal (1.5 inch top size).

Density	Yield %	Ash %	Tot S %	Exp	PYRITIC SULFUR, %	
					Calc Eq. 4 (flot.)	Calc Eq. 6 (wash.)
1.30	50.2	3.04	1.73	0.51	0.44	0.55
1.35	66.1	3.89	1.98	0.78	0.67	0.82
1.40	72.3	4.43	2.15	0.97	0.83	1.00
1.60	81.9	5.79	2.51	1.38	1.18	1.39
1.80	85.4	6.73	2.69	1.58	1.38	1.58
Total	100.0	15.30	4.67	3.64	3.41	3.71

Table 7.18 - Comparison of the pyritic sulfur content determined experimentally using the ASTM Procedure and calculated using both Eq. 4 and Eq. 6 for flotation samples of 100-mesh Pittsburgh No. 8 coal.

Sample Specification	Ash %	Tot S %	PYRITIC SULFUR, %		
			Exp —	Calc Eq. 4 (flot.)	Calc Eq. 6 (wash.)
0-0.25 min. Conc.					
+ 200 mesh	5.40	2.83	1.32	1.34	1.71
200x400 mesh	5.81	3.26	1.70	1.65	2.15
- 400 mesh	6.80	3.04	1.58	1.60	1.94
0.25-0.5 min. Conc.					
+200 mesh	5.35	2.70	1.33	1.26	1.57
200x400 mesh	5.53	3.12	1.63	1.54	2.01
- 400 mesh	6.22	2.96	1.47	1.50	1.85

environments, due to the solubility of sulfate sulfur, the constants in the regression equation will change. Therefore, in order to calculate pyritic sulfur content using the proposed procedure, it is important to obtain the regression constants in Eq. 2 not only for representative coal samples but also for the same cleaning process.

7.6.3 Summary and Conclusions

A procedure was developed for estimating the pyritic sulfur content of coal separation products from their total sulfur and ash content. By assuming that the organic sulfur content in combustible materials in a coal is constant, a linear relationship is found among the pyritic sulfur, total sulfur and ash content of a coal sample. This linear relationship has been confirmed by the experimental results of both flotation samples and washability samples. The values of the proportionality constants in the linear equation for a specific coal are determined from the experimental pyritic sulfur, total sulfur and ash contents of several coal separation products of the same coal using linear regression analysis. After the parameters in the equation have been established for a particular coal, the pyritic sulfur content of other separation products of the same coal can be calculated based on their total sulfur and ash content using the equation. The results have shown that the calculated pyritic sulfur contents of various coal samples agree very well with their experimental values. The main advantage of this procedure is that it is reliable and rapid because total sulfur and ash content of coal can be determined easily using microprocessor-controlled machines.

7.7 References

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8.0 EFFECT OF GRINDING ENVIRONMENT

The overall objective of this task was to determine the effect of fine grinding under various environments on the flotation behavior of coal and its associated mineral matter, particularly pyrite. The grinding environment studied included air and inert gases, wet and dry grinding methods, grinding with a ceramic mill, and grinding with collector and frother. Detailed discussions of the results are given in the following sections.

8.1 Grinding and Flotation under Air and Inert Gases

In order to study the effect of grinding and flotation under air and inert gas, coal samples were dry and wet ground to 200 mesh in the rod mill under either air or inert (argon) environments maintaining all other grinding conditions the same as those specified for the standard grinding test. For wet grinding in an inert environment, triply-distilled water, which had first been deoxygenated by purging with argon, was used. The ground samples were then floated with either air or argon bubbles. The standard dosages of collector (dodecane) and frother (MIBC) were added directly to the flotation cell. Flotation yields were obtained following the standard flotation test conditions.

The combustible material recovery for the three base coals, dry- and wet-ground under air or inert atmospheres and floated with air or argon bubbles are tabulated in Table 8.1. The results indicate that for all the combinations of grinding environment and bubble composition, wet grinding gives a higher combustible material recovery than dry grinding for both Illinois No. 6 and Upper Freeport PA coals. However, in the case of Pittsburgh No. 8 coal, the CMR of the wet ground material is slightly less than the dry-ground material. It should be noted that the collector dosage for wet-ground Pittsburgh No. 8 coal is lower than that for dry-ground material whereas for the other two coals it is the same.

The absence of any significant effect of oxygen present during grinding may be due to a number of reasons. Most likely, it is because the grinding mill is a closed container with a limited volume, and as new coal surfaces are being generated the entrapped oxygen is consumed

Table 8.1 - The effect of grinding atmosphere and flotation gas composition on the combustible material recovery of 200-mesh feed.

Coal	Grinding Method	COMBUSTIBLE MATERIAL RECOVERY, %			
		AG/AF	AG/IF	IG/AF	IG/IF
Illinois No. 6	Dry	59.6	63.6	60.3	60.3
	Wet	84.0	88.3	88.2	87.9
Pittsburgh No. 8	Dry	92.0	91.0	82.7	85.9
	Wet	80.5	80.9	81.8	80.6
Upper Freeport PA	Dry	69.4	67.3	71.9	71.5
	Wet	80.4	81.0	79.4	77.9

AG = air grind AF = air float IG = inert grind IF = inert float

so rapidly that the majority of the new surface generation takes place in a nitrogen atmosphere. This explanation assumes that any oxygen that contacts the coal will indeed react.

Bubble composition (air or argon) does not seem to affect the flotation yields of any of the three base coals for either dry or wet-ground coal. In the case of Illinois No. 6 coal, wet grinding under argon marginally improves the flotation yield. However, the flotation yields of Pittsburgh No. 8 and Upper Freeport PA coals are not affected by the wet-grinding environments. Dry-grinding environment shows no discernible trends on flotation yields for the three coals.

In view of the results, the grinding and flotation of coal in an inert atmosphere does not seem to improve the flotation yield and, therefore, an uncontrolled (air-saturated) grind and float is appropriate for industrial purposes.

8.2 Wet and Dry Grinding Methods

The mode of grinding, dry or wet, can have different effects on the surface properties of coal and the gangue minerals and it is important to understand its role in determining the flotation performance. The majority of the recent investigations on fine coal flotation has been conducted with coal ground either dry or wet (1-4). However, the effect of different grinding methods on the separation of pyrite and other ash-forming minerals from coal is not clear from these studies. Miller and Guzzo (5) have compared the effect of dry- and wet-grinding methods for deep cleaning ultrafine coal by flotation. Their study was preliminary in nature, however, and

Figure 8.1 - Effect of the grinding mode on the separation of non-pyritic minerals from Pittsburgh No. 8 coal and a comparison of washability and flotation selectivity curves for 28 mesh grind.

the effect of the grinding method on pyrite rejection, the primary objective of our investigation, was not reported. In the current study, the effect of wet and dry grinding methods on the separation of pyrite and non-pyritic ash from coal has been studied with both 28- and 200-mesh Pittsburgh No. 8 coal.

The results are presented in terms of flotation selectivity curves in which pyritic sulfur rejection is represented by the y-axis and combustible material recovery by the x-axis. A shift of the curve towards the upper-right-hand corner signifies improved selectivity in these plots. Iso-efficiency lines are shown in Figure 8.1 to demonstrate their relationship to the selectivity curves. In all the tests reported in this section dodecane dosage was maintained at the standard dosage level of 1.9 lb/T and the MIBC dosage was varied to obtain different combustible material recoveries.

The effect of wet and dry grinding on the separation efficiency of non-pyritic mineral matter for 28-mesh grind is shown in Figure 8.1. It can be seen from this figure that the slope of the iso-efficiency index lines is higher (more negative) than the selectivity curves in the low

Table 8.2 - Particle size distribution of various grinding and washability samples of Pittsburgh No. 8 coal.

Grinding Mode	Grind Size Mesh	SIZE, MICRONS				
		d ₉₀	d ₇₅	d ₅₀	d ₂₅	d ₁₀
Dry	28	520	340	180	67	20.0
Wet	28	520	340	195	82	34.0
Washability	28	--	--	--	76	--
Dry	200	62	32	21	9.5	4.1
Wet	200	62	42	23	12.0	5.6
Washability	200	60	50	27	10.5	5.5

combustible material recovery region, while in the region of high combustible material recoveries the slope of the selectivity curves is higher. This suggests that at low recoveries the impurities are carried over primarily due to entrainment (mechanical carry-over), while at high recoveries, in addition to entrainment, carry-over of locked particles also occurs.

Figure 8.1 also shows that the selectivity obtained for the wet-ground feed is not significantly different from that obtained for the dry-ground feed. Further, this figure shows that for a given coal recovery, the amount of non-pyritic mineral matter rejected is about 15 percent lower than that predicted by the washability data. Since non-pyritic mineral matter is not naturally hydrophobic, it is reasonable to assume that it is unlikely to be rendered hydrophobic by the addition of a non-specific collector such as n-dodecane. Therefore, carry over of the mineral matter into the froth product can be attributed to the entrainment of solids due to water flow since aggregation of fine mineral matter with coal (heterocoagulation), that can also contribute to the loss in selectivity, is not likely to play a major role with these feeds as they contain less than 10 percent of particles smaller than 20 microns (Table 8.2).

The selectivity curves given in Figure 8.2 show that for 200 mesh Pittsburgh No. 8 coal, a better selectivity is achieved for wet-ground feed than for dry-ground feed. One possible reason for this is the difference in the particle size distribution, which can result in varying degrees of i) entrainment of fines due to water flow, ii) heterocoagulation between coal and mineral matter, and iii) liberation of mineral matter from coal. With decreasing particle size, the first two factors will affect the separation detrimentally, while the third will have a favorable effect.

Figure 8.2 - Effect of the grinding mode on the separation of non-pyritic minerals from Pittsburgh No. 8 coal and comparison of washability and flotation selectivity curves for 200-mesh grind.

Examination of particle size distributions of the 200-mesh grinds show that the average particle size of the dry-ground feed is 21 microns compared to 23 microns obtained for the wet ground feed (Table 8.2). To determine whether this difference significantly affects the separation efficiency, selectivity curves obtained for dry-ground 28-mesh and 200-mesh Pittsburgh No. 8 coal are compared in Figure 8.3. It can be seen from this figure that there is no significant difference in the separation achieved with the 28-mesh grind (average particle size = 180 microns) than that with the 200-mesh grind (average particle size = 21 microns). This suggests that, in the size range studied, entrainment is independent of particle size. Also, since the particle size distributions of wet and dry-ground feeds are similar (Table 8.2), heterocoagulation effects can also be considered as not significant. To examine how the degree of liberation can contribute to the observed difference between the separation efficiencies achieved with dry and wet-ground 200 mesh grinds, the washability curves of 28-mesh grind and 200-mesh grind were compared and are shown in Figure 8.3. It can be seen from this figure that the degree of liberation in the 200-mesh grind

Figure 8.3 - Comparison of flotation selectivity and washability curves of the 28 mesh and 200 mesh grinds of Pittsburgh No. 8 coal.

is only marginally higher than in the 28-mesh grind. This suggests that differences in the liberation characteristics also do not contribute significantly to the observed difference in the separation efficiency.

Other possible reasons for the relatively poor separation achieved with the dry-ground feed are the oxidation of coal surface and dry-state aggregation of the mineral matter with coal during grinding (6). To determine whether oxidation of coal surfaces can result in a loss of selectivity, tests were conducted with chemically oxidized feed. This sample was obtained by conditioning the ground coal slurry with 78 ml of 30 wt.% of hydrogen peroxide (oxidizing agent) for 30 minutes in the same grinding mill and at the same grinding speed after removing the grinding rods. Flotation results obtained with the chemically oxidized feed are compared with the unoxidized feed in Table 8.3. The results show that, although twice as much frother is required for oxidized coal to achieve a yield equivalent to that of the unoxidized coal, the ash content of the froth products are the same, indicating that oxidation of the coal surface diminishes only the floatability of coal and not the selectivity.

Table 8.3 - Effect of hydrogen peroxide addition on flotation performance of 200-mesh wet-ground Pittsburgh No. 8 coal.

<u>Coll.</u> <u>lb/T</u>	<u>Frot.</u> <u>lb/T</u>	<u>H₂O₂</u> <u>wt%</u>	<u>Flot. Yield</u> <u>wt%</u>	<u>Ash in</u> <u>float, wt%</u>
1.92	0.30	0.0	72.5	5.1
1.92	0.60	3.0	72.0	5.1

The above discussions suggest that dry-state aggregation of coal with the mineral matter is the most probable reason for the relatively poor separation achieved with the dry-ground feed. Since the degree of aggregation will be more severe for finer material, dry-state aggregation does not play a significant role in the flotation of 28-mesh coal.

Selectivity curves obtained for 28-mesh and 200-mesh grinds are presented in Figures 8.4 and 8.5 along with the washability curves. It can be seen from the plots given in Figure 8.4 that the mode of grinding does not play a significant role in determining the selectivity achieved during flotation in the case of the 28-mesh grind whereas with the 200-mesh grind, higher selectivity was obtained with the wet-ground feed in comparison to that achieved with the dry-ground feed. Another important observation is that the shift of the selectivity curves for pyrite for the washability tests is greater than the shifts in the curves for non-pyritic ash (compare Figures 8.1 and 8.2, with Figures 8.4 and 8.5, respectively). For a given coal recovery, the difference between the washability and the selectivity curves in terms of the percentage of non-pyritic ash rejection is 10-15 percent compared to 15-25 percent for pyrite. The additional loss of selectivity in the case of pyrite may be due to the higher natural hydrophobicity and higher density of pyrite than non-pyritic ash-forming minerals.

In summary, at coarser grind sizes (28 mesh) flotation selectivity was not significantly affected by grinding mode whereas for fine coal (200 mesh), the selectivity achieved with the wet-ground feed was higher than that obtained with the dry grind. The poor separation achieved with dry grinding was attributed to dry-state aggregation of the mineral matter with coal.

Figure 8.4 - Effect of grinding mode on the separation of pyrite from Pittsburgh No. 8 coal and a comparison of washability and flotation selectivity curves for the 28-mesh grind.

Figure 8.5 - Effect of grinding mode on the separation of pyrite from Pittsburgh No. 8 coal and a comparison of the washability and flotation selectivity curves for the 200-mesh grind.

8.3 Grinding with Ceramic Mill

In order to study the effect of different grinding media on the flotation behavior of coal, Illinois No. 6 coal was ground using a ceramic and a steel ball mill and the products were floated following the standard flotation procedure. In order to obtain a particle size distribution similar to that produced with the stainless steel rod mill, a 95% minus 200-mesh flotation feed was prepared by step-grinding in the ball mills. Preliminary grinding tests were performed to determine the number of steps and the grinding time for each step. For both the ceramic and steel ball mills, 500 grams of Illinois No. 6 coal were loaded to the mill along with 700 ml of distilled water. The ceramic mill was not able to comminute 1/4" x 0 material and it was necessary to pre-crush the standard feed to 85% passing 8-mesh before charging the ceramic mill. This additional size reduction step was not required for the feed to the steel-ball mill. The procedure for loading the mill was similar to that for the standard wet-grinding tests, that is, 500-grams of coal and 300 ml of water were first added to the mill, then the grinding media was loaded and finally the remaining 400 ml of distilled water was poured into the grinding mill.

In order to produce a narrow size distribution, similar to that obtained from a rod mill, a special experimental procedure was developed that included sieving the intermediate ground product at 150 mesh (105 microns) after each grinding step and then recharging the mill while trying to maintain the same liquid/solids ratio. Table 8.4 summarizes the experimental procedure used for step grinding the coal.

In addition, the grinding mill was purged with argon and the sample blanketed with argon during the classification and decantation of the minus 150-mesh size fraction to help prevent oxidation. Preliminary tests showed that to grind Illinois No. 6 coal in the ceramic mill a total of four steps were necessary, with 20 minutes of grinding for the first two steps and 30 minutes for the remaining steps. In the case of the steel ball mill, four 10-minute steps were adequate.

The particle size distribution of the ground products (flotation feed) was determined by wet sieve analysis at 200- to 400-mesh sieves followed by particle size analysis using an L&N Microtrac for the minus 400-mesh fraction. In order to avoid flocculation of fine particles, Calgon (sodium hexametaphosphate) was added to the suspension to disperse particles when using the Microtrac particle size analyzer. The particle size distribution, perhaps the most

Table 8.4 - Experimental procedure for step grinding tests using ceramic and steel ball mills.

Step No.	Name	DESCRIPTION	
		Ceramic mill	Steel ball mill
1	Feed	85 % minus 8 mesh	100 % minus 1/4 inch (standard)
2	Load	500 g Ill. No. 6 300 ml 1-D water Pebbles 400 ml, 1-D water Purge with argon	500 g Ill. No. 6 300 ml 1-D water Balls 400 ml, 1-D water Purge with argon
3	Grind	20 or 30 minutes	10 minutes
4	Unload	Over coarse screen to remove pebbles. Pulp to a bucket	Over coarse screen to remove balls. Pulp to a bucket
5	Sieving	Sieve at 150 M and Decant to recover water Measure water recovery	Sieve at 150 M and Decant to recover water Measure water recovery
6	Cleaning and Recharge	Clean pebbles and return to the mill Recharge +150 M to the mill Purge with argon Return decantate and additional water to mill	Clean balls and return to the mill. Recharge +150 M to the mill Purge with argon Return decantate and additional water to mill
7	2nd grind	20 or 30 minutes	10 minutes.
8	Repeat steps 5 through 7.	Until 95 % minus 200 M	Until 95 % minus 200 M.

important flotation variable, of the products is plotted in Figure 8.6. As can be seen from the figure, the slope of the curves changes between the different grinding mills, showing a narrower size distribution in the case of the standard wet grind (rod mill) than that obtained with the ceramic and steel-ball mills. The finest size distribution was produced by the standard-dry-grind procedure. Even though all the grinding products were approximately 93% minus 200 mesh, Figure 8.6 shows that the amount of fine particles for various methods of grinding increased in the following order:

standard wet grind < steel-ball mill grind < ceramic-mill grind < standard-dry grind.

Figure 8.6 - Particle size distribution for Illinois No. 6 research sample grind under standard dry and wet conditions and in a ceramic and steel ball mill.

The flotation results for the ceramic, steel and rod mill products are shown in Table 8.5. As can be seen from these results and those presented in Figure 8.6, the flotation yield is inversely proportional to the amount of fines present. The amount of fines present may have more impact on the efficiency index than any specific interaction between the grinding mill and the coal (for instance, galvanic effects). The difference in the efficiency index for coal that had been ground wet in the rod mill and that ground wet in the ceramic mill is within the statistical variance of the flotation process. The flotation product of the steel-ball-mill-ground coal contained the least amount of pyritic sulfur but the reduction was not considered to be significant. From these results we concluded that grinding with a ceramic mill does not have a significant effect on the flotation performance of Illinois No. 6 coal.

8.4 Grinding with Collector and Frother

The rationale of adding flotation reagents to the grinding mill is to ensure the adsorption of reagent as new surface is being generated during the comminution of the coarse particles.

Figure 8.7 - Effect of adding collector to the grinding mill on the yield, ash and total sulfur content of the concentrate for minus 28-mesh wet-ground Illinois No. 6 coal.

Since coal is susceptible to oxidation, adding reagents to the grinding mill may also help to minimize oxidation of the newly-formed surfaces. Either collector (dodecane in this case) or frother (MIBC) was charged to the grinding mill with the coal feed, and the material was ground for the pre-determined time to achieve the desired particle size. The grinding was carried out under wet conditions so that the reagent would be well dispersed and thereby adsorb more uniformly on the coal surface.

8.4.1 Effect of Grinding with Collector and Frother on Flotation Yield and Separation Efficiency

In the first set of experiments performed with Illinois No. 6 coal, the collector dosage added to the mill was varied from 0.45 lb/T to 4.8 lb/T. The frother dosage corresponding to the standard condition (1.2 lb/T) was added to the flotation cell. No additional collector was added to the flotation cell. All flotation yields reported in this section were obtained at flotation times of 5 minutes. In Figure 8.7 the yield, ash and total sulfur content of the flotation product of Illinois No. 6 coal are plotted as a function of collector addition to the grinding mill for minus

Table 8.5 - Flotation results of different grinding products for Illinois No. 6 research samples.

Grind	Grind Mode	PRODUCT ANALYSIS				REJECTION		El _H
		Yield %	CMR %	Ash %	Pyr S %	Ash %	Pyr S %	
Rod mill	dry	60.4	64.8	9.3	1.36	63.7	68.0	32.8
Ceramic mill	wet	63.6	69.1	8.0	1.24	67.1	69.2	38.3
Steel ball mill	wet	67.6	73.8	7.6	1.07	66.8	71.8	45.6
Rod mill	wet	77.0	83.3	8.4	1.57	57.6	52.7	36.0

28-mesh flotation feed. The flotation yield increases as the collector dosage to the mill is increased. The yield under standard conditions (1.2lb/T frother, 4.8 lb/T collector) appears to be roughly equivalent to those obtained when collector is added during grinding.

There is also a similar increasing trend in the ash content of the concentrate, however, there is very little difference in the reduction of total sulfur in the float product. Figure 8.8 shows the effect of adding part of the collector to the mill and the balance quantity (to make up the dosage corresponding to that of the Standard Test) directly to the flotation cell. It can be seen from this figure that the flotation yield increases slightly with an increase in the fraction of collector added to the mill. Similar behavior was observed for flotation tests carried out using minus 200-mesh feed (Figures 8.9 and 8.10). There also appears to be no appreciable advantage of collector addition in the grinding mill with regard to the selectivity of separation. However, we did observe that flotation was much more rapid when the coal was ground with collector.

The effect of adding frother to the grinding mill on the flotation behavior of both 28-mesh and 200-mesh Illinois No. 6 coal are presented in Figure 8.11 along with results corresponding to adding the entire frother dosage to the flotation cell. It can be seen that the addition of the standard amount of frother (1.2 lb/T) to the grinding mill is detrimental to flotation, and unless more is added to the flotation cell, the yield is greatly reduced. The reduction in flotation yield probably results from the adsorption of frother by the coal during the grinding process, thereby removing it from the system so that it is not available to help stabilize the froth. Furthermore, no improvement in either ash or sulfur rejection was observed when the coal was ground with frother. The results of the tests performed to evaluate the effect of collector and frother addition during the grinding stage for Illinois No. 6 coal are summarized in Tables 8.6 and 8.7.

Figure 8.8 - Effect of adding part of the collector to the grinding mill and the balance to the flotation cell on the yield, ash and total sulfur content of the concentrate of minus 28-mesh wet-ground Illinois No. 6 coal.

Figure 8.9 - Effect of adding collector to the grinding mill on the yield, ash and total sulfur content of the concentrate of minus 200-mesh wet-ground Illinois No. 6 coal.

Figure 8.10 - Effect of adding part of the collector to the grinding mill and the balance to the flotation cell on the yield and ash content of the concentrate of minus 200-mesh wet-ground of Illinois No. 6 coal.

Figure 8.11 - Effect of adding frother (MIBC) to the grinding mill and in the flotation cell on the yield of the concentrate obtained using Illinois No. 6 coal.

Table 8.6 - The effect of collector and/or frother addition to mill during grinding on 28-mesh Illinois No. 6 coal.

Collector to Mill lb/T	Collector to Cell lb/T	Frother to Mill lb/T	Frother to Cell lb/T	PRODUCT ANALYSIS			EI _H
				Yield %	Ash %	Pyr S %	
4.8	--	--	1.2	90.1	11.4	2.19	17.4
2.4	--	--	1.2	85.0	11.4	1.98	23.4
2.4	2.4	--	1.2	86.8	11.8	2.06	20.8
1.9	--	--	1.2	82.0	11.2	1.90	25.3
1.9	2.9	--	1.2	87.9	11.0	2.10	20.5
0.95	--	--	1.2	72.5	10.3	1.98	20.9
0.95	3.9	--	1.2	82.4	11.4	2.25	14.0
--	4.8	1.2	--	84.8	10.4	2.10	20.4
--	4.8	--	1.2	87.8	10.9	1.83	29.8
--	4.8	0.4	--	39.5	7.1	1.20	24.9
--	4.8	0.4	0.8	85.7	10.8	1.80	30.2

Similar flotation tests were also carried out with Pittsburgh No. 8 coal. Evaluation of the effect of collector and frother addition to the mill and to the cell at different levels showed (Tables 8.8 and 8.9) that the highest value of the Efficiency Index (EI_H) obtained for the 200-mesh wet-ground material was 50.4 which was produced by adding 50% of the collector in the mill with the remainder being added to the cell along with all the frother. This corresponded to a combustible material yield of 82% and a pyrite rejection of 67%. Also, when the collector was added to the mill, the efficiency index was only marginally lower in cases where the full amount of frother was added to the mill (EI_H≈47) or to the cell (EI_H≈48). In the case of 28-mesh feed, for a given collector and frother dosage, the highest value of the Efficiency Index (EI_H≈36) was obtained by adding the full dosage of collector to the cell and frother to the mill. The foregoing results indicate that for 200-mesh coal, the optimum grinding conditions with respect to a combination of combustible material recovery and pyrite rejection for Pittsburgh No. 8 can be achieved by wet grinding with collector followed by frother addition to the flotation cell.

The flotation results of tests with collector and frother additions during the grinding of Upper Freeport PA coal are given in Tables 8.10 and 8.11. The results in these tables show that

Table 8.7 - The effect of collector and/or frother addition to the mill during wet grinding on the flotation of 200-mesh Illinois No. 6 coal.

Collector to Mill lb/T	Collector to Cell lb/T	Frother to Mill lb/T	Frother to Cell lb/T	PRODUCT ANALYSIS			El _H
				Yield %	Ash %	Pyr S %	
5.8	--	--	1.2	87.2	9.3	2.20	18.7
2.9	--	--	1.2	80.0	8.6	1.81	30.0
2.9	2.9	--	1.2	88.0	9.4	1.64	38.0
1.9	3.9	--	1.2	80.8	8.3	1.78	31.5
--	5.8	2.0	--	75.8	8.7	1.65	33.0
--	5.8	1.2	--	45.8	6.5	1.20	29.2
--	5.8	0.4	0.8	85.7	10.8	1.80	30.2

Table 8.8 - The effect of collector and/or frother addition to the mill during wet grinding on 28-mesh Pittsburgh No. 8 coal.

Collector to Mill lb/T	Collector to Cell lb/T	Frother to Mill lb/T	Frother to Cell lb/T	PRODUCT ANALYSIS			El _H
				Yield %	Ash %	Pyr S %	
2.0	--	--	0.40	90.0	8.2	2.17	25.3
1.0	1.0	--	0.40	94.8	7.9	2.16	27.3
--	2.0	0.40	--	84.8	7.2	2.02	29.3
--	2.0	0.40	--	90.6	7.4	1.87	35.9

Table 8.9 - The effect of collector and/or frother addition to the mill during wet grinding on 200-mesh Pittsburgh No. 8 coal.

Collector to Mill lb/T	Collector to Cell lb/T	Frother to Mill lb/T	Frother to Cell lb/T	PRODUCT ANALYSIS			El _H
				Yield %	Ash %	Pyr S %	
1.9	--	--	0.30	68.4	4.7	1.08	48.4
0.95	0.95	--	0.30	75.3	4.9	1.18	50.4
--	1.9	0.30	--	50.6	4.4	0.88	39.6
1.9	--	0.30	--	46.6	3.7	0.75	47.3

Table 8.10 - The effect of collector and/or frother addition to the mill during wet grinding on 28-mesh Upper Freeport PA coal.

Collector to Mill lb/T	Collector to Cell lb/T	Frother to Mill lb/T	Frother to Cell lb/T	PRODUCT ANALYSIS			EI _H
				Yield %	Ash %	Pyr S %	
--	0.24	--	0.26	75.5	6.42	0.63	47.8
0.12	0.12	--	0.26	68.8	5.94	0.55	47.0
0.24	--	--	0.26	82.4	6.77	0.67	49.9
0.24	--	0.26	--	73.2	6.46	0.55	50.0
0.24	--	0.26	--	75.0	6.60	0.54	51.5
--	0.24	0.26	--	58.0	5.43	0.53	40.3

the addition of dodecane to the grinding mill has very little effect on the flotation recovery and the overall efficiency index while the addition of frother to the mill decreases both the flotation yield and the efficiency index. The influence of the reagent addition point is not as profound for Upper Freeport PA coal as for the other two coals, probably owing to it being more hydrophobic and having a froth that is difficult to break.

8.4.2 Effect of Grinding with Collector on Flotation Kinetics

The objective of this part of the investigation was to compare the effect of adding the standard dosage of collector (dodecane) to the rod mill before wet grinding on the flotation kinetics of the three base coals. This study was undertaken because the results of grinding with

Table 8.11 - The effect of collector and/or frother addition to the mill during wet grinding on 200-mesh Upper Freeport PA coal.

Collector to Mill lb/T	Collector to Cell lb/T	Frother to Mill lb/T	Frother to Cell lb/T	PRODUCT ANALYSIS			EI _H
				Yield %	Ash %	Pyr S %	
--	0.48	--	0.21	68.2	5.28	0.51	48.2
0.24	0.24	--	0.21	66.0	4.82	0.51	46.6
0.24	0.24	--	0.21	71.4	5.13	0.55	48.8
0.48	--	--	0.21	69.5	5.07	0.58	46.1
0.48	--	0.21	--	44.0	4.11	0.33	36.2
0.48	--	0.21	--	42.8	4.15	0.32	35.4
--	0.48	0.21	--	51.9	4.68	0.52	36.3

collector added to mill showed that even though the efficiency was not necessarily improved, the kinetics of flotation were changed.

In this study, the coal feed for flotation was wet-ground using the standard grinding procedure to obtain a product 95% finer than 200 mesh. Grinding was carried out under wet conditions to ensure that the collector would be well dispersed and thereby adsorb more uniformly on the coal surface. All operating conditions in these flotation tests were the same as the standard flotation test conditions. In the case of the coal samples ground with collector, the conditioning period with dodecane in the standard flotation test procedure was also carried out except no dodecane was added to the flotation cell. In both cases, flotation concentrates were collected at 0.5, 1.0, 2.0, 4.0 and 5.0 minutes, dried, and weighed.

Figures 8.12 through 8.14 present the flotation yield of Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA coals as a function of flotation time for conditions corresponding to addition of the standard dosage of collector to the flotation cell after wet grinding and to the mill before grinding. The fitting curves for these flotation results for a first-order kinetic model using a sine distribution of flotabilities* and the corresponding fitting parameters are also shown in these figures. The results given in these figures indicate that the flotation recovery and rate constant of Illinois No. 6 is greater when the collector was added to the mill prior to grinding, as compared with that when the collector was added to the flotation cell. On the other hand, the flotation yield of Pittsburgh No. 8 coal decreases while its flotation rate constant was the same as that when ground with collector. For Upper Freeport PA coal, both the flotation yield and flotation rate constant decreased when the standard dosage of collector was added to the mill. The different flotation behavior of the three base coals when they are ground with collector may be due either to the difference in hydrophobicity of the coals and/or to the difference in collector dosage used for the three coals.

In order to delineate the reason for the different flotation behavior of the three base coals, additional studies were carried out (about 6 months later) at a relatively low collector dosage and at equivalent collector dosage for all three coals. In this series of tests, the frother dosage was

*This model is discussed in detail in Chapter 11 - Flotation Kinetics.

Figure 8.12 - Flotation yield of 200-mesh Illinois No. 6 coal obtained by adding 5.8 lb/T of dodecane to the grinding mill or flotation cell.

Figure 8.13 - Flotation yield of 200-mesh Pittsburgh No. 8 coal obtained by adding 1.9 lb/T of dodecane to the grinding mill or flotation cell.

Figure 8.14 - Flotation yield of 200-mesh Upper Freeport PA coal obtained by adding 0.5 lb/T of dodecane to the grinding mill or flotation cell.

held at the standard level for each coal and flotation concentrates were collected at 0.5, 1, 2, 4 and 8 minutes. Figures 8.15 through 8.20 plot the flotation yield of the three base coals as a function of flotation time for various dosages of collector to the flotation cell after wet grinding and to the mill before grinding along with their fitting curves and fitting parameters. It can be seen from Figures 8.12 to 8.20 that the simulation curves fit the experimental curves quite well, indicating that the flotation kinetics of coal follows the first-order kinetic model with a sine distribution of flotabilities. The ultimate flotation recovery, R_{∞} , and flotation rate constant, K , of the three base coals obtained with various dosages of collector are summarized in Table 8.12.

The results presented in Figures 8.12 to 8.20 and Table 8.12 show that for all three coals, both the flotation yield and flotation rate constant generally increase when the collector dosage is increased regardless of the point of addition. This is expected because the coal particles will be more hydrophobic with increased collector dosage, with an attendant increase in the flotation yield and flotation rate constants. The exception to this general observation is that when adding collector to the flotation cell, the flotation rate constant of Illinois No. 6 coal decreases slightly

Table 8.12 - Summary of the effect of collector addition on the ultimate recovery and flotation rate constant for the three base coals obtained by fitting the first-order kinetic model with sine distribution of flotabilities.

Coal	MIBC lb/T	Dodecane lb/T	Collector to Cell		Collector to Mill	
			R_{∞} %	K , min ⁻¹	R_{∞} %	K , min ⁻¹
Illinois No. 6	1.2	0.7	64.4	0.75	55.5	0.62
		2.9	76.7	1.33	81.7	2.77
		5.8*	86.8	1.27	87.5	2.80
Pittsburgh No. 8	0.3	0.7	78.2	0.51	42.2	0.68
		1.9*	86.5	1.05	62.2	1.10
		2.9	84.1	1.27	68.0	1.75
Upper Freeport PA	0.2	0.24	79.6	0.96	73.4	0.73
		0.5*	84.8	1.43	82.7	1.08
		2.9	85.6	2.81	86.9	1.61

when the collector dosage was increased from 2.9 to 5.9 lb/T and the ultimate flotation recovery of Pittsburgh No. 8 coal decreased slightly when the collector dosage was increased from 1.9 to 2.9 lb/T. This may be due to variations in the coal samples and operation errors because these experiments were conducted by different researchers at different times (about six months apart) and may also be due to the well-known R_{∞} versus K trade-off discussed in the literature (7).

However, except for Illinois No. 6 coal at 0.7 lb/T of dodecane, these results show that the effect of collector addition to the flotation cell after grinding on the flotation rate constant compared to that when collector was added to the mill is the same as that at the standard level of collector for each coal. For the less hydrophobic Illinois No. 6 coal, both flotation recovery and flotation rate constant, obtained with collector dosages of 2.9 and 5.8 lb/T were higher when the collector was added to the mill than that when added to the cell. At very low collector dosage (0.7 lb/T) the flotation recovery and flotation rate constant of Illinois No. 6 coal is lower when adding collector to the mill than to the cell. On the other hand, for the most hydrophobic coal (Upper Freeport PA) both flotation recovery and flotation rate constant are lower when grinding with collector at all dosages studied except at the highest collector dosage of 2.9 lb/T. For Pittsburgh No. 8 coal the flotation recovery is lower and the flotation rate constant is slightly higher when grinding with collector for the collector dosages studied. Furthermore, the difference between the flotation recovery obtained when collector is added to the mill compared

Figure 8.15 - Flotation yield of 200-mesh Illinois No. 6 coal obtained by adding 0.7 lb/T of dodecane to grinding mill or flotation cell.

Figure 8.16 - Flotation yield of 200-mesh Illinois No. 6 coal obtained by adding 2.9 lb/T of dodecane to grinding mill or flotation cell.

Figure 8.17 - Flotation yield of 200-mesh Pittsburgh No. 8 coal obtained by adding 0.7 lb/T of dodecane to grinding mill or flotation cell.

Figure 8.18 - Flotation yield of 200-mesh Pittsburgh No. 8 coal obtained by adding 2.9 lb/T of dodecane to grinding mill or flotation cell.

Figure 8.19 - Flotation yield of 200-mesh Upper Freeport PA coal obtained by adding 0.24 lb/T of dodecane to grinding mill or flotation cell.

Figure 8.20 - Flotation yield of 200-mesh Upper Freeport PA coal obtained by adding 2.9 lb/T of dodecane to grinding mill or flotation cell.

to that when added to the cell decreases with increasing collector dosage for all three coals. Therefore, these results indicate that the effect of grinding with collector on flotation kinetics and recovery depends on both the hydrophobicity of coal and the reagent dosage used in the system. In general, in order to achieve better flotation recovery and kinetics, the collector should be added to the flotation cell after grinding if the coal is more hydrophobic and/or the collector dosage is relatively low for that coal.

The variation in the flotation response to the point of addition of the collector can be explained in terms of the spreading of dodecane on coal surface and penetration of dodecane into the pores of coal particles. In general, better surface spreading of insoluble oils can improve flotation recovery and pore penetration of insoluble oils can decrease flotation recovery as shown by Moxon et al.(8). Grinding coal with dodecane improves spreading of dodecane on coal surfaces but also increases penetration of dodecane into the pores of the coal. The overall effect of grinding coal with dodecane depends on which of the two phenomena is more significant for a particular coal. For a hydrophilic coal, such as Illinois No. 6, the spreading of dodecane on the wet coal surface is more difficult and penetration of dodecane to the pores is slow, thus the improvement in flotation due to spreading of dodecane on the coal surface is more significant than penetration into the pores if the collector dosage is not too low. Therefore, flotation recovery and kinetics of the less hydrophobic Illinois No. 6 coal is improved when grinding with dodecane. However, if the dodecane dosage is too low, even small amounts of dodecane lost due to pore penetration during grinding may be significant resulting in flotation recovery and kinetics when grinding with the collector than when adding the collector to the flotation cell. On the other hand, for hydrophobic coals such as Pittsburgh No. 8 and Upper Freeport PA coals, because the spreading of dodecane on the wet coal surface is easy and the penetration of dodecane into the pores is fast, a decrease in surface coverage of dodecane due to penetration of dodecane to the pores is more significant than the improvement of spreading of dodecane on the coal surface during grinding with collector. Therefore, the flotation performance of Pittsburgh No. 8 and Upper Freeport PA coals ground with dodecane is not as good as that when the dodecane is added to the flotation cell. The relatively small effect on flotation with Upper Freeport PA coal when the dodecane is added to the flotation cell and the grinding mill

compared to Pittsburgh No. 8 coal may be due to the higher hydrophobicity and shorter grinding time (11 minutes for this coal versus 16 minutes for Pittsburgh No. 8).

In order to test this hypothesis further, samples of Illinois No. 6 coal ground with and without collector and samples of Pittsburgh No. 8 coal ground without collector were aged under water for 24 hours at room temperature prior to flotation. For coal samples ground without collector, dodecane was added to the flotation cell after ageing. Figures 8.21 and 8.22 shows the effect of aging on the flotation behavior of the coal ground with and without dodecane. The results presented in these figures clearly show that aging of coal samples ground without dodecane decreases slightly the flotation yield of Illinois No. 6 coal, and both flotation yield and rate constants of Pittsburgh No. 8 coal. This may be due to some oxidation of the surfaces of coal particles during the aging process. Because Pittsburgh No. 8 is more hydrophobic than Illinois No. 6 coal, the decrease in flotation recovery is smaller as compared to Illinois No. 6. However, when aging Illinois No. 6 sample ground with dodecane, both flotation yield and flotation rate constant decreased significantly. Some of this decrease may be due to the oxidation of coal surface during aging as shown in the case when the coal was aged without dodecane. The majority of the decrease is due to penetration of dodecane into the pores of coal, thus reducing the collector coverage on the coal surface. Due to the long aging time (24 hours), dodecane slowly penetrates into the pores of Illinois No. 6 coal even though it is quite hydrophilic. These results further support the explanation that the decrease in flotation recovery of hydrophobic coals when ground with collector is due to the absorption of collector to the pores, which results in a decrease in collector coverage on the external surface of coal particles.

While the addition of collector to the grinding stage could improve the flotation kinetics of Illinois No. 6 coal, the selectivity is virtually unaltered, as can be seen from Figure 8.23, where the ash and pyrite rejection was plotted as a function of the combustible material recovery. Therefore, hydrophilic coal ground with dodecane can reduce the consumption of collector at similar separation efficiency and flotation yield.

Figure 8.21 - The effect of aging after grinding on the flotation yield of 200-mesh Illinois No. 6 coal obtained by adding 2.9 lb/T of dodecane to grinding mill or flotation cell.

Figure 8.22 - The effect of aging after grinding on the flotation yield of 200-mesh Pittsburgh No. 8 coal obtained by adding 2.9 lb/T of dodecane to flotation cell.

Figure 8.23 - Ash and pyritic sulfur rejection as a function of combustible material recovery of 200-mesh Illinois No. 6 coal obtained by adding 2.9 lb/T of dodecane to grinding mill or flotation cell.

8.5 References

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9.0 SURFACE MODIFICATION

In flotation, the separation of one material from another occurs by selectively altering and controlling the hydrophobicity of the various interfaces present. In advanced coal flotation, the control of the relative hydrophobicities of the carbonaceous material to that of pyrite and other ash minerals is central to near total pyrite removal at high Btu recovery. One way to enhance through separation flotation is to selectively increase the hydrophobicity of the coal while leaving the mineral surface unchanged. If successful this would lead to increased Btu recoveries with better ash and pyrite rejection. The results of work with various types of surface modifiers and their effect on advanced coal flotation are reported in this chapter.

Another sometimes-overlooked aspect of flotation is the role of frothers. The addition of a selective collector is to alter the solid/liquid interface, whereas the purpose of adding a frother is to control the air/liquid interface and, as the name implies, the stability of the froth in order to carry the hydrophobic particles attached to the air bubbles from flotation cell. The physical process occurring within the froth layer is paramount to the success of the flotation process. It is within this layer that unwanted (hydrophilic) material must settle out leaving only the desired material to be recovered. If the froth is unstable, little or no material will be recovered. If it is too tenacious a large amount of unwanted material may be carried over. In coal flotation, the process is complicated in that the frother molecules also adsorb on the coal matrix. In view of the importance of frothers on flotation, a detailed study was completed with a series of alcohols.

Another important aspect of flotation is the state of dispersion of the flotation pulp. When oily collectors are added to a system containing minus 200-mesh particles, agglomeration of the particles may take place. If these agglomerates contain only coal particles then no detrimental effect on flotation will occur and the kinetics of flotation may be enhanced. However, as is more likely the case, ash and pyrite will be trapped within these agglomerates reducing the efficiency of the process. Therefore, the effect of dispersants on flotation was also investigated and the results reported here.

9.1 Polymerizable Monomers

Task 6 in the RFP stipulated that an investigation be carried out to determine the possibility of utilizing free radicals generated by the rupture of carbon-carbon bonds during the grinding step to form graft polymers at the surface, thereby selectively enhancing the hydrophobicity of the coal particles. For this purpose, organic monomers, which can readily polymerize to form large hydrocarbon macromolecules, were added to the grinding mill to determine if conditions in the mill might be such that a polymerization reaction could be induced to take place between the monomer and the newly generated coal surface. If they could be produced, such graft polymers might then result in better flotation separations.

The rationale for the coal free-radical initiator was based on the premise that during the grinding process a carbon-carbon double bond might be broken, forming free radicals:

These free radicals might act as an initiator for the polymerization at the surface in the following manner:

If such a surface polymerization process was successful, it could build a hydrophobic polystyrene chain on the surface of the coal particle until a chain termination step took place.

These experiments were conducted while grinding the coal under dry conditions with the monomer present so as not to have any inhibiting effect from the water dispersing the monomer. An initiator was not added to the system because we did not want to cause the monomer to polymerize by itself. In our work plan, we had originally identified four possible monomers that could form graft polymers with the coal surface: styrene, vinyl benzaldehyde, isobutylene and methacrylonitrile. Of these four monomers, only styrene was practical for use in coal flotation. Vinyl benzaldehyde was considered to be commercially impractical due to its cost and therefore vinyl acetate was chosen in its place. Isobutylene, gaseous at room temperature, would pose special handling problems, so diisobutylene was used instead. Finally, the high toxicity of methacrylonitrile led us to choose its methyl ester, methyl methacrylate, which can undergo polymerization either through a free-radical mechanism or condensation via a 1,4 Michael addition.

It was recognized that any beneficial effect on flotation upon addition of the monomers could simply be a result of the added hydrocarbon and not of graft polymer formation. In order to help delineate which process was responsible for changes in the flotation behavior of the coal with polymerizable reagents, flotation tests were also conducted using the corresponding saturated equivalent (no reactive double bond) of the four monomers. The reagents used were the following:

<u>Monomer</u>	<u>Saturated Equivalent</u>
ϕC_2H_3 Styrene	ϕC_2H_5 Ethyl benzene
$CH_3COO-CH=CH_2$ vinyl acetate	$CH_3COOCH_2CH_3$ ethyl acetate
$(CH_3)_3C-CH_2-CCH_3=CH_2$ Diisobutylene	$(CH_3)_3C-CH_2-CH(CH_3)_2$ 2,2,4-Trimethylpentane
$CH_2=C-CH_3-COOCH_3$ Methyl methacrylate	

Table 9.1 - Summary of the effect of various polymerizable monomers and their saturated equivalents (4.8 lb/T except where noted) on the flotation efficiency index of the three base coals. Standard frother and collector dosages were used in all tests.

<u>Monomer</u>	<u>FLOTATION EFFICIENCY, EI_H</u>		
	<u>Illinois No. 6</u>	<u>Pittsburgh No. 8</u>	<u>Upper Freeport PA</u>
Styrene*	41.6 (40.6)	47.3 (45.0)	38.5 (42.5)
Vinyl Acetate	38.6 (42.8)	47.9	40.1
Methyl Methacrylate	39.7	46.6	46.4
Diisobutylene	45.8 (40.7)	37.3 (30.8)	42.5
Standard Test (no monomer)	38.4	49.2	41.4

* 6.4 lb/T styrene (ethyl benzene) used for flotation of Illinois No. 6 coal.
Values in (brackets) are the results for the saturated equivalents.

These reagents were added to the mill during dry grinding in an inert atmosphere and all tests conducted using 200-mesh grinds of the three base coals. Dry grinding was used not only to enable the monomer to diffuse more readily to the coal surface but primarily to prevent competition of the monomer molecules with water at the newly formed reactive sites; the inert atmosphere was used to prevent negative effects from oxygen (that is, reaction with free radicals). The experiments were designed in such a way that both the collecting and the surface-modifying properties of the reagents could be brought out. The collecting property of the monomers was evaluated at the standard MIBC dosage (with no dodecane added), while its surface modifying characteristics were evaluated using the standard collector and frother additions. The collecting properties of all of the reagents tested were minimal, with no reagent being able to produce a yield equivalent to that obtained with dodecane.

The results of the flotation tests with both the monomers and the saturated analogues for the three base coals are shown in Tables 9.1 to 9.4, with Table 9.1 presenting a summary of the EI_H values for the three base coals. The flotation yields increased only for the Illinois No. 6 and Pittsburgh No. 8 coals by the addition of a monomer or its saturated analog; Upper Freeport PA coal was not affected. The lack of change in the flotation of Upper Freeport PA coal probably stems from its inherent hydrophobicity and the addition of more hydrocarbon to the system does not cause the recovery to increase. The results for Illinois No. 6 and Pittsburgh No. 8 coal

Table 9.2 - Effect of polymerizable monomer addition on the flotation of 200-mesh dry-ground Illinois No. 6 coal (4.8 lb/T of modifier used except where noted).

Modifier	Yield %	FLOTATION PRODUCT ANAL				REJECTION		EI _H —
		CMR %	Ash %	Tot S %	Pyr S* %	Ash %	Pyr S %	
Standard test (no modifier)	60.4	66.0	9.31	3.57	1.46	68.6	72.4	38.4
Vinyl Acetate	62.9	69.0	8.92	3.66	1.54	68.7	69.6	38.6
Ethyl Acetate	67.6	74.3	8.82	3.60	1.47	66.7	68.5	42.8
Methyl Methacrylate	61.8	67.7	9.05	3.56	1.44	68.8	72.0	39.7
Styrene**	67.3	74.1	8.61	3.66	1.53	67.6	67.5	41.6
Ethylbenzene	65.5	71.8	9.07	3.64	1.52	66.8	68.8	40.6
Diisobutylene	71.1	78.2	8.74	3.61	1.49	65.3	67.6	45.8
Trimethylpentane	67.0	73.6	8.78	3.85	1.73	63.8	67.1	40.7

*Pyritic sulfur calculated from total sulfur and ash.

**6.4 lb/T styrene used.

coal show that the effect of monomer addition on the flotation product was nearly identical to the effect of the corresponding saturated equivalent. This suggests that any increase in recovery with the monomer is not due to graft polymer formation but rather to an increase in the amount of hydrocarbons in the flotation pulp. Furthermore, the increase in CMR was generally offset

Table 9.3 - Effect of polymerizable monomer addition on the flotation of 200-mesh dry-ground Pittsburgh No. 8 coal (4.8 lb/T of modifier used for all tests).

Modifier	Yield %	FLOTATION PRODUCT ANAL				REJECTION		EI _H —
		CMR %	Ash %	Tot S %	Pyr S* %	Ash %	Pyr S %	
Standard test (no modifier)	77.5	82.1	6.54	2.59	1.78	57.0	67.1	49.2
Vinyl Acetate	84.9	89.8	6.66	2.78	1.37	52.1	58.3	47.9
Methyl methacrylate	87.1	92.1	6.75	2.86	1.45	50.2	54.5	46.6
Styrene	83.6	88.5	6.63	2.78	1.37	53.0	58.8	47.3
Ethylbenzene	84.5	89.1	6.95	2.85	1.45	50.0	55.9	45.0
Diisobutylene	90.2	95.1	7.01	3.17	1.78	46.4	42.2	37.3
Trimethylpentane	89.6	94.0	7.43	3.35	1.96	43.6	36.8	30.8

*Pyritic sulfur calculated from total sulfur and ash.

Table 9.4 - Effect of polymerizable monomer addition on the flotation of 200-mesh dry-ground Upper Freeport PA coal (4.8 lb/T modifier used for all tests).

<u>Modifier</u>	<u>Yield</u> <u>%</u>	<u>FLOTATION PRODUCT ANAL</u>				<u>REJECTION</u>		<u>El_H</u>
		<u>CMR</u> <u>%</u>	<u>Ash</u> <u>%</u>	<u>Tot S</u> <u>%</u>	<u>Pyr S*</u> <u>%</u>	<u>Ash</u> <u>%</u>	<u>Pyr S</u> <u>%</u>	
Standard test (no modifier)	74.1	78.6	7.18	1.41	0.73	57.5	62.8	41.4
Vinyl Acetate	74.0	78.5	7.16	1.44	0.76	57.6	61.6	40.1
Methyl methacrylate	72.5	77.0	7.11	1.26	0.62	58.8	69.4	46.4
Styrene	74.7	79.3	7.12	1.49	0.80	57.5	59.2	38.5
Ethylbenzene	74.0	78.5	7.13	1.38	0.71	57.8	64.0	42.5
Diisobutylene	76.2	80.4	7.67	1.40	0.73	53.3	62.1	42.5

*Pyritic sulfur calculated from total sulfur and ash.

by a decrease in pyritic sulfur rejection, resulting in the flotation efficiency index remaining nearly constant or exhibiting a slight decrease compared to the standard flotation test.

Commercially available monomers are generally sold with an inhibitor to prevent the possible occurrence of a violent polymerization reaction. In addition to preventing self-polymerization, it was thought that the inhibitor may also be preventing the formation of graft polymers with the coal surface. In order to determine the inhibitor's effect on this process, vinyl acetate was distilled to remove the inhibitor and the pure monomer was ground with Illinois No. 6 and Pittsburgh No. 8 coals to compare with the previous results of vinyl acetate containing inhibitor. The results for the two coals, presented in Tables 9.5 and 9.6, show that the absence of inhibitor did not significantly affect the flotation results of either coal. Therefore, the lack

Table 9.5 - Effect of vinyl acetate (4.8 lb/T) with and without inhibitor on the flotation response of 200-mesh dry-ground Illinois No. 6 coal.

	<u>Yield</u> <u>%</u>	<u>FLOTATION PRODUCT ANAL</u>				<u>REJECTION</u>		<u>El_H</u>
		<u>CMR</u> <u>%</u>	<u>Ash</u> <u>%</u>	<u>Tot S</u> <u>%</u>	<u>Pyr S*</u> <u>%</u>	<u>Ash</u> <u>%</u>	<u>Pyr S</u> <u>%</u>	
With Inhibitor	62.9	69.1	8.92	3.66	1.44	67.9	68.8	37.9
Inhibitor Removed	63.9	70.0	8.99	3.84	1.58	67.2	65.0	35.0
Standard Test	60.4	66.0	9.31	3.57	1.36	68.6	72.4	38.4

* Pyritic sulfur calculated from total sulfur and ash.

Table 9.6 - Effect of vinyl acetate (4.8 lb/T) with and without inhibitor on the flotation of 200-mesh dry-ground Pittsburgh No. 8 coal.

	Yield %	FLOTATION PRODUCT ANAL				REJECTION		El _H
		CMR %	Ash %	Tot S %	Pyr S* %	Ash %	Pyr S %	
With Inhibitor	84.9	89.8	6.66	2.78	1.37	52.1	58.1	47.9
Inhibitor Removed	87.3	92.4	6.64	2.98	1.58	50.1	50.3	42.7
Standard Test	77.5	82.1	6.54	2.59	1.18	57.0	67.1	49.2

* Pyritic sulfur calculated from total sulfur and ash.

of graft polymer formation can be attributed to chemical properties of the coal and the monomer, and not to the presence of the inhibitor. It was pointed out by Ingberman of EOS Technologies that free radicals formed during the grinding process could be dissipated by delocalization by the highly aromatic nature of the coal, yielding a stable, low-energy free radical (1).

Finally, the effect of the mode of grinding, dry or wet, and the point of monomer addition, to the mill or to the cell, was investigated and the results presented in Tables 9.7 and 9.8. The results in Table 9.7 indicate that wet grinding with diisobutylene results in approximately the same increase in yield over the Standard Flotation Test as did the dry-grinding tests. The addition of diisobutylene to the cell (Table 9.8) results in a larger increase in the flotation yield and ash in the clean coal than did its addition to the mill. This is probably due to the absorption of diisobutylene by the coal during grinding.

Contact angles were measured on Pittsburgh No. 8 and Upper Freeport PA coals that had been treated with diisobutylene. The results are presented in Table 9.9 along with the contact angles of the coals without any modifier present. The increases in the contact angles indicate

Table 9.7 - Effect of diisobutylene (4.8 lb/T) addition to the mill on the flotation response of 200-mesh wet and dry-ground Pittsburgh No. 8 coal.

REAGENT DOSAGE			Yield %	PRODUCT ANALYSIS			REJECTION		El _H
Coll. lb/T	Frot. lb/T	Grind mode		CMR %	Ash %	Pyr S %	Ash %	Pyr S %	
1.90	0.30	wet	79.4	85.2	5.39	1.17	63.7	66.6	52
2.16	0.40	dry	90.2	95.1	7.01	1.78	46.4	42.2	37

Table 9.8 - Comparison of the effect of diisobutylene addition (4.8 lb/T) to the mill with that to the cell on the flotation response of 200-mesh wet-ground Pittsburgh No. 8 coal.

Point of Diisobutylene Addition	Yield %	CMR %	Ash %	Ash Rej %
to the cell	89.4	93.2	8.05	39.0
to the mill	79.4	85.2	5.39	63.7

that the diisobutylene makes the surface of the coals more hydrophobic. However, as previous results suggest, the mechanism responsible for this increase is probably not the formation of graft polymers at the surface. In the case of Upper Freeport PA coal, the increases in hydrophobicity is not associated with a significant increase in the flotation yield (Table 9.4) probably due to the high degree of hydrophobicity characteristic of this coal. On the other hand, the increase in the contact angle of the treated Pittsburgh No. 8 sample correlates well with the increase in flotation yield. The efficiency index of the flotation test with diisobutylene (Table 9.3) indicates that this reagent is not preferentially rendering the carbonaceous material more hydrophobic but the sulfur-bearing components as well.

9.2 Effect of Anionic Reagents

The anionic reagents tested in this study were Aerosol OT, 4-tert-butylpyridine and 4-(1-butylpentyl) pyridine. Aerosol OT is a water-soluble reagent, while the latter two are oily reagents. Aerosol OT, 2-ethylhexylsulfosuccinate, is a widely used class of surfactants in the mineral industry. The alkylated pyridines were chosen primarily because they could interact with the acid groups, $-\text{COO}^-\text{H}^+$ and RO^-H^+ , produced on the coal surface by air oxidation that tend

Table 9.9 - Contact angle measurement by the sessile drop method on flotation feed samples of Pittsburgh No. 8 and Upper Freeport PA coal treated with diisobutylene.

Sample	ADVANCING CONTACT ANGLE, DEGREES	
	Pittsburgh No. 8	Upper Freeport PA
Without Diisobutylene	38	47
With Diisobutylene	51	61

to make the coal surface hydrophilic and, thus restore the hydrophobicity to its unoxidized levels. The reaction product was conjectured to be a water insoluble pyridinium salt. The majority of the studies with these anionic surface-modifying reagents were carried out with minus 200-mesh wet-ground Pittsburgh No. 8 coal. The results are presented in terms of selectivity curves described by plots of pyritic sulfur or non-pyritic ash rejection versus combustible material recovery.

9.2.1 Aerosol OT

The effect of Aerosol OT (AOT) was evaluated under different conditions and using three different reagent schemes: addition of AOT to the cell, addition of AOT to the mill, and addition of AOT and MIBC to the cell. Selectivity curves obtained for the flotation of wet-ground Pittsburgh No. 8 coal with AOT are compared with that achieved using the standard collector (n-dodecane) addition and varying amounts of MIBC in Figures 9.1 and 9.2 for pyrite and non-pyritic ash rejections respectively. It can be seen from these figures that the point of addition of AOT (mill or cell) or with or without MIBC did not cause a significant improvement in either the rejection of pyrite or the non-pyritic minerals over that obtained with dodecane. In fact, there appears to be a marginal decrease in selectivity (less pronounced in the case of non-pyritic minerals) with AOT addition.

The effect of AOT addition to the mill on the rejection of pyrite and non-pyritic minerals from minus 200-mesh wet-ground Illinois No. 6 coal is shown in Figures 9.3 and 9.4 respectively. It can be seen from these figures that although the regions spanned by the curves with and without AOT addition are somewhat different, there is a region where a comparison can be made. This comparison shows that the addition of AOT, may cause a reduction in the selectivity over that of the standard test. This is in concurrence with the results obtained for Pittsburgh No. 8 coal.

In the case of Upper Freeport PA coal, the performance of AOT is similar to those obtained for Pittsburgh No. 8 and Illinois No. 6. The addition of AOT had no significant effect on the rejection of pyritic sulfur (Figure 9.5) or non-pyritic minerals (Figure 9.6).

Figure 9.1 - Effect of Aerosol OT addition on the rejection of pyrite from wet ground minus 200-mesh Pittsburgh No. 8 coal.

Figure 9.2 - Effect of Aerosol OT addition on the rejection of non-pyritic minerals from wet ground minus 200-mesh Pittsburgh No. 8 coal.

Figure 9.3 - Effect of Aerosol OT addition on the rejection of pyrite from wet ground minus 200-mesh Illinois No. 6 coal.

Figure 9.4 - Effect of Aerosol OT addition on the rejection of non-pyritic minerals from wet ground minus 200-mesh Illinois No. 6 coal.

Figure 9.5 - Effect of Aerosol OT addition on the rejection of pyrite from wet ground minus 200-mesh Upper Freeport PA coal.

Figure 9.6 - Effect of Aerosol OT addition on the rejection of non-pyritic minerals from wet ground minus 200-mesh Upper Freeport PA coal.

9.2.2 Alkylated pyridines

In the case of alkylated pyridines, 4-tert-butylpyridine and 2-(1-butylpentyl)pyridine, the reagents were added to the mill, while dodecane and MIBC were added to the cell in all experiments. Dosage of the reagents were chosen based on preliminary studies. It can be seen from the selectivity curves for pyritic sulfur shown in Figures 9.7 through 9.10 that the selectivity obtained in the presence of these reagents is lower than the obtained using the dodecane-MIBC combination. However, in terms of the rejection of non-pyritic minerals, the addition of alkylated pyridines has no significant effect on selectivity (Figures 9.8 and 9.10). Alkylated pyridines do not appear to improve the froth characteristics. However, the addition of alkylated pyridines seems to have enhanced the hydrophobicity of pyrite, relative to that of coal, as observed with alkylated thiophene, a non-ionic reagent also tested and reported later in this chapter.

9.2.3 Summary

The results of bench scale flotation studies conducted to determine the effect of water soluble and oily anionic reagents on the rejection of pyrite and non-pyritic minerals from the three base coals, can be summarized as follows:

- AOT addition was found to cause either no effect or a marginal reduction in the rejection of pyritic sulfur and non-pyritic minerals from the three base coals. The marginal decrease in selectivity due to AOT addition is probably due to enhanced water flow that can cause increased entrainment (mechanical carry-over) of the gangue/impurity minerals.
- Increase in pyrite hydrophobicity, relative to coal, due to oily reagents addition was attributed to the decrease in selectivity obtained with respect to the rejection of pyritic sulfur from Pittsburgh No. 8 coal upon addition of 4-tert-butylpyridine and 2-(1-butylpentyl)pyridine.

Figure 9.7 - Effect of 4- tert. butylpyridine addition on the rejection of pyrite from wet-ground minus 200-mesh Pittsburgh No. 8 coal.

Figure 9.8 - Effect of 4- tert. butylpyridine addition on the rejection of non-pyritic minerals from wet ground minus 200 mesh Pittsburgh No. 8 coal.

Figure 9.9 - Effect of 4-(1-butylpentyl)pyridine addition on the rejection of pyrite from wet ground minus 200-mesh Pittsburgh No. 8 coal.

Figure 9.10 - Effect of 4-(1-butylpentyl)pyridine addition on the rejection of non-pyritic minerals from wet ground minus 200-mesh Pittsburgh No. 8 coal.

9.3 Flotation with Non-ionic Surfactants

Characterization studies of the three base coals have shown repeatedly that Illinois No. 6 is the most hydrophilic coal. In order to float this coal at fine sizes, a substantial amount of collector is needed. However, kerosene (or dodecane) is not highly selective and large additions of oil to the flotation pulp can result in low ash and pyritic sulfur rejections. For relatively hydrophilic coal, larger dodecane additions are needed to recover the combustible material and hence the amount of pyritic sulfur and ash in the clean coal also increases. Therefore, consideration has been given to reagents that were thought to function as collectors with better selectivity than dodecane. A variety of non-ionic reagents were tested. The final set of reagents, referred to as the GH series, contain hydrocarbon groups with various functional groups attached. Another type of reagent tested was an alkylated thiophenyl that was chosen because it may interact with organic sulfur on the coal surface. Finally, the experimental results of a study on the effect of dodecane, a non-ionic surfactant, on pyritic sulfur rejection is presented.

9.3.1 GH Series of Reagents

The flotation response of 200-mesh wet-ground Illinois No. 6 coal floated with dodecane and the GH reagents are shown in Figure 9.11. The upper set of plots shows the yield as a function of reagent dosage in pounds per ton and the bottom set presents the same data plotted as moles per ton. This figure clearly shows that the GH series of reagents function as collectors and produce comparable results at one-tenth the molar dosage required with dodecane. As can be seen from the summary given in Table 9.10, the flotation products obtained when GH0 is used in place of dodecane result in efficiency indexes that are equivalent to or better than those obtained with dodecane. It should be noted that GH0 is commercially available and at this dosage level would be more economical than dodecane.

The effectiveness of the GH series of reagents was also tested on Pittsburgh No. 8 coal and the results are shown in Figure 9.12 and Table 9.11. These results were not encouraging and indicate that the interaction of the reagents is coal specific. The results for Upper Freeport PA showed the GH reagents have even less of an effect compared to that with Pittsburgh No. 8 coal.

Table 9.10 - Effect of GH reagents on the flotation of 200-mesh wet-ground Illinois No. 6 coal.

REAGENT DOSAGE			Yield %	PRODUCT ANALYSIS			REJECTION		EI _H
Coll. lb/T	MIBC lb/T	Mod. lb/T		CMR %	Ash %	Pyr S %	Ash %	Pyr S %	
5.75	1.16	-	78.8	83.1	8.94	1.54	48.5	59.5	43
8.63	1.16	-	80.6	85.4	8.52	1.47	49.8	60.5	46
11.50	1.16	-	84.3	89.3	8.58	1.42	47.1	60.1	49
14.38	1.16	-	83.0	87.7	8.79	1.50	46.6	58.5	46
GH0	1.16	0.52	75.3	79.3	9.03	1.25	50.3	68.6	48
	1.16	0.78	82.8	86.9	9.43	1.37	42.9	62.2	49
	1.16	1.17	91.8	95.5	10.18	1.51	31.6	53.8	49
GH1.5	1.16	0.58	62.9	66.3	9.04	1.29	58.4	73.0	39
	1.16	0.86	77.6	80.9	9.99	1.54	43.3	60.2	41
	1.16	1.30	87.5	90.6	10.64	1.77	31.9	48.4	39
GH4	1.16	0.58	66.2	69.5	9.35	1.61	54.7	64.5	34
	1.16	0.88	76.8	81.2	8.78	1.26	50.7	67.7	49
	1.16	1.17	82.3	86.6	9.21	1.20	44.6	67.1	54
	1.16	1.31	86.5	90.3	9.84	1.69	37.7	51.3	42
GH6	1.16	0.59	26.1	27.9	7.84	0.94	85.0	91.8	20
	1.16	0.88	37.0	39.5	7.80	1.03	78.9	87.3	27
	1.16	1.18	47.3	50.4	7.95	1.09	72.5	82.8	33
	1.16	1.32	66.6	70.8	8.21	1.21	60.0	73.1	44

Table 9.11 - Effect of GH reagents on the flotation of 200-mesh wet-ground Pittsburgh No. 8 coal.

REAGENT DOSAGE			Yield %	PRODUCT ANALYSIS			REJECTION		EI _H
Coll. lb/T	MIBC lb/T	Mod. lb/T		CMR %	Ash %	Pyr S %	Ash %	Pyr S %	
1.92	0.30	-	77.0	82.6	5.27	1.17	65.3	67.6	50
2.88	0.30	-	81.7	87.5	5.44	1.27	62.0	62.7	50
3.84	0.30	-	82.9	89.0	5.20	1.21	63.2	63.9	53
5.76	0.30	-	81.3	86.8	5.68	1.61	60.5	52.9	40
GH0	0.30	0.78	85.9	91.5	5.94	1.64	56.4	49.3	41
	0.30	0.91	85.5	90.8	6.21	1.73	54.6	46.8	38
	0.30	1.04	88.9	94.6	6.06	1.58	54.0	49.5	44
GH4	0.30	0.58	52.0	56.1	4.82	0.95	78.8	82.2	38
	0.30	0.88	65.7	70.5	5.38	1.03	70.0	75.7	46
	0.30	1.17	87.7	93.1	6.38	1.47	52.6	53.6	47
GH6	0.30	0.88	47.2	51.0	4.67	1.06	81.2	82.0	33
	0.30	1.03	53.6	57.6	5.03	1.16	77.0	77.6	35
	0.30	1.18	82.2	87.7	5.78	1.48	59.4	56.2	44

Figure 9.11 - Comparison of the collecting ability of the GH series of reagent with that of dodecane in the flotation of 200-mesh wet-ground Illinois No. 6 coal.

Figure 9.12 - Comparison of the collecting ability of GH series with that of dodecane in the flotation of 200-mesh wet-ground Pittsburgh No. 8 coal.

Table 9.12 - The effect of GH0 and GHB on the flotation of 200-mesh wet-ground Illinois No. 6 coal.

<u>REAGENT DOSAGE</u>			<u>Yield</u> <u>%</u>	<u>PRODUCT ANALYSIS</u>			<u>REJECTION</u>		<u>EI_H</u> <u>—</u>
<u>Dodecane</u> <u>lb/T</u>	<u>MIBC</u> <u>lb/T</u>	<u>Mod.</u> <u>lb/T</u>		<u>CMR</u> <u>%</u>	<u>Ash</u> <u>%</u>	<u>Pyr S</u> <u>%</u>	<u>Ash</u> <u>%</u>	<u>Pyr S</u> <u>%</u>	
<u>GH0</u>									
0.0	1.16	0.52	75.3	79.3	9.03	1.25	53.4	59.9	40
0.0	1.16	0.78	82.8	86.9	9.43	1.37	46.5	51.7	40
0.0	1.16	1.17	91.8	95.5	10.18	1.51	36.0	41.0	38
<u>GHB</u>									
0.0	1.16	0.52	55.1	59.0	7.51	--	--	--	--
0.0	1.16	0.78	64.2	68.6	7.73	--	--	--	--
0.0	1.16	1.17	77.8	82.9	8.04	--	--	--	--

The GH series of surface modifiers contains a phenolic group which may play an important role in the interaction of the reagents with the surface of coal. This interaction may be enhanced if the surface of the coal contains numerous oxygen-containing groups as in the case of Illinois No. 6 coal or a coal that has been weathered. In order to verify this hypothesis GHB, that has a structure similar to GH0 but lacks the phenolic group in its structure, was added as a flotation modifier for Illinois No. 6 coal. Indeed, the results (Table 9.12) show that at equal additions, the yields obtained with GH0 are significantly higher than that obtained with GHB. The ash in the clean coal is also reduced by about 28% with GHB. It is postulated that higher yields obtained with GH0 are due to the interaction of its phenol group with the hydrophilic sites of the coal. Likewise the phenolic group is probably responsible for the high ash recoveries as compared to dodecane and to the improved flotation results obtained with Illinois No. 6, which contains more oxygen function groups than the other two coals.

Because of the large number of hydrophilic oxygen functional groups present on oxidized coal, the effect of the GH series of reagents on laboratory-oxidized coal was investigated. Laboratory-oxidized coal was prepared by first grinding Pittsburgh No. 8 coal to minus 200-mesh following our standard grinding procedures. The ground coal was then stored in slurry form for 120 hours at 60°C and additional distilled water was added periodically to keep the sample from drying out. This sample was then used for flotation tests with dodecane, GH0, and GH4 added

Table 9.13 - The effect of dodecane, GH0 and GH4 reagents on the flotation of lab-oxidized 200-mesh wet-ground Pittsburgh No. 8 coal.

	Dodecane lb/T	MIBC lb/T	Modifier lb/T	Yield %	Ash %	CMR %	Ash Rej %
	1.92	0.30	0.0	16.9	4.44	18.3	93.6
	3.84	0.30	0.0	47.1	4.50	50.9	81.9
GH0	0.0	0.30	0.13	47.0	4.83	50.7	80.6
	0.0	0.30	0.78	80.7	6.17	85.8	57.4
GH4	0.0	0.30	1.02	79.4	6.98	83.6	52.6

to the flotation cell. From the results presented in Table 9.13, it can be seen that the yield is very low when dodecane is used as collector. When either GH0 or GH4 is used, however, the yield returned to levels equivalent to those obtained with unoxidized coals. These results indicate that both GH0 and GH4 appear to have the ability to restore the flotability of oxidized coal. However, the ash reporting to the clean coal is higher than that before the sample was oxidized. There is not enough data to determine if the GH reagents are less selective in terms of ash than dodecane for oxidized coal.

9.3.2 Alkylated thiophene

The effect of 2,n-butylthiophene addition on the rejection of pyritic sulfur and non-pyritic minerals is shown in Figures 9.13 and 9.14 respectively. It can be seen from these figures that this reagent has no effect on the efficiency of pyrite removal, as seen from the overlap of the selectivity curves in Figure 9.13, while the rejection of non-pyritic minerals showed an improvement as seen by the shift of the selectivity curve towards the upper right hand corner (higher selectivity region). Enhanced non-pyritic minerals rejection can be due to possible improvement in froth characteristics (less viscous froth) caused by 2,n-butylthiophene addition and, as a result, more rapid drainage of impurities from the froth layer. However, since pyrite rejection did not improve with 2,n-butylthiophene addition, the above hypothesis would imply that increased pyrite rejection obtained due to improved froth characteristics is probably offset by enhanced pyrite flotation due to possible increase in pyrite hydrophobicity (relative to that of coal).

Figure 9.13 - Effect of 2,n-butylthiophene addition on the rejection of pyrite from wet ground minus 200 mesh Pittsburgh No. 8 coal.

Figure 9.14 - Effect of 2,n-butylthiophene addition on the rejection of non-pyritic minerals from wet ground minus 200 mesh Pittsburgh No. 8 coal.

9.3.3 Dodecane

Results obtained with alkylated thiophene and alkylated pyridines suggested that the percentage increase in the hydrophobicity of pyrite was higher than that obtained for coal with the addition of these oily reagents. Since pyrite surfaces apparently possess no groups capable of bonding with the functional groups present in either alkylated thiophenes or the alkylated pyridines, the only possible mechanism by which pyrite surface hydrophobicity can be enhanced by these oily reagents is by physically coating the particle surface by oily drops due to van der Waals forces and hydrophobic interactions. In order to test this hypothesis, tests were conducted to determine the effect of an increase in dodecane dosage on pyrite and non-pyritic minerals rejection. Since dodecane does not possess any functional group, hydrophobic or van der Waals attraction is the only mechanism by which this reagent can coat the surface of either the coal or the pyrite. The effect of increase in dodecane dosage on the rejection of pyrite is plotted in Figure 9.15. This figure clearly shows that the selectivity is reduced with increasing dodecane dosage. The detrimental effect of dodecane can be more clearly seen in Figure 9.16 in which the results are shown for a two-stage flotation test. In the case of non-pyritic minerals, an increase in dodecane dosage has no significant effect on selectivity (Figures 9.17 and 9.18) suggesting that the froth characteristics are not altered by the addition of dodecane and that the effect observed for pyrite is probably due entirely to changes in surface properties. These results strongly support the hypothesis that oily reagents (alkylated thiophenes and pyridines) coat the surface of coal and pyrite through non-specific or low energy interactions and that they increase the pyrite hydrophobicity relative to that of coal. The reason for the latter phenomenon may be due to the inherent hydrophobicity of coal surface relative to that of pyrite. This is also manifested in high coal yields ($\sim 90\text{wt}\%$) with frother only as compared to low pyrite yield ($\sim 25\text{wt}\%$). Therefore, the percentage increase in the hydrophobicity of pyrite surface is likely to be higher for coal than pyrite.

In conclusion, the hypothesis that the increase in the hydrophobicity of pyrite due to the addition of oily reagents is higher than that for coal was further evidenced from the tests conducted with dodecane. In these tests, it was found that pyrite rejection deteriorated upon increasing the dosage of dodecane. Since dodecane dosage had no effect on the non-pyritic

Figure 9.15 - Effect of dodecane addition on the rejection of pyrite from wet ground minus 200-mesh Pittsburgh No. 8 coal - single stage flotation results.

Figure 9.16 - Effect of dodecane addition on the rejection of pyrite from wet ground minus 200-mesh Pittsburgh No. 8 coal - two-stage flotation results.

Figure 9.17 - Effect of dodecane addition on the rejection of non-pyritic minerals from wet ground minus 200-mesh Pittsburgh No. 8 coal - single stage flotation results.

Figure 9.18 - Effect of dodecane addition on the rejection of non-pyritic minerals from wet ground minus 200 mesh Pittsburgh No. 8 coal - two-stage flotation results.

Table 9.14 - Effect of methanol addition to the grinding mill on the flotation response of 200-mesh wet-ground Upper Freeport PA coal.

REAGENT DOSAGE			Yield %	PRODUCT ANALYSIS			REJECTION		EI _H —
Dodecane lb/T	MIBC lb/T	Mod. lb/T		CMR %	Ash %	Pyr. S %	Ash %	Pyr. S %	
0.48	0.21	0.0	68.2	73.8	5.3	0.51	71.1	78.5	52.3
0.48	0.00	2.0	32.4	35.5	4.2	0.27	89.6	94.7	30.2
0.48	0.09	2.0	54.1	59.2	4.3	0.31	81.5	89.7	50.0
0.48	0.18	2.0	67.5	73.3	5.0	0.27	73.3	88.8	62.1
0.48	0.27	2.0	82.7	88.3	6.5	0.43	58.2	78.1	66.4
0.48	0.00	6.0	35.5	38.8	4.4	0.28	87.4	93.6	32.3
0.48	0.09	6.0	63.7	69.3	4.9	0.30	75.7	88.2	57.5
0.48	0.18	6.0	74.1	79.8	5.7	0.38	67.0	82.7	62.5
0.48	0.27	6.0	80.3	86.3	6.0	0.38	60.4	81.2	67.5
0.48	0.00	12.0	36.1	39.6	4.0	0.30	88.5	93.1	32.7
0.48	0.09	12.0	51.8	56.6	4.5	0.30	81.8	90.5	47.0
0.48	0.18	12.0	64.4	70.1	4.4	0.32	75.4	87.2	57.3
0.48	0.27	12.0	80.0	85.9	6.0	0.40	62.1	80.3	66.1

minerals rejection, the possibility that the increase was due to a change in froth properties and its consequent effect on pyrite rejection was rejected. The relative increase in pyrite hydrophobicity, thus, appeared to be the reason for reduced pyrite rejection obtained upon the addition of oily reagents. In the light of these findings, it is recommended that the use of oily reagents be minimized in coal/pyrite flotation systems. Addition of oily reagents can significantly improve the coal recovery and, since they do not have any significant effect on non-pyritic minerals rejection, oily reagents can be used to achieve improved performance in coal/non-pyritic minerals (low sulfur coals) flotation system.

9.4 Reactive non-Polymerizable Reagents

9.4.1 Alcohol Homologues

Initial studies with reactive but nonpolymerizable reagents involved the delineation of the effect of methanol and ethanol addition to the grinding mill on the flotation separation of 200-mesh wet-ground coal. Results with methanol on the flotation of Upper Freeport coal (Table 9.14) indicate that methanol improves the separation efficiency (EI_H) by as much as 15 percent, as compared to the standard flotation test; however, beyond 2 lb/T an increase in methanol does not result in improved efficiency. In addition, methanol does not replace the need for a frother

Table 9.15 - Effect of methanol addition on the flotation response of 200-mesh wet-ground Pittsburgh No. 8 coal.

REAGENT DOSAGE			PRODUCT ANALYSIS				REJECTION		EI _H
Dodecane lb/T	MIBC lb/T	Mod. lb/T	Yield %	CMR %	Ash %	Pyr. S %	Ash %	Pyr. S %	
1.9	0.30	--	74.7	80.0	5.1	1.16	66.6	69.3	49.3*
1.9	0.00	6.0	25.6	27.5	4.3	0.80	89.9	92.7	27.5
1.9	0.10	6.0	41.4	44.6	4.2	0.82	84.0	88.0	32.5
1.9	0.20	6.0	67.1	71.8	4.7	0.94	71.0	77.6	49.4
1.9	0.30	6.0	73.8	78.7	4.7	0.93	67.6	75.7	54.4

*Standard test

and the flotation performance is very sensitive to the MIBC dosage. With no MIBC the efficiency index (EI_H) was about 30 regardless of the methanol addition (above 2 lb/T) and with 0.09 lb/T MIBC, the EI_H increased to around 50 percent.

The results with Pittsburgh No. 8 (Table 9.15) show that methanol has only a slight effect on the efficiency index with the maximum improvement limited to about a 5% increase in the performance. The flotation results with Illinois No. 6 with and with out methanol were identical to each other indicating that this alcohol has no effect on the flotation of this coal.

The effect of alcohol on the flotation performance of fine coal was then investigated for ethanol, the next alcohol in the homologous series after methanol. The results of this investigation are given in Table 9.16 for Upper Freeport PA coal. Unlike methanol, the efficiency index appears to go through a slight maximum at 6 lb/T ethanol addition as the index is consistently 2-5 % higher than the results obtained with 3 or 9 lb/T addition. The increase in efficiency index brought about by alcohol addition is roughly ten percent over that obtained for the standard flotation test.

As with methanol, ethanol increases the flotation efficiency index of Pittsburgh No. 8 (Table 9.17) by about 10 percent and has no effect on the flotation of Illinois No. 6. These improved results with ethanol over those of methanol suggests that longer chain alcohols may have an even greater beneficial effect on the flotation of Upper Freeport PA coal. Therefore, iso-propanol, a branched three-carbon alcohol, and butanol, a straight chained four-carbon alcohol were tested with Upper Freeport coal. The results obtained with each of the four alcohols

Table 9.16 - Effect of ethanol addition on the flotation response of 200-mesh wet-ground Upper Freeport PA coal.

REAGENT DOSAGE			Yield %	PRODUCT ANALYSIS			REJECTION		EI _H —
Dodecane lb/T	MIBC lb/T	Mod. lb/T		CMR %	Ash %	Pyr. S %	Ash %	Pyr. S %	
0.48	0.21	0.0	68.2	73.8	5.3	0.51	71.1	78.5	52.3
0.00	0.00	1.00	24.2	26.7	3.5	0.24	93.2	96.4	23.1
0.00	0.00	2.00	34.3	37.7	3.8	0.27	89.5	94.3	32.0
0.00	0.00	3.00	34.5	38.0	3.5	0.25	90.4	94.7	32.7
0.00	0.00	6.00	48.5	53.3	3.9	0.27	84.9	91.9	45.2
0.23	0.23	3.00	81.5	86.5	7.1	0.55	55.0	72.4	58.9
0.23	0.27	3.00	73.7	79.0	6.2	0.44	64.1	80.0	59.0
0.23	0.36	3.00	81.8	87.1	6.7	0.48	57.5	75.8	62.9
0.23	0.45	3.00	85.2	90.5	7.1	0.51	51.6	73.2	63.7
0.23	0.23	6.00	82.7	87.5	7.5	0.53	51.9	72.9	60.4
0.23	0.27	6.00	81.1	86.4	6.8	0.46	57.1	77.0	63.4
0.23	0.36	6.00	83.8	89.0	7.1	0.47	52.8	75.7	64.7
0.23	0.45	6.00	85.4	89.0	7.4	0.52	50.3	72.6	61.6
0.23	0.23	9.00	84.4	89.7	7.0	0.56	52.3	70.8	60.5
0.23	0.27	9.00	77.0	82.5	6.2	0.46	61.6	78.2	60.7
0.23	0.36	9.00	78.3	83.9	6.2	0.47	60.0	77.3	61.2
0.23	0.45	9.00	82.3	87.8	6.6	0.52	55.9	73.6	61.4

(6 lb/T) with neither collector nor frother, with collector alone, and with both collector and frother, are given in Table 9.18. This table shows that flotation efficiencies greater than 60 percent are produced by butanol regardless of collector or MIBC addition, by isopropanol and butanol when dodecane is also added, and by all four alcohols when both collector and frother

Table 9.17 - Effect of ethanol addition on the flotation response of 200-mesh wet-ground Pittsburgh No. 8 coal.

REAGENT DOSAGE			Yield %	PRODUCT ANALYSIS			REJECTION		EI _H —
Dodecane lb/T	MIBC lb/T	Mod. lb/T		CMR %	Ash %	Pyr. S %	Ash %	Pyr. S %	
1.9	0.30	--	74.7	80.0	5.1	1.16	66.6	69.3	490*
1.9	0.00	6.00	39.4	42.3	4.7	0.89	83.7	87.6	29.9
1.9	0.10	6.00	54.0	58.1	4.5	0.88	78.2	83.2	41.2
1.9	0.20	6.00	69.3	74.3	4.7	0.99	70.4	75.6	49.9
1.9	0.30	6.00	82.2	88.4	5.1	1.03	64.3	70.0	58.3

*Standard test

Table 9.18 - Flotation results using alcohol homologs in the mill with 200-mesh wet-ground Upper Freeport PA coal. 6 lb/T of alcohol was used for each test.

REAGENT DOSAGE			PRODUCT ANALYSIS				REJECTION		EI _H
Modifier	Dodecane lb/T	MIBC lb/T	Yield %	CMR %	Ash %	Pyr. S %	Ash %	Pyr. S %	
Methanol	0.0	0.0	25.5	28.7	3.77	0.23	92.3	96.6	25.3
Ethanol	0.0	0.0	48.5	53.2	3.91	0.27	84.9	91.9	45.1
I-propanol	0.0	0.0	47.2	52.1	3.88	0.26	85.8	92.4	44.5
Butanol	0.0	0.0	73.6	80.3	4.99	0.32	71.6	85.5	65.8
Methanol	0.48	0.0	36.1	39.6	3.97	0.30	88.5	93.1	32.7
Ethanol	0.48	0.0	58.6	63.7	4.96	0.29	76.5	89.4	53.1
I-propanol	0.48	0.0	71.4	77.6	4.80	0.38	72.6	83.3	60.9
Butanol	0.48	0.0	87.6	93.5	7.00	0.50	52.3	72.9	66.4
Methanol	0.48	0.27	79.9	85.9	5.95	0.40	62.1	80.3	66.2
Ethanol	0.48	0.27	84.8	90.6	6.52	0.41	54.5	78.6	69.2
I-Propanol	0.48	0.27	88.1	93.9	7.98	0.55	51.2	70.1	64.0
Butanol	0.48	0.27	89.7	95.3	7.33	0.61	48.4	66.2	61.5

are added to the flotation cell. In general, when higher molecular weight alcohols are added as flotation reagents the amount of collector and frother can be decreased.

Optimization tests with butanol were performed on Upper Freeport coal to determine the best separation that could be obtained with this alcohol. Representative results are presented in Table 9.19 and show that by using butanol in place of MIBC (however, at a higher concentration) an efficiency index of 70.4 percent can be achieved. This represents a CMR of close to 90 percent and a pyrite rejection of over 80 percent. This compares with the optimized separation efficiency of 65.6 with 1.0 lb/T of MIBC and no collector.

During the development of the standard flotation test and further work on the optimization of the flotation of Upper Freeport PA coal, it was found that several different

Table 9.19 - Optimized flotation results using butanol added to the mill with 200-mesh wet-ground Upper Freeport PA coal.

REAGENT DOSAGE			PRODUCT ANALYSIS				REJECTION		EI _H
Dodecane lb/T	MIBC lb/T	Mod. lb/T	Yield %	CMR %	Ash %	Pyr. S %	Ash %	Pyr. S %	
0.3	0.0	4	81.5	87.5	5.58	0.34	62.3	82.9	70.4
0.4	0.1	4	83.6	90.0	6.78	0.45	57.7	76.8	66.8
0.3	0.1	6	87.8	93.0	6.08	0.44	57.7	76.2	69.2
0.4	0.0	6	86.9	92.1	5.94	0.44	54.2	76.4	68.5

combinations of frother and collector produced equivalent efficiency indexes. This included a slightly elevated collector addition with no frother, and vice-versa, a slightly elevated frother addition with no collector. It was obvious from the optimization and grinding studies that the frother, MIBC, was also adsorbed (absorbed) by the coal and not solely at the air/liquid interface as an ideal frother should. The alcohols tested in this program also appear to behave in the same manner as MIBC which is expected since MIBC (methylisobutylcarbinol) is 4-methyl-2-pentanol. Branching of the alcohol probably has an effect on the flotation performance and particularly on the dosage required since the results presented in Table 9.18 were all obtained with 6 lbs/T alcohol addition and similar results with MIBC are achieved with an order of magnitude less.

To verify this observation, a systematic study of the effect of molecular weight and structure of three different alcohols on the flotation of minus 200-mesh Pittsburgh No. 8 coal was completed with n-butanol, n-hexanol and MIBC. n-butanol was chosen since its molecular weight is lower than that of MIBC, while n-hexanol was selected because it has the same molecular weight but is structurally different (straight chain; smaller molecular dimension) from MIBC. A comparison of the performance of these frothers in terms of reagent consumption is shown in Figure 9.19. This figure shows that, to achieve a given flotation recovery, the amount of n-butanol required is much higher compared to those of the hexanol and MIBC. Between the 6 carbon alcohols, the performance of MIBC (secondary alcohol) is better than that of n-hexanol (a straight chain primary alcohol). These observations imply that the smaller the molecule the larger its consumption. This may be due to the sorption of frothers by coal, since, depending on the pore size, smaller molecules can penetrate into more pores than the larger molecules can. Therefore, it appears that more butanol is lost by absorption into the pores of the coal particles than either n-hexanol or MIBC, which leaves less reagent available for producing froth.

The effect of frother type on the selectivity with respect to pyrite and non-pyritic minerals is shown in Figures 9.20 through 9.23, respectively. It can be seen from these figures that the type of frother has no effect on the selectivity obtained in either single-stage flotation or two-stage flotation, for either pyrite or non-pyritic mineral rejection for the Pittsburgh No. 8 coal.

Figure 9.19 - Comparison of performances of butanol, hexanol and MIBC in terms of reagent consumption.

Figure 9.20 - Effect of frother type on pyrite rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal -- single-stage flotation.

Figure 9.21 - Effect of frother type on pyrite rejection from wet-ground minus 200 mesh Pittsburgh No. 8 coal -- two-stage flotation.

Figure 9.22 - Effect of frother type on non-pyritic minerals rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal -- single-stage flotation.

Figure 9.23 - Effect of frother type on non-pyritic minerals rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal -- two-stage flotation.

9.4.2 Butyl benzaldehyde

Flotation experiments with alkyl benzaldehydes were proposed in the work plan to determine if these compounds had an effect on coal flotation. Therefore, flotation experiments were performed with butyl benzaldehyde added to the grinding mill with Upper Freeport coal. The results for coal ground to minus 28 mesh and minus 200 mesh are given in Tables 9.20 and 9.21, respectively. These results show that the addition of butyl benzaldehyde does increase the flotation yield. However, the results for the minus 28-mesh material did not show any improvement over that of the standard flotation test and clearly show that an excess amount of this reagent is detrimental due to low ash and pyritic sulfur rejections. Associated with the low ash and pyritic sulfur rejection are CMR recoveries of over 90 percent. The reason for the low rejections may be poor selectivity of the reagent or particle hydrodynamics (poor frothing characteristics, poor drainage of froth, entrapment, entrainment).

Results with butyl benzaldehyde with minus 200-mesh material were slightly better than that of the standard test. An improvement in the efficiency index of close to 10 percent was

Table 9.20 - Effect of butyl benzaldehyde addition on the flotation response of 28-mesh wet-ground Upper Freeport PA coal.

REAGENT DOSAGE				PRODUCT ANALYSIS			REJECTION		El _H
Dodecane lb/T	MIBC lb/T	Mod. lb/T	Yield %	CMR %	Ash %	Pyr. S %	Ash %	Pyr. S %	
0.23	0.00	0.80	25.0	27.2	4.6	0.44	90.8	93.2	20.4
0.23	0.05	0.80	38.5	42.1	4.4	0.45	86.4	89.3	31.4
0.23	0.14	0.80	55.2	59.7	5.3	0.55	76.4	81.3	41.0
0.23	0.23	0.80	88.1	92.9	7.7	0.81	45.6	56.0	48.9
0.23	0.00	1.60	38.4	41.9	4.5	0.44	86.2	89.6	31.5
0.23	0.05	1.60	49.3	53.5	5.1	0.51	79.8	84.5	38.0
0.23	0.14	1.60	73.1	78.0	6.6	0.69	61.8	68.9	46.9
0.23	0.23	1.60	95.2	99.4	8.6	0.81	34.3	52.4	52.8
0.23	0.00	2.40	86.5	91.1	7.4	1.05	45.1	43.9	35.0
0.23	0.05	2.40	90.5	95.1	8.0	0.81	43.9	54.8	50.0
0.23	0.14	2.40	91.3	95.2	8.8	1.02	35.8	42.5	37.7
0.23	0.23	2.40	92.1	95.7	9.1	0.99	33.0	43.7	39.4

Standard El_H = 52.5

Table 9.21 - Effect of butyl benzaldehyde addition on the flotation response of 200-mesh wet-ground Upper Freeport PA coal.

REAGENT DOSAGE				PRODUCT ANALYSIS			REJECTION		El _H
Dodecane lb/T	MIBC lb/T	Mod. lb/T	Yield %	CMR %	Ash %	Pyr. S %	Ash %	Pyr. S %	
0.48	0.00	0.80	45.8	49.7	5.1	0.38	81.4	89.3	38.9
0.48	0.09	0.80	59.8	64.7	5.3	0.41	74.5	84.9	49.6
0.48	0.18	0.80	73.6	79.4	5.7	0.45	66.5	79.5	58.9
0.48	0.27	0.80	79.3	85.3	5.9	0.47	62.9	77.0	62.3
0.48	0.00	1.60	46.0	49.7	5.5	0.40	79.9	88.6	35.8
0.48	0.09	1.60	56.9	61.2	5.0	0.38	77.4	86.7	45.0
0.48	0.18	1.60	74.5	80.5	5.5	0.42	67.6	80.7	56.9
0.48	0.27	1.60	80.9	86.9	6.2	0.45	60.2	77.5	60.2
0.48	0.00	2.40	33.6	36.5	5.1	0.39	86.4	91.9	28.4
0.48	0.09	2.40	60.0	65.1	5.0	0.41	75.8	84.8	49.9
0.48	0.18	2.40	75.7	81.4	5.9	0.49	64.4	77.1	58.5
0.48	0.27	2.40	88.4	94.6	6.3	0.52	55.7	71.6	66.3
0.00	0.00	0.80*	32.8	35.5	5.4	0.39	85.9	92.1	27.6
0.00	0.00	1.60*	43.7	47.5	5.0	0.36	82.8	90.3	37.8
0.00	0.00	2.40*	47.5	51.5	5.2	0.36	80.3	89.4	40.9

* modifier addition made to the cell

Standard El_H = 49.5

Table 9.22 - Effect of glyoxal addition on the flotation response of 28-mesh wet-ground Upper Freeport PA coal.

REAGENT DOSAGE			PRODUCT ANALYSIS				REJECTION		EI _H
Dodecane lb/T	MIBC lb/T	Mod. lb/T	Yield %	CMR %	Ash %	Pyr. S %	Ash %	Pyr. S %	
0.23	0.00	1.00	18.4	20.7	4.6	0.36	93.5	95.9	16.0
0.23	0.05	1.00	26.9	29.4	4.6	0.44	90.4	92.7	22.0
0.23	0.14	1.00	49.6	54.3	4.3	0.45	83.2	86.2	40.5
0.23	0.23	1.00	75.9	81.3	6.2	0.59	62.2	72.4	53.7
0.23	0.00	2.00	15.9	17.4	4.5	0.32	94.3	95.9	13.3
0.23	0.05	2.00	26.3	28.8	4.2	0.40	91.3	93.5	22.3
0.23	0.14	2.00	41.2	45.0	4.4	0.36	85.7	90.9	35.8
0.23	0.23	2.00	67.0	72.3	5.7	0.43	70.3	82.2	54.5
0.23	0.00	6.00	11.3	12.3	4.4	0.38	96.1	97.4	9.7
0.23	0.05	6.00	27.8	30.4	4.2	0.44	90.9	92.5	22.9
0.23	0.14	6.00	49.2	53.4	5.0	0.52	80.5	84.2	37.6
0.23	0.23	6.00	70.4	75.6	6.1	0.49	66.3	78.7	54.3

realized with an addition of 0.8 lb/T of butyl benzaldehyde along with the standard flotation collector and frother dosages.

Flotation tests for the other two base coals and this reagent showed that the flotation products with and without butyl benzaldehyde were nearly identical to that of the standard flotation test indicating that as with the alcohols, butyl benzaldehyde effects are most pronounced for Upper Freeport coal.

9.4.3 Glyoxal

The effect of glyoxal addition to the mill on the flotation performance was evaluated using 28-mesh wet-ground Upper Freeport PA coal. Three dosages of surface modifier were tested and at each dosage flotation tests were carried out at four frother levels. The results of these test are given in Table 9.22. An examination of these results indicate that, in the presence of MIBC, there is little effect of glyoxal dosage on the efficiency index. In the absence of MIBC, on the other hand, the efficiency index varies inversely with the glyoxal dosage. The maximum value of efficiency index obtained using this reagent (EI_H = 54.5) is similar to that achieved under the standard condition (EI_H = 52.0). The results for the flotation of Illinois No. 6 coal with glyoxal, shown in Table 9.23 further show that this reagent has little beneficial effect on coal flotation.

Table 9.23 - Effect of glyoxal addition on the flotation of 28-mesh wet-ground Illinois No. 6 coal.

REAGENT DOSAGE			Yield	PRODUCT ANALYSIS			REJECTION		El _H
Dodecane	MIBC	Mod.		CMR	Ash	Pyr. S	Ash	Pyr. S	
lb/T	lb/T	lb/T	%	%	%	%	%	%	—
5.3	--	--	42.3	79.5	6.89	1.00	83.5	45.9	29.4
5.3	--	1.0	37.0	86.7	7.01	0.93	86.6	40.6	27.2
5.3	1.3	--	75.0	57.2	9.06	1.03	69.8	81.0	50.8
5.3	1.3	1.0	77.5	55.6	8.91	1.14	65.5	83.5	49.0

9.4.4 Other Reagents

A number of other organic reagents that have functional groups that could react with a particular property of the coal surface and possibly alter the flotation behavior were also examined. These included ethylene glycol, polyethylene glycol, succinic acid, pentyl ether, butylamine and dipentylamine. In general, dodecane and MIBC produced equivalent or better separations than these reagents. For instance, of the polyethylene glycol series, PEG-200 (molecular weight of 200) produced an efficiency index of 66 for Upper Freeport coal. This was only slightly better than two other PEG reagents (PEG-400, PEG-1000) but much better than either PEG-6000 or ethylene glycol which did not promote coal flotation even at high additions (up to 16 lbs/T).

Of the remaining reagents tested, both the alkylamines and the pentylether were able to produce efficiencies close to 65. However, only the pentylamine was able to produce a separation efficiency equivalent to that produced by MIBC at dosages comparable to that required for MIBC (less than 1 lb/T). The butylamine and pentylether both required 2 - 4 lb/T for equivalent separation efficiencies. Succinic acid, even at very high dosages (4 lb/T), only produced an efficiency of 56.

9.5 pH Effect

The surface of coal and pyrite are known to be modified significantly by pH changes (2,3). In the case of coal, the phenolic and the carboxylic functional groups undergo ionization ($-\text{OH} \rightarrow -\text{O}^-$; $-\text{COOH} \rightarrow -\text{COO}^-$) or protonation ($-\text{O}^- \rightarrow -\text{OH}$; $-\text{COO}^- \rightarrow -\text{COOH}$) depending on whether the pH is increased or decreased. Such changes can render the surface either relatively hydrophilic or hydrophobic. In the case of pyrite, surface reactions are more

Figure 9.24 - Solution - species diagram of aqueous iron sulfide system.

complex and depend, in addition to pH, on the type and concentration of inorganic species, especially iron, in solution and also whether or not the surface of pyrite is oxidized. A solution species diagram of aqueous iron sulfide system given in Figure 9.24 shows the type of species that will exist depending on the pH and Eh (electrode potential) of the system. Since both the type of inorganic species and its concentration in solution will depend on the type and the composition of the mineral matter in coal, the type of species that exist on the pyrite surface at a given pH can vary for different coals. Therefore, the effect of pH can be different for different coals.

In this work, a major part of the study was conducted using minus 200-mesh, wet-ground Pittsburgh No. 8 coal. Therefore, these results will be discussed first and in more detail. Also, most of the tests were carried out using caustic soda (sodium hydroxide, NaOH) as the pH modifier. The performance of caustic soda is compared with that of lime (calcium hydroxide, $\text{Ca}(\text{OH})_2$), since in addition to being a pH modifier, lime is also a known depressant for pyrite (4). Further, lime is a cheaper reagent compared to caustic soda and, therefore, commercially more attractive.

Figure 9.25 - Comparison of effects of sodium hydroxide addition to the mill and to the cell on coal flotation recovery.

The effect of the pH modifiers was tested by adding them both to the cell and to the mill. Flotation tests were carried out both in a single stage and in two stages and in the pH range of 4 to 10. This pH range was selected since the natural pH values of Pittsburgh No. 8 and Upper Freeport PA samples were around pH 4 and because below this pH value pyrite is highly soluble. The upper limit of pH 10 was selected since the alkali consumption increases steeply above this pH and will not be attractive commercially.

9.5.1 Pittsburgh No. 8 coal

The effect of pH on the recovery of coal is shown in Figure 9.25, for the addition of sodium hydroxide to the cell and to the mill. In the case of sodium hydroxide addition to the mill, up to pH 8, there is no significant effect of pH on the coal recovery. Between pH 8 and 10, the coal recovery falls sharply with pH increase, an effect that can be attributed to the ionization of surface phenolic and carboxylic groups that can render the surface hydrophilic (3). The flotation behavior can be seen to be drastically different when sodium hydroxide was added to the cell with

the recovery decreasing steeply with pH increase even in the region of pH 4 to 8. One of the reasons for these differences in flotation behavior in the pH region of 4 to 8 for the two cases is the effect of dissolved inorganic species.

The effect of interaction between dissolved inorganic species and pH on the floatability of coal has been investigated in the past (5). In that study it was shown that the effect is especially drastic in the case of trivalent ions, such as ferric iron and aluminum. However, the effect of pH was not investigated systematically, as it appears that in the past work the pH was adjusted randomly (neither in the direction of decreasing pH nor in the direction of increasing pH) and since depending on whether or not the pH is decreasing or increasing there can either be precipitation or dissolution of inorganic species which can give rise to markedly different effects. Therefore, in this study changes in the pH of the system during the course of grinding, aging, conditioning (with reagents) and flotation were monitored in order to determine the possible changes in the pH - time curve for the two cases of sodium hydroxide addition, that is, to the mill or to the flotation cell.

The variation of pH as a function of time, in the sequence of grinding, aging, conditioning and flotation, is shown in Figure 9.26 for the two modes of sodium hydroxide addition. It can be seen from this figure that when sodium hydroxide is added to the mill, the pH decreases during grinding and remains constant during aging, conditioning and flotation. On the other hand, when sodium hydroxide is added to the cell (grinding carried out in distilled water at pH ~6 instead of in alkaline solution), the pH decreases during grinding and stabilizes at the natural pH value, which was around 4 during course of these experiments. During conditioning, as the sodium hydroxide is added to the cell, the suspension follows the direction of increasing pH. Thus, it is seen that depending on whether sodium hydroxide is added to the mill or to the cell, the solids will be subjected to an entirely different pH-time cycle. The effect of such changes in pH on the precipitation/dissolution characteristics of inorganic species was studied under the same conditions used in bench scale flotation studies.

The relationship between pH and dissolved inorganic species in the supernatant of wet ground Pittsburgh No. 8 coal for sodium hydroxide addition to the mill and to the cell are shown

Figure 9.26 - Effect of mode of sodium hydroxide addition (mill or cell) on pH - time relationship.

in Figures 9.27 and 9.28 respectively. These figures show that only calcium, magnesium, iron and aluminum are present at detectable (by inductively coupled plasma spectrophotometer) levels.

A comparison of Figures 9.26 and 9.27 shows that when sodium hydroxide is added to the mill, dissolution of inorganic species will occur in the mill since pH decreases during grinding. However, since no pH changes occur during aging, conditioning or flotation, the concentration of dissolved ions will remain constant, their levels depending on the pH. The lower the pH, the higher will be the concentration of dissolved ions, especially iron. Comparison of Figures 9.26 and 9.28, indicates that for sodium hydroxide addition to the cell, dissolution of inorganic species will occur in the mill as the pH decreases during grinding; the concentration of dissolved ions will remain constant during aging. However, during conditioning, as sodium hydroxide is added to the cell, precipitation of dissolved inorganic species will occur since the concentration of dissolved species decreases with increasing pH. Precipitation of iron will occur in the range of 4 to 7, aluminum in the range of 4 to 5, and calcium and magnesium in the entire pH range of 4 to 10. When these precipitates coat the particle surface, due either to direct precipitation onto the

Figure 9.27 - Effect of pH (adjusted by adding sodium hydroxide in mill) on dissolved species concentration in the supernatant of wet-ground minus 28 mesh Pittsburgh No. 8 coal.

Figure 9.28 - Effect of pH (adjusted by adding sodium hydroxide in cell) on dissolved species concentration in the supernatant of wet-ground minus 200 mesh Pittsburgh No. 8 coal.

surface or adhesion of the bulk precipitates, they can render the surface hydrophilic. Thus, precipitation of inorganic species with increased pH and coating of the particle surface with these precipitates, appears to be the reason for the decrease in flotation recovery with increase of pH, for sodium hydroxide addition in the cell.

The above discussion shows that when sodium hydroxide is added to the cell, precipitation of inorganic species will occur, whereas such precipitation will not occur when sodium hydroxide is added in the mill. Thus, the addition of sodium hydroxide to the mill will provide non-precipitation conditions and the addition of sodium hydroxide to the cell, will provide precipitation conditions.

In order to verify the detrimental effect of precipitation of metal ion species on coal flotation, experiments were conducted in which the effect of externally added iron salt (ferrous chloride) was tested. In this case, to eliminate the interference from the dissolved inorganic species from the coal sample itself, the sample was washed several times with distilled water before grinding until all the soluble species were removed. This was checked by analyzing the conductivity and the concentration of the dissolved inorganic species in the supernatant of washed coal. Flotation tests were then conducted as a function of concentration of the dissolved iron species/ferrous chloride. The procedure followed for washed coal was similar to that employed for the unwashed coal with sodium hydroxide added to the cell. In the case of washed coal, however, the grinding was carried out in ferrous chloride solutions of varying concentrations instead of distilled water which was used in the case of unwashed coal. The effect of ferrous chloride concentration on the flotation recovery of coal at pH 8 (pH 8 was chosen for this study since precipitation of iron will be complete at this pH; refer to Figs. 9.27 and 9.28) is shown in Figure 9.29. It can be seen that increase in iron concentration results in a decrease of flotation recovery of washed coal indicating that precipitation indeed reduces the floatability of coal.

Studies conducted with washed coal showed that precipitation of inorganic species results in a decrease of flotation recovery. However, this does prove that the precipitates coat the surface of the particles and render it hydrophilic. It has been shown, both theoretically and experimentally, that hydrophilic particles can reduce the stability of froths to a considerable extent (6). Thus, the precipitates can reduce the flotation recovery also by altering the froth

Figure 9.29 - Effect of ferrous chloride addition on washed coal flotation recovery.

characteristics. To test whether the precipitates do coat the surface of the particles, electrokinetic studies were conducted, using a Penkem 501 Laser Zee meter, under both the precipitation and the non-precipitation conditions. The coal sample used for the study was prepared under the same conditions as those used in the bench scale flotation studies.

The electrokinetic behavior (zeta potential vs. pH) of both the coal (including mineral matter) particles and the precipitates is illustrated by the plots given in Figure 9.30. It can be seen from this figure that the particle zeta potential of the sample prepared under the precipitation condition is less negative than that of the sample prepared under the non-precipitation condition. It can also be seen that the precipitates are positively charged in the entire pH range. Therefore, it is clear that the uptake of precipitates would render the surface of the coal/mineral matter particles less negative. These electrokinetic results substantiate that the coal particles are indeed coated with the precipitates, and that this coating of hydrophilic particles metal hydroxides is the reason for reduction in the flotation recovery under the conditions.

Figure 9.30 - Zeta potential behaviors of coal samples prepared by grinding coal in mill and in cell, and metal-hydroxides precipitated from coal supernatant.

The effect of pH on selectivity under the precipitation (addition of sodium hydroxide to the cell) and non-precipitation (sodium hydroxide addition to the mill) conditions is illustrated in Figures 9.31 and 9.32 respectively. These figures show that under both the precipitation and non-precipitation conditions increasing the pH (up to pH 8) results in improved selectivity, but increasing the pH above 8, however, causes no further improvement in selectivity. A comparison of the selectivities obtained under the precipitation and non-precipitation conditions, at pH 8, is presented in Figure 9.33. It can be seen that a marginally lower selectivity is obtained under the precipitation condition compared to that achieved under the non-precipitation condition. This shows that the percentage decrease in the hydrophobicity of coal is marginally higher than the percentage decrease in the pyrite hydrophobicity, due to metal hydroxide precipitation. The above observation suggests the possibility that the (hydrophilic) precipitates have a marginally higher affinity for coal surface compared to that for pyrite, a hypothesis which is supported by the results of the electrokinetic study. The zeta potential-vs.-pH curves of coal, pyrite and the precipitates are plotted in Figure 9.34. It can be seen in this figure that the coal particles are

Figure 9.31 - Effect of sodium hydroxide addition in cell on pyrite rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal.

Figure 9.32 - Effect of sodium hydroxide addition in mill on pyrite rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal.

Figure 9.33 - Comparison of effects of sodium hydroxide additions in mill and cell on pyrite rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal.

Figure 9.34 - Zeta potential behaviors of coal and pyrite samples prepared by grinding the feeds in mill, and metal-hydroxides precipitated from coal supernatant.

Figure 9.35 - Solution - species diagram of aqueous iron sulfide system: location of experimental data.

more negatively charged than pyrite and that the precipitates are positively charged. Therefore, the degree of electrostatic attraction between the coal and the precipitates is likely to be higher than that between pyrite and the precipitates. Thus, the higher affinity of the precipitates to coal surface compared to that for the pyrite surface can be at least one of the reasons for the marginally lower selectivity obtained under precipitation conditions as compared to that achieved under non-precipitation conditions. As will be shown in the next section, increased mechanical carry-over (entrainment due to water flow) might also contribute to the lower pyrite rejection obtained under the former condition.

From the aforementioned results, it can be inferred that the favorable effect of increasing pH on selectivity is due only to the decrease in the hydrophobicity of pyrite since it was shown that pH has no significant effect on the flotation recovery of coal in the pH range of 4 to 8 (it is only affected due to precipitation in that range, Figure 9.25). The reason for the reduction in the hydrophobicity of pyrite can be explained on the basis of the Eh-pH diagram for the iron sulfide- H_2O system.

Figure 9.35 shows the phase diagram of the aqueous iron sulfide system. It can be seen from this diagram that at acidic pH values, in the Eh range of flotation, the surface of the pyrite particle will dissolve to ferrous iron to maintain equilibrium. This implies that as long as there is enough material, the surface will be pyrite. However, on increasing the pH, the surface will be transformed into $\text{Fe}(\text{OH})_3$ or FeS_2 . Since pyrite is partially covalently bonded, it is more hydrophobic than $\text{Fe}(\text{OH})_3$ which has no covalent bonds. Therefore, on increasing the pH from acidic to alkaline values, the surface of pyrite, depending upon the Eh value, can be transformed from partially hydrophobic FeS_2 to hydrophilic $\text{Fe}(\text{OH})_3$.

In order to determine the nature of the pyrite species that exist under the experimental conditions used in our study, the Eh of the system was measured under the flotation conditions. Two types of electrodes, namely Ag/AgCl and Pt/cal., were used in order to ensure that the results obtained are reliable. The Eh-pH curve obtained under the flotation condition is shown in Figure 9.35. These results suggested that under flotation conditions, the pyrite surface will exist as partially hydrophobic FeS_2 at lower pH values ($< \text{pH } 5$) and transform to hydrophilic $\text{Fe}(\text{OH})_3$ upon increase of pH. Thus, the observed pH effect on selectivity of pyrite removal can be explained on the basis of transformation of pyrite surface from partially hydrophobic FeS_2 to hydrophilic $\text{Fe}(\text{OH})_3$.

The effect of pH on selectivity for non-pyritic minerals rejection is shown in Figures 9.36 and 9.37 for the tests carried out under precipitation conditions (sodium hydroxide to the cell) and non-precipitation conditions (sodium hydroxide to the mill), respectively. These figures show that under non-precipitation conditions there is no significant effect of pH on selectivity. Since non-pyritic minerals are inherently hydrophilic (non-floatable), changes in surface properties of these minerals due to pH change will not affect its flotability.

Under precipitation conditions, an increase of pH up to 8 results in a decrease in flotation selectivity. This indicates that precipitation causes increased mechanical carry over of non-pyritic minerals since these are non-floatable (hydrophilic). Increased mechanical carry-over of non-pyritic minerals due to precipitation can be explained as follows: Since precipitation depresses the flotability of coal, to achieve a given coal recovery, a much higher dosage of MIBC had to

Figure 9.36 - Effect of sodium hydroxide addition in cell on the rejection of non-pyritic minerals from wet ground minus 200 mesh Pittsburgh No. 8 coal.

Figure 9.37 - Effect of sodium hydroxide addition in mill on the rejection of non-pyritic minerals from wet ground minus 200 mesh Pittsburgh No. 8 coal.

be used under precipitation conditions compared to that used under non-precipitation conditions (Figure 9.38). Since increasing the MIBC dosage increases the rate of water flow (Figure 9.39), mechanical carry over will be enhanced as a consequence. Increased mechanical carry over can also be a reason for the marginally reduced selectivity obtained for pyrite rejection under the precipitation condition compared to that achieved under the non-precipitation condition (Figure 9.33).

The effect of calcium hydroxide (lime) on pyrite and non-pyritic minerals rejection is compared with that of sodium hydroxide (caustic soda) in Figures 9.40 and 9.41 respectively. These figures show that the type of pH modifier used has no effect on the rejection of either the pyrite or the non-pyritic minerals. These results also show that the coal-pyrite depressing effect of lime is only through pH modification.

Although the effect of lime is similar to that of caustic soda in depressing pyrite, it is a much cheaper reagent. However, it was found in our tests that to achieve a comparable coal recovery, a higher dosage of frother is required when lime was used as the pH modifier compared to that consumed when caustic soda was used (Figure 9.42).

The results of an investigation of the effect of pH on the rejection of pyrite from 200-mesh dry-ground Pittsburgh No. 8 coal sample using calcium hydroxide as the pH modifier are plotted in Figure 9.43. This figure shows that although selectivities at pH 4, 6 and 10 are not significantly different, the point at pH 8 (at 65 % CMR) is clearly above the selectivity curve generated for pH 4 and towards the region of higher selectivity (upper right hand corner).

The effect of pH, modified using calcium hydroxide, on the flotation of wet and dry 28-mesh Pittsburgh No. 8 coal was tested only at the standard dosages of collector and frother. Due to the limited availability of data, selectivity curves (pyritic sulfur rejection, PSR vs. combustible material recovery, CMR) could not be drawn. Therefore, the selectivity has been expressed in terms of the Hancock Efficiency Index ($EI_H = CMR + PSR - 100$). Recall that an EI_H value of 0 represents no separation, while a value of 100 represents complete separation. When the results are expressed in terms of the Hancock efficiency index, it is better to compare the selectivity at comparable combustible material recoveries. This is because for a given reagent combination (collector and frother), the efficiency index does not vary linearly with the

Figure 9.38 - Effect of frother dosage on coal flotation recovery under precipitation (sodium hydroxide in cell) and non-precipitation conditions (sodium hydroxide in mill).

Figure 9.39 - Effect of frother dosage on total water flow out of cell.

Figure 9.40 - Comparison of effects of sodium and calcium hydroxides in mill on pyrite rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal.

Figure 9.41 - Comparison of effects of sodium and calcium hydroxides in mill on non-pyritic minerals rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal.

Figure 9.42 - Effect of frother dosage on coal flotation recovery : comparison of frother consumptions in the presence of sodium hydroxide and calcium hydroxide.

Figure 9.43 - Effect of calcium hydroxide addition in cell on pyrite rejection from dry ground minus 200 mesh Pittsburgh No. 8 coal.

combustible material recovery (Figures 8.1 and 8.2). This is because the combustible material recovery is not a linear function of the pyritic sulfur rejection.

Figures 9.44 and 9.45 show the effect of pH on combustible material recovery (CMR) and the efficiency index (El_H) obtained under the precipitation condition (calcium hydroxide in cell), for the wet and dry ground samples of minus 28 mesh Pittsburgh No. 8 coal. In the case of 28 mesh, it can be seen that even under precipitation conditions, the effect of pH on combustible material recovery is not very significant unlike that observed in the case of 200-mesh grind (compare Figure 9.25 with Figures 9.41 and 9.42). This suggests that the reduction in hydrophobicity values brought about by the precipitation of inorganic species* is not significant enough to affect the flotation recovery in the case of coarse particles. The efficiency index, on the other hand, can be seen clearly to increase with increasing pH, as was also observed with the 200-mesh grinds. Thus, these results suggest that, in the case of the coarser grind, the reduction in hydrophobicity due to precipitation of inorganic species does not significantly affect the flotation recovery, but the reduction in the hydrophobicity of pyrite is significant enough to improve the selectivity.

9.5.2 Illinois No. 6 coal

The effect of pH on the flotation of 200-mesh wet-ground Illinois No. 6 coal was tested using both calcium hydroxide and hydrochloric acid as pH modifiers since the natural pH of the suspension was not in the lower part of the tested range (6 to 10) but at about the mid-point (~8). The tests were carried out using standard dosages of the collector and the frother and adding pH modifiers to the flotation cell (pH modifier addition to the mill will represent precipitation conditions only for alkali and not for acid) and the selectivity has been expressed in terms of the Hancock Efficiency Index (El_H). It can be seen from the results, plotted in Figure 9.46, that the effect of pH on combustible material recovery is significant only at pH 10,

* It has been shown in leaching studies that the inorganic species content and composition of the 28 mesh grind supernatant of Pittsburgh No. 8 coal is similar to that of the supernatant of 200 mesh grind. Hence, precipitation is likely to occur also in the case of 28 mesh grind.

Figure 9.44 - Effect of calcium hydroxide addition in cell combustable material recovery and efficiency index for wet-ground minus 28 mesh Pittsburgh No. 8 coal.

Figure 9.45 - Effect of calcium hydroxide addition in cell on combustable material recovery and efficiency index for dry-ground minus 28 mesh Pittsburgh No. 8 coal.

Figure 9.46 - Effect of calcium hydroxide addition in cell on combustible material recovery and efficiency index for wet-ground minus 200 mesh Illinois No. 6 coal.

unlike the case for the 200-mesh grind Pittsburgh No. 8 coal in which the effect of pH, under the precipitation condition, could be seen over the entire pH range. This is due to the fact that precipitation condition is not encountered in the pH range of 6 to 8 in the case of Illinois No. 6 coal since pH was modified with an acid. The effect of precipitation of inorganic species due to pH increase, however, can be clearly seen for the results obtained between pH 8 and 10. The efficiency index passes through a maximum around pH 7.5. The steep decrease in the efficiency index between pH 8 and pH 10 is due to the sharp decrease in the combustible material recovery. As discussed in an earlier section (9.5.1), the efficiency index values cannot be compared at extremely different combustible material recovery values. Therefore, the efficiency index at pH 10 cannot be compared with that shown for the other pH values. However, the efficiency index values in the pH range of 6 to 8 can be compared since the combustible material recovery values are similar in this range. This comparison shows that the efficiency index (pyrite rejection efficiency) increases with increase in pH from 6 to 7.5, as also observed for Pittsburgh No. 8 coal.

Figure 9.47 - Effect of calcium hydroxide addition in cell on combustible material recovery and efficiency index for wet-ground minus 200 mesh Upper Freeport PA coal.

9.5.3 Upper Freeport PA coal

The effect of pH on wet-ground 200-mesh Upper Freeport PA coal, as in the case of Pittsburgh No. 8 sample, was tested using only alkali (calcium hydroxide) since the natural pH of the suspension was in the lower limit ($\sim$ pH 4) of the pH range tested. The tests were conducted using the standard dosages of collector and frother and the alkali was added in the cell (precipitation condition).

The results, plotted in Figure 9.47, show that the increase of pH under precipitation conditions does not result in any significant decrease in combustible material recovery. In fact, there is a marginal increase in combustible material recovery between pH 3.5 and 6. This results from the situation that the supernatant of the Upper Freeport PA coal sample contains no detectable levels of Al, which was found in the supernatant of Pittsburgh No. 8 and Illinois No. 6 samples, and insignificant (for the precipitation effect) levels of Fe (< 1 ppm). The amount of Ca (~ 8 ppm) and Mg (~ 1 ppm) is much below their precipitation values (Ca $\sim 40,000$ ppm; Mg $\sim 24,000$ ppm). Thus, precipitation is not likely to occur in the case of the Upper Freeport PA

coal sample. The efficiency index (pyrite rejection efficiency) increases with increase in pH up to pH 8, which was also observed with Pittsburgh No. 8 and Illinois No. 6 samples.

9.5.4 Summary

- The effect of pH on coal recovery and selectivity was tested using sodium hydroxide (caustic soda) and calcium hydroxide (lime) for Pittsburgh No. 8 sample, using calcium hydroxide and hydrochloric acid for Illinois No. 6 sample, and using only calcium hydroxide for Upper Freeport PA sample.
- In the case of wet-ground minus 200-mesh Pittsburgh No. 8 coal, the effect of pH modifier was tested by adding the reagent both to the mill and to the flotation cell. In all other cases the pH modifier effect was tested by adding the reagent only to the cell.
- In the case of Pittsburgh No. 8 and Illinois No. 6 coal samples, precipitation of dissolved cationic species occurred when alkali was added to the cell.
- Precipitation of dissolved cationic species caused depression of coal flotability in the case of fine grinds (200 mesh), due to coating of the particles by the hydrophilic precipitates; in the case of coarse grind (28 mesh), precipitation effect on coal recovery was not significant.
- For all three coals, increasing the pH (in the range of 4 to 8) caused enhanced rejection of pyrite and this was attributed to the transformation of pyrite surface from partially hydrophobic FeS_2 to hydrophilic $\text{Fe}(\text{OH})_3$.
- In the case of wet-ground minus 200-mesh Pittsburgh No. 8 coal, it was found that pH has no effect on the separation of non-pyritic minerals when alkali was added to the mill (non-precipitation conditions), whereas lower selectivity was obtained with pH increase when the alkali was added to the cell (precipitation conditions); this was attributed to the increased mechanical carry-over (entrainment due to water flow) at higher pH caused by higher MIBC addition.

9.5.5 Recommendation

The effect of pH on both the coal flotability and the separation of pyrite and non-pyritic minerals was studied in detail. The investigation showed that if an alkali is used to modify the

pH, the reagent should be added to the mill, and not directly to the flotation cell, to take advantage of its pyrite-depressing effect without detrimentally affecting the combustible material recovery and non-pyritic minerals rejection.

9.6 Dispersants

In suspensions of fine particles, aggregation due to van der Waals attraction is a common phenomenon. If aggregation occurs between coal and pyrite, selectivity will be lost during flotation even if the surface properties (hydrophobicity/hydrophilicity) of the particles are vastly different. Aggregation can be minimized by increasing the electrostatic repulsion between the particles that can be achieved by increasing the surface charge (provided all particles have the same sign of charge). One of the ways by which the surface charge can be increased is by adding ionic reagents (dispersants) to the system. In our study, two of the commonly used dispersants, namely sodium silicate and sodium hexametaphosphate were tested. The effect of sodium silicate was tested by adding the dispersant either entirely to the mill or by distributing them evenly between the mill and the cell. The effect of ultrasonication was also tested in the presence of sodium silicate. The other dispersant, sodium hexametaphosphate, was added only to the slurry in the cell.

The effect of sodium silicate on the efficiency of pyrite and non-pyritic minerals rejection from minus 200-mesh wet-ground Pittsburgh No. 8 coal is shown in Figures 9.48 and 9.49, respectively. In these figures, flotation time increases (from 0 to 5 minutes) along the curve in the direction of increasing combustible material recovery. These figures show that neither sodium silicate nor ultrasonication has a significant effect on either the rejection of pyrite or non-pyritic minerals. The addition of sodium hexametaphosphate also had no effect on the rejection of pyrite and non-pyritic minerals (Figures 9.50 and 9.51). These results show that the heterocoagulation effect is not significant. It is to be noted here that the same observation was also made in the comparative study of the effects of dry and wet grinding on flotation performance of Pittsburgh No. 8 sample (Section 7.4).

Figure 9.48 - Effect of sodium silicate addition on pyrite rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal.

Figure 9.49 - Effect of sodium silicate addition on non-pyritic minerals rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal.

Figure 9.50 - Effect of sodium hexametaphosphate addition on pyrite rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal.

Figure 9.51 - Effect of sodium hexametaphosphate addition on non-pyritic minerals rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal.

9.7 References

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10.0 PYRITE DEPRESSANTS

The flotation of pyrite, iron disulfide, commonly associated with other base metal sulfides, is often avoided in mineral processing plants by simply controlling the pulp pH. This prevents the formation of a hydrophobic surface on the pyrite when thiol-based collectors are added to the system. However, in coal flotation a high pH (>10) also results in low Btu recovery. Therefore, a reagent that adsorbs specifically on either the coal or pyrite and enhances the difference in hydrophobicity between the two would be an ideal advanced flotation reagent. This chapter discusses various attempts at decreasing the pyritic sulfur content of clean coal by the addition of reagents that would preferentially decrease the pyrite hydrophobicity. Additionally, results of reverse flotation tests (hydrophilic coal, hydrophobic pyrite) are presented and discussed.

10.1 Organic Pyrite Depressants

A number of organic reagents with different functional groups were tested to determine their pyrite depressing effect. These reagents were selected based on two important properties. First, the molecule must have a functional group that has some affinity for pyrite and second, the molecule must have such a structure that it will impart a hydrophilic character to the pyrite surface once it has adsorbed. The reagents tested were the following:

Glycerol monothioglycolate	$\text{HSCH}_2\text{COOCH}_2\text{CHOHCH}_2\text{OH}$ and $\text{HSCH}_2\text{COOCH}(\text{CH}_2\text{OH})_2$
Mercaptoacetic acid	HSCH_2COOH
2-Mercaptoethanesulfonic acid	$\text{HSCH}_2\text{CH}_2\text{SO}_3\text{H}$
Aminoacetic acid	$\text{H}_2\text{NCH}_2\text{COOH}$
Aminoethanesulfonic acid	$\text{H}_2\text{NC}_2\text{H}_4\text{SO}_3\text{H}$
Sulfosalicylic acid	$5\text{-HO}_3\text{SC}_6\text{H}_3\text{-2-(OH)COOH}$

Hallimond tube flotation tests on pyrite indicated that glycerol monothioglycolate (GMTG) is an effective pyrite depressant. However, when 1.0 lb/T of GMTG was added to the cell during flotation of the three coals, there was virtually no improvement in pyrite rejection. The apparent inability of GMTG to depress pyrite during coal flotation may have been due to the consumption of the depressant by soluble iron present in the pulp (due to oxidized pyrite)

Figure 10.1 - Flowsheet of the experiment to test GMTG as a pyrite depressant.

that has been observed after wet grinding the three coals to 200 mesh. To offset the possible consumption of GMTG by dissolved iron, which would render GMTG non-effective, a set of experiments involving two-stage grinding and flotation, with GMTG added during the grinding stage was completed. The experimental flowsheet is shown in Figure 10.1 where the dashed lines indicate optional steps. Since the natural pH of the coals was around 3.5, it was expected that a single-stage grind to minus 28 mesh would minimize the amount of dissolved iron in the pulp. The addition of GMTG to the grinding stage should further facilitate interaction between the pyrite and GMTG. The concentrate from the flotation of 28-mesh coal was reground to minus 200 mesh and floated again. The standard dosage of collector was added to the first stage of flotation only, while frother was added to both. The flotation time in the first stage was two minutes. An additional base-line flotation test was carried out with Illinois No. 6 coal ground to minus 200 mesh in one step with 1.0 lb/T GMTG.

The flotation results presented in Tables 10.1 to 10.3 show that for both the Illinois No. 6 and Pittsburgh No. 8 coals, the addition of GMTG to the grinding stage gives about a five-percent increase in pyritic sulfur rejection for the same combustible material recovery. In the

Table 10.1 - Two-stage flotation test results for Pittsburgh No. 8 coal with and without GMTG addition during grinding.

Stage	REAGENT ADDITION			CMR %	Ash Rej. %	Pyr. S Rej. %	El _H
	Collector lb/T	Frother lb/T	GMTG lb/T				
First (28 mesh)	1.92	0.30	--	95.0	39.4	29.8	24.9
Second (200 mesh)	--	0.30	--	93.9	35.7	40.8	34.7
Combined				89.2	58.5	61.0	50.2
First (28 mesh)	1.92	0.30	1.0	92.0	48.3	35.6	27.6
Second (200 mesh)	--	0.30	--	93.1	37.9	41.4	34.5
Combined				85.7	62.9	67.9	53.6

Table 10.2 - Single and two-stage flotation test results for Illinois No. 6 coal, in the presence and absence of GMTG.

Stage	REAGENT ADDITION			CMR %	Ash Rej. %	Pyr. S Rej. %	El _H
	Collector lb/T	Frother lb/T	GMTG lb/T				
First (28 mesh)	5.76	1.17	--	80.7	54.7	62.9	43.6
Second (200 mesh)	--	1.17	--	95.0	31.6	30.8	25.8
Combined				76.7	69.0	74.4	51.1
First (28 mesh)	5.76	1.17	1.0	81.8	54.8	66.0	47.7
Second (200 mesh)	--	1.17	--	95.3	34.0	34.6	29.9
Combined				77.9	70.2	77.8	55.7
Single (200 mesh)	5.76	1.17	--	83.8	51.6	54.3	35.4
Single (200 mesh)	5.76	1.17	1.0	88.2	50.0	57.8	38.2

Table 10.3 - Two-stage flotation test results for Upper Freeport PA coal with and without GMTG addition during grinding.

Stage	REAGENT ADDITION			CMR %	Ash Rej. %	Pyr. S Rej. %	El _H
	Collector lb/T	Frother lb/T	GMTG lb/T				
First (28 mesh)	0.24	0.26	--	86.0	52.7	60.3	46.3
Second (200 mesh)	--	0.26	--	88.2	42.4	62.6	50.8
Combined				75.9	72.7	85.2	61.1
First (28 mesh)	0.24	0.26	1.0	81.1	54.4	65.8	46.9
Second (200 mesh)	--	0.26	--	89.6	40.2	62.8	52.4
Combined				72.7	72.7	87.3	60.0

case of Upper Freeport PA coal, the addition of GMTG apparently results in slightly lower combustible material recovery in both stages.

Incremental flotation products were obtained during the second stage of flotation and the product analyzed. These results are plotted as CMR versus pyritic sulfur and ash rejection in Figure 10.2 for Pittsburgh No. 8. This figure clearly shows that the addition of GMTG to the grinding stage results in improved pyritic sulfur rejection at equivalent CMR. The ash rejection, however, remains essentially unchanged. These results indicate that the addition of GMTG to the mill is beneficial to flotation and results in a rejection of more pyrite than dodecane and MIBC alone.

The results for Illinois No. 6 and Upper Freeport PA coals are presented in Figures 10.3 and 10.4. Illinois No. 6 behaves analogously to Pittsburgh No. 8, with pyrite rejection increasing with GMTG addition but with the ash rejection remaining unchanged. The flotation of Upper Freeport, however, shows little change in either ash or pyritic sulfur rejection when GMTG is added. This may be due to the already high pyritic sulfur rejection obtained by two-stage flotation of Upper Freeport PA coal.

Figure 10.5 shows the effect of the other depressants on the combustible material and pyritic sulfur recovery for single-stage and two-stage flotation of Pittsburgh No. 8 coal. It is apparent from these results that all the reagents result in improved pyrite rejection, in both single-stage and two-stage flotation. However, at 1.0 lb/T, all the depressants seem to be equally effective. This probably implies that the reagent dosage was too high to differentiate between the various functional groups on the pyrite rejection characteristics. Further investigation might allow determination of the optimal reagent dosage for each reagent.

On the other hand, there is a noticeable difference in the effect of these reagents on flotation kinetics. Figure 10.6 shows the flotation kinetics of combustible material and pyrite sulfur for single-stage flotation under standard conditions, with mercaptoethanesulfonic acid and sulfosalicylic acid as depressants. While mercaptoethanesulfonic acid slows down the kinetics and ultimate recovery of pyritic sulfur without affecting the flotation of combustible material, sulfosalicylic acid affects the flotation behavior of both combustible material and pyritic sulfur.

Figure 10.2 - Effect of grinding with GMTG on combustibles matter recovery and ash and pyritic sulfur rejection in the flotation of Pittsburgh No. 8 coal.

Figure 10.3 - Effect of grinding with GMTG on combustible matter recovery and ash and pyritic sulfur rejection in the flotation of Illinois No. 6 coal.

Figure 10.4 - Effect of grinding with GMTG on combustible matter recovery and ash and pyritic sulfur rejection in the flotation of Upper Freeport PA coal.

Using the influence on flotation kinetics as the measure, it could be concluded that mercaptoethanesulfonic acid is potentially the best among the pyrite depressants studied so far.

10.2 Calcium cyanide

Pyrite, the most common sulfide mineral, is often associated with other base sulfide minerals. During flotation of these ores, two common methods used to prevent pyrite from floating in the presence of collectors are to raise the pH (>10) or to add calcium cyanide. The effect of pH was discussed extensively in Chapter 8. In order to determine the effect of cyanide on the flotation behavior of coal pyrite, an extensive experimental program was undertaken. Pittsburgh No. 8 coal, wet-ground to minus 200 mesh, was used for most of this study. Flotation tests were performed following the standard procedure except when it was necessary to compensate for any effect of the cyanide or lime addition.

The results of the first set of preliminary flotation tests with calcium cyanide and lime showed that the froth was weak and the flotation response of the coal was slowed down considerably. This effect seemed to be due to the increased pH. Therefore, a higher than

Figure 10.5 - Effect of grinding with depressants on combustible material recovery and pyritic sulfur rejection in the flotation of Pittsburgh No. 8 coal.

Figure 10.6 - Effect of grinding with pyrite depressants on the kinetics of combustible material and pyritic sulfur recovery in the flotation of Pittsburgh No. 8 coal.

standard frother dosage was used in further tests. The first set of flotation results with and without calcium cyanide are given in Table 10.4. The slurries were conditioned for two additional minutes after the calcium cyanide was added. This increase in conditioning time was done so that in those tests in which calcium cyanide was used, the coal would have time to interact with the reagent. The overall conditioning time was also increased for tests when no cyanide was added. As can be seen from the results presented in these tests, there is little difference between the results with and without calcium cyanide.

In order to complete the investigation of the effect of conditioning time, an additional set of experiments was run at constant pH. Since the efficiency index seems to level off at about pH 8.5, experiments to evaluate the effect of conditioning time with and without calcium cyanide on pyrite depression were carried out at $\text{pH } 8.5 \pm 0.3$ and the results are summarized in Table 10.5. It can be seen that with no calcium cyanide added, the CMR remains almost constant, whereas the pyritic-sulfur rejection increases from 52.8% with one additional minute of conditioning, reaching a maximum of 61.9% with two additional minutes of conditioning. A further increase in the conditioning time to four minutes resulted in a lower pyritic-sulfur rejection (57.7%). Because the CMR values are nearly identical, the EI_H values follow the same trend as the pyritic sulfur rejection values. Initially, it increases from 43.6 at one extra minute of conditioning time to 51.8 at two extra minutes of conditioning, decreasing to 47.7 at four extra minutes of conditioning. However, the differences in pyritic-sulfur content of the coal products are minor and these differences in the pyritic-sulfur rejections should be considered accordingly.

Calcium cyanide had no significant effect on the flotation of Pittsburgh No. 8. Variations in the results shown in Table 10.5 are all within the statistical variability for the comparable tests.

The final series of tests was run to evaluate the effect of calcium cyanide concentration on the flotation response of Pittsburgh No. 8 at $\text{pH } 8.5 \pm 0.5$ and to study the effect of the point of addition. The results summarized in Table 10.6 show that the CMR varied from 86.5% to 94.2% over a range of calcium cyanide dosage from 0.03 lb/T to 0.52 lb/T while the pyritic sulfur rejection varied from 47.5% to 57.2%. However, neither the CMR nor the pyritic sulfur varied systematically with cyanide addition and suggests that the observed differences are due to experimental variability rather than due to cyanide.

Table 10.4 - Effect of pH and calcium cyanide on pyrite depression of Pittsburgh No. 8 coal with 2 minutes additional conditioning time.

pH	Lime lb/T	Ca(CN) ₂ lb/T	PRODUCT ANALYSIS					REJECTION		EI _H
			Yield %	CMR %	Ash %	Tot. S %	Pyr. S %	Ash %	Pyr. S %	
7.8	9.9	--	87.9	92.9	6.5	2.7	1.4	50.6	56.8	49.7
8.4	9.7	--	85.1	90.3	6.1	2.8	1.4	45.4	59.9	50.2
8.7	12.2	--	84.7	89.9	6.1	2.7	1.3	55.3	61.9	51.8
9.3	11.4	--	79.8	84.9	5.8	2.6	1.2	59.6	66.0	50.9
6.7	8.9	0.14	86.9	91.8	6.5	2.8	1.5	51.0	56.4	48.2
8.2	10.7	0.14	86.8	92.0	6.2	2.7	1.3	53.4	61.2	53.4
8.8	9.8	0.14	85.1	90.2	6.2	2.7	1.4	54.1	59.6	49.8
9.5	11.5	0.14	79.8	84.8	6.0	2.5	1.2	58.6	66.5	51.3

Table 10.5 - Effect of conditioning time on pyrite depression for Pittsburgh No. 8 coal in the absence of calcium cyanide.

pH	Time, minutes	Lime lb/T	Ca(CN) ₂ lb/T	PRODUCT ANALYSIS					REJECTION		EI _H
				Yield %	CMR %	Ash %	Tot. S %	Pyr. S %	Ash %	Pyr. S %	
8.5	1	10.0	--	85.8	90.8	6.4	2.7	1.4	52.7	52.8	43.6
8.7	2	12.2	--	84.7	89.9	6.1	2.7	1.3	55.3	61.9	51.8
8.8	4	11.9	--	84.6	90.0	6.6	2.7	1.4	54.2	57.7	47.7
8.7	1	10.2	0.14	86.6	91.7	6.4	2.8	1.4	50.1	53.5	45.2
8.2	2	10.7	0.14	86.8	92.0	6.2	2.7	1.3	53.4	61.2	53.2
8.6	4	11.5	0.14	86.9	92.4	6.3	2.7	1.3	54.0	56.6	49.0

Table 10.6 - Flotation results for Pittsburgh No. 8 coal as a function of calcium cyanide addition at pH 8.5 ± 0.5 (reagents added to the cell).*

pH	Lime lb/T	Ca(CN) ₂ lb/T	PRODUCT ANALYSIS				REJECTION		EI _H
			Yield %	CMR %	Ash %	Pyr S %	Ash %	Pyr S %	
8.2	10.5	0.03	88.4	93.6	7.0	1.62	49.2	47.5	41.1
8.2	10.7	0.06	88.7	94.2	6.8	1.54	51.0	50.0	44.2
8.1	12.0	0.13	84.5	89.2	6.6	1.38	51.4	57.2	46.4
8.1	11.2	0.26	85.7	90.5	6.6	1.42	51.2	55.4	45.9
8.4	12.8	0.39	81.7	86.5	6.1	1.34	56.0	58.5	45.0
8.2	11.2	0.52	85.4	90.2	6.3	1.40	52.5	54.9	45.1

* 0.65 lb/T frother; 1.92 lb/T collector and two additional minutes of conditioning time.

Table 10.7 - Flotation results for Illinois No. 6 in the presence and absence of calcium cyanide at pH 7.8 ± 0.1 . Conditioning time was the standard time plus an additional 2 minutes with a collector dosage of 5.76 lb/T and frother dosage of 1.17 lb/T.

pH	Lime lb/T	Ca(CN) ₂ lb/T	PRODUCT ANALYSIS					REJECTION		EI _H
			Yield %	CMR %	Ash %	Tot S %	Pyr S %	Ash %	Pyr. S %	
6.0*	--	--	77.0	83.3	8.4	--	1.57	57.6	52.7	36.0
7.8	4.76	--	9.3	74.3	8.4	3.73	1.32	60.0	59.4	33.7
7.7	5.30	--	66.4	71.3	7.8	3.71	1.32	63.4	59.3	30.6
7.8	5.40	0.13	71.1	76.1	8.5	3.69	1.29	58.3	58.6	34.7
7.7	5.30	0.13	73.2	78.3	8.3	3.76	1.38	57.3	55.4	33.7

* Standard flotation conditions.

Previous results with Pittsburgh No. 8 coal showed that flotation improved by adding caustic to the mill before grinding, as opposed to addition of caustic directly to the flotation cell. Therefore, a final series of tests was run with Pittsburgh No. 8 coal to determine if addition of these reagents to the mill has any effect on the flotation separation. The preliminary flotation test results showed that regardless of the point of addition, cyanide had no beneficial effect on pyrite rejection during Pittsburgh No. 8 coal flotation.

Because the flotation behavior of Pittsburgh No. 8 coal is different from that of Illinois No. 6 and Upper Freeport PA coals, four tests were performed with the other two base coals using lime and calcium cyanide to determine if these reagents had any beneficial effect on pyrite rejection. For these tests the standard flotation conditions were used, except that the conditioning time was increased by two minutes after repulping and measuring the pH to allow enough time for the calcium cyanide to interact with the pyrite surface. Both reagents, lime and calcium cyanide, were added to the flotation cell, giving a pH for the flotation tests of 7.8 for Illinois No. 6 and 8.2 - 8.7 for Upper Freeport PA coal, with a calcium cyanide addition of 0.13 lb/T. The flotation results of these tests are presented in Tables 10.7 and 10.8. As with Pittsburgh No. 8 coal, there was no significant difference upon the addition of calcium cyanide on the efficiency index for the two coals, showing that calcium cyanide is not an effective pyritic sulfur depressant in coal flotation.

10.3 Polymeric xanthates

Polymeric xanthates are designed so as to take advantage of their dual function, namely,

Table 10.8 - Flotation results for Upper Freeport PA in the presence and absence of calcium cyanide at pH 8.5 ± 0.3 . Conditioning time was the standard time plus an additional 2 minutes with a collector dosage of 0.48 lb/T and frother dosage of 0.21 lb/T.

pH	Lime lb/T	Ca(CN) ₂ lb/T	PRODUCT ANALYSIS					REJECTION		EI _H
			Yield %	CMR %	Ash %	Tot S %	Pyr S %	Ash %	Pyr. S %	
3.7*	--	--	68.5	73.8	5.3	--	0.51	71.1	78.5	52.3
8.2	9.81	--	84.9	90.0	7.0	1.27	0.50	51.9	66.3	56.3
8.7	9.97	0.13	85.5	90.5	7.1	1.28	0.50	50.3	64.7	55.2
8.7	9.31	0.13	84.7	89.8	6.6	1.26	0.47	53.4	67.3	57.1

* Standard flotation conditions.

specific adsorption of the xanthate group on the surface of pyrite and the hydrophilic nature of polymeric molecules. Due to this dual nature, polymeric xanthates can adsorb specifically on the surface of pyrite and impart hydrophilicity to it. In the past, polymeric xanthates based on various monomers have been used for the depression of pyrite by different investigators but with little success (1,2). The past results showed that the polymeric reagents adsorbed not only on the pyrite surface but also on the surface of coal. The interaction of polymer with the coal surface is likely to be hydrophobic although it is not explicitly stated so in the literature. The monomer selected for our study, citric acid, was chosen so as to minimize the hydrophobic interaction of the polymer with the coal surface. Two other monomers used in the past studies, acrylic acid and sucrose, were also chosen for our study in order to compare the performance of citric acid based xanthate with those based on the latter reagents. All three xanthated reagents were synthesized using the following procedure (1):

- 1) 4.5 grams of acrylic acid (or 4.8 grams of citric acid or 4.3 grams of sucrose) was added to 25 ml of distilled water in a 150 ml cylindrical glass vessel provided with a jacket. Cold water was circulated through the jacket in order to maintain the contents at $8 \pm 1^\circ\text{C}$.
- 2) Six grams of sodium hydroxide pellets were added slowly into the vessel and the contents were stirred till the solution became clear.
- 3) 11.4 grams of carbon disulfide (CS₂) were then added to the solution in several increments, and continuously stirred between additions. The incremental dosage was chosen such that the temperature of the solution could be maintained within $\pm 1^\circ\text{C}$.
- 4) After completion of carbon disulfide addition, the solution was further stirred for 5 hours at $8 \pm 1^\circ\text{C}$.

- 5) The contents of the vessel were then transferred to a 250 ml Erlenmeyer flask and stored in the refrigerator.
- 6) Unreacted CS_2 was removed at the end of the reaction period by extracting with n-heptane and removing the organic layer using a separating funnel.

Since two of the monomers used in our study, namely citric acid and sucrose, were different from that used by Attia et al. (1) (acrylic acid), it is possible that the reaction did not proceed to completion. Therefore, the dosage of xanthated polymers has been reported in terms of the monomer content. Also, in the flotation tests the xanthated polymers were added to the cell prior to the addition of dodecane and the conditioning time with the polymer was set at five minutes. All other flotation conditions were identical to those used in the standard flotation test.

The effect of xanthated polyacrylic acid (polyacrylate-acryldithiocarbonate) and xanthated sucrose on the rejection of pyrite and non-pyritic minerals from Pittsburgh No. 8 coal (200 mesh wet ground) is shown in Figures 10.7 and 10.8, respectively. It is to be noted that, although the collector and frother dosages used in the tests conducted with the various xanthated reagents were different, the variation in the collector and frother dosages, within the range used, moves the points along the selectivity curve and not across it. In other words, the selectivity will not be affected by changes in collector and frother dosages and, therefore, comparisons made will still be valid irrespective of the collector and frother dosages. It can be seen from Figures 10.7 and 10.8 that the effect of xanthated reagents, positive or negative, on rejection of either pyrite or non-pyritic minerals is unclear. Similar observation can also be made in the case of 325-mesh wet-ground Pittsburgh No. 8 coal in which the effects of xanthated sucrose and xanthated citric acid are shown in terms of the selectivity curves for pyrite (Figure 10.9) and non-pyritic minerals (Figure 10.10).

10.4 Effect of hydrogen peroxide

In this study, hydrogen peroxide (H_2O_2) was added to the mill as an aqueous solution and its concentration was varied in the range of 0 to 3.0 weight percent*. The results, plotted in

*After the completion of grinding, the rods were taken out of the mill and a desired amount of hydrogen peroxide solution was added to the mill. The mill (without the rods) was then rolled for 30 minutes at 56 rpm (same speed used during grinding).

Figure 10.7 - Effect of xanthated polyacrylic acid and xanthated sucrose additions on pyrite rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal.

Figure 10.8 - Effect of xanthated polyacrylic acid and xanthated sucrose additions on non-pyritic minerals rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal.

Figure 10.9 - Effect of xanthated sucrose and xanthated citric acid additions on pyrite rejection from wet ground minus 325 mesh Pittsburgh No. 8 coal.

Figure 10.10 - Effect of xanthated sucrose and xanthated citric acid additions on non-pyritic minerals rejection from wet ground minus 325 mesh Pittsburgh No. 8 coal.

Figures 10.11 and 10.12, show that the addition of hydrogen peroxide has no effect on the rejection of either the pyrite or the non-pyritic minerals. It was found that although hydrogen peroxide addition reduced the floatability of pyrite, the floatability of coal was also reduced to a corresponding extent. Thus, hydrogen peroxide was found not to be a selective oxidant.

10.5 Reverse Flotation

The flotation of pyrite from coal, known as reverse flotation, is usually attempted by adding a suitable depressant to keep the coal from floating. Additionally, a suitable pyrite collector to enhance the flotation of the pyrite must be added. However, Illinois No. 6 coal is relatively hydrophilic and almost no material floats without the addition of an oily collector. Therefore, the reverse flotation of Illinois No. 6 was attempted by adding pyrite-specific reagents with the aim of producing a hydrophobic coating on the pyrite surface.

Initial flotation studies with various pyrite collectors and promoters were performed on ore pyrite samples. The objective of these studies was to determine the optimal conditions (pH, reagent addition) for pyrite flotation. Using the optimal condition for pyrite flotation, preliminary flotation tests with Illinois No. 6 were then performed using the Denver 2-liter cell with 0.57 lb/T potassium amyl xanthate (KAX). These results showed that the xanthate, a common sulfide mineral collector, also recovered substantial amounts of coal. Selective flotation of pyrite at high recovery from coal requires that the collector preferentially absorb on pyrite rather than on the coal. However, the hydrophobic chain of the collector may adsorb on the coal due to hydrophobic interaction. To reduce collector adsorption on the coal during pyrite flotation, a xanthate with a shorter hydrocarbon chain, such as potassium ethyl xanthate (KEX), was used instead of the 5-carbon amyl xanthate.

To further improve the selectivity of the flotation of pyrite from Illinois No. 6 coal, flotation tests were performed at pH 8. At this pH, the coal is expected to be slightly depressed, whereas the flotation of pyrite with xanthate shows a maxima. The results of these tests, which are presented in Table 10.9, show that at pH 8 pyrite flotation was poor; furthermore, the recovery of coal increases with increasing MIBC addition. A possible explanation for the increase in coal recovery with added frother may be that the frother was adsorbed by the coal during

Figure 10.11 - Effect of hydrogen peroxide addition on pyrite rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal.

Figure 10.12 - Effect of hydrogen peroxide addition on non-pyritic minerals rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal.

Table 10.9 - Flotation of pyrite from Illinois No. 6 coal 200 mesh grind as a function of MIBC addition in the presence of 0.57 lb/Ton potassium ethyl xanthate (KEX) at pH 8 ± 0.5 .

MIBC lb/T	Yield %	PRODUCT ANALYSIS				RECOVERY		EI _H —	Q —
		1-Y %	CMR %	Ash %	Pyr. S %	Ash %	Pyr. S %		
0.0	11.3	88.7	11.5	12.7	1.51	10.28	9.61	1.9	1.2
0.39	9.9	90.1	10.0	12.7	1.53	8.69	7.83	2.2	1.3
0.78	14.0	86.0	14.6	10.2	0.96	10.33	8.00	6.6	1.8
1.17	29.9	70.1	31.1	10.4	0.89	22.83	17.83	13.3	1.8

conditioning. In the case of pyrite, the poor flotation recovery (shown as pyritic sulfur recovery) could be related to the degree of oxidation of the pyrite surface in Illinois No. 6 coal, resulting in soluble iron species that precipitate the xanthate in solution and to the adsorption of xanthate by the coal. Consequently, a higher collector dosage was required to float the pyrite from Illinois No. 6 coal. As can be seen from the flotation results given in Table 10.9 the efficiency index is positive (for reverse flotation an EI_H of -80 represents 90-90) and Q, the ratio of the CMR to the pyritic sulfur recovery is only greater than one.

Flotation tests of pyrite from Illinois No. 6 coal were also performed at pH 4, the second pyrite flotation maximum, using the standard flotation conditions but with the addition of frother 15 seconds before flotation. The flotation results are presented in Table 10.10. As can be seen by comparing Tables 10.9 and 10.10 a reduction in pH from 8 to 4 increases both the pyritic sulfur and combustible material recovery. In general, at equal MIBC additions, the efficiency index of the flotation of pyrite from coal at pH 4 was higher indicating that the separation at

Table 10.10 - Flotation of pyrite from Illinois No. 6 coal 200 mesh grind as a function of MIBC addition in the presence of 0.57 lb/T potassium ethyl xanthate (KEX) at pH 4 ± 0.1 . Frother conditioning time: 15 seconds.

MIBC lb/T	Yield %	PRODUCT ANALYSIS				RECOVERY		EI _H —	Q —
		1-Y %	CMR %	Ash %	Pyr. S %	Ash %	Pyr. S %		
0.0	12.1	87.9	12.3	13.2	1.93	11.0	10.1	2.2	1.2
0.39	16.7	83.3	17.2	12.5	1.60	14.0	12.4	4.8	1.4
0.78	31.0	69.0	32.6	10.1	1.50	21.4	21.1	11.5	1.5
1.17	58.2	41.8	60.8	10.5	1.55	41.1	39.3	21.5	1.5

Table 10.11 - Step flotation of pyrite from Illinois No. 6 coal 200 mesh grind in the presence of potassium ethyl xanthate (KEX) and MIBC at pH 4 $\pm$ 0.1.

MIBC lb/T	KEX lb/T	FLOTATION PRODUCT ANALYSIS					RECOVERY		EI _H	Q
		Yield %	1-Y %	CMR %	Ash %	Pyr. S %	Ash %	Pyr. S %		
0.24	1.42	16.1	83.9	16.3	12.9	2.26	14.4	13.4	2.9	1.2
0.24	2.85	17.4	82.6	17.7	12.7	1.85	15.9	18.2	-0.3	1.0
0.48	2.85	22.7	77.3	23.1	12.4	2.26	19.9	28.1	-5.0	0.8
0.79	5.70	67.1	32.9	69.6	10.5	3.18	51.2	83.2	-13.6	0.8
1.04	5.70	87.8	12.2	91.8	10.3	2.82	63.6	94.9	-3.9	1.0

pH 8 is better. Although more pyrite floated at pH 4 than pH 8, the combustible material floating with the pyrite increased almost two-fold.

Based on these results, a step flotation procedure was applied for the study of the effect of KEX dosage on pyrite recovery from Illinois No. 6 coal in an effort to reduce the effects of MIBC adsorption on the coal and to help avoid the adsorption of KEX on the coal at higher concentrations. In addition, a longer conditioning time was used to ensure the adsorption of the collector on pyrite. The procedure consisted of a 2-minute repulping and conditioning time, one minute for pH adjustment and twelve minutes of conditioning with xanthate. MIBC was added 15 seconds before the two-minute flotation stage. Xanthate was added three more times with a conditioning (5 minutes each) and flotation step between each addition. The second and third flotation stages were for 2 minutes, and the final stage was for 5 minutes. The addition of the frother was distributed among the three first flotation steps.

Table 10.11 presents the composite results of the step flotation test for the removal of pyrite from minus 200-mesh Illinois No. 6 coal as a function of KEX and MIBC additions at pH 4. As was expected, the pyritic sulfur recovery increases with increasing KEX addition at constant MIBC addition. Furthermore, as the KEX addition increases from 1.42 lb/ton to 2.85 lb/ton KEX, the ratio CMR/PSR (Q) decreases, suggesting that the collector preferentially adsorbs on pyrite. Regardless, the CMR increases with increasing KEX addition, showing that Illinois No. 6 coal also adsorbs xanthate. The efficiency index of -13.6 shows that the recovery of pyrite was somewhat larger than the CMR.

Even though it was demonstrated that pyrite can be floated from coal with xanthate, the CMR also increased and selectivity was poor. As a consequence, work was performed to study

Table 10.12 - Step flotation results for pyrite flotation from Illinois No. 6 coal at 200 mesh grind in the presence of potassium ethyl xanthate (KEX), hydrogen peroxide (H_2O_2), and 0.78 lb/T of MIBC.

KEX lb/T	H_2O_2 %	FLOTATION PRODUCT ANALYSIS					RECOVERY		El_H —	Q —
		Yield %	1-Y %	CMR %	Ash %	Pyr. S %	Ash %	Pyr. S %		
0.57	--	31.0	69.0	32.6	9.3	1.29	21.4	21.1	11.5	1.5
0.57*	--	24.3	75.7	25.4	10.0	1.33	17.1	15.1	10.3	1.7
5.70	--	67.1	32.9	69.6	10.5	3.18	51.2	83.2	-13.6	0.8
5.70*	0.05	42.3	57.7	44.1	10.8	2.91	31.9	57.0	-12.9	0.8
5.70	0.10	31.3	68.7	32.0	12.2	3.52	27.3	43.3	-11.3	0.7

*sample was filtered after 3 minutes of conditioning (before xanthate addition).

the depression of coal with H_2O_2 . Also, because the stability of xanthate is reduced at pH 4 (3), other sulfhydryl collectors such as dithiophosphate reagents were also tested.

Flotation tests were performed using potassium ethyl xanthate (KEX) and Aerofloat 25 Promoter (dicresyldithiophosphoric acid and cresylic acid) as collectors and hydrogen peroxide (H_2O_2) as coal depressant. For some tests, the step flotation procedure described in the preceding paragraphs was slightly modified with an additional step that incorporated filtration of the pulp to remove ferrous (or ferric) ions before the addition of the collector after an initial conditioning time of 3 minutes. Since hydrogen peroxide is a powerful oxidant, this reagent was added to oxidize the surface of the coal and, hence, to depress it. At the same time, the addition of hydrogen peroxide may also enhance the flotability of pyrite by slightly oxidizing its surface and/or by forming dixanthogen. Therefore, the modified flotation procedure was used to establish the effect of the hydroxy complexes formed in the bulk after the addition of the hydrogen peroxide on the flotation behavior of pyrite contained in Illinois No. 6 coal.

The results of the step flotation of pyrite from Illinois No. 6 coal using KEX or Aerofloat 25 with hydrogen peroxide, and MIBC, are reported in Tables 10.12 and 10.13. These tables show that under the conditions tested, potassium ethyl xanthate is a better collector for the reverse flotation of pyrite from Illinois No. 6 coal. These tables also show that hydrogen peroxide not only suppresses the flotation of coal but pyrite as well. Therefore, hydrogen peroxide is not beneficial to the reverse flotation of coal nor does Aerofloat 25 present any advantage over KEX

Table 10.13 - Step flotation results for pyrite flotation from Illinois No. 6 coal at 200 mesh grind in the presence of Aerofloat 25 Promoter, hydrogen peroxide (H_2O_2), and MIBC.

MIBC lb/T	A25P lb/T	FLOTATION PRODUCT ANALYSIS						RECOVERY		EI_H	Q
		H_2O_2 %	Yield %	1-Y %	CMR %	Ash %	Pyr. S %	Ash %	Pyr. S %		
0.39	2.85	--	18.5	81.5	19.1	10.8	1.36	14.2	12.2	6.9	1.6
0.39	5.70	--	95.8	4.2	98.0	12.1	2.13	82.5	94.9	3.1	1.0
1.04	5.70	--	96.8	3.2	99.2	12.3	2.74	82.5	99.2	0.0	1.0
0.39	5.70	0.20	17.3	82.7	17.6	13.7	1.59	15.6	15.1	2.5	1.2
0.39	5.70	0.10	17.2	82.8	17.6	13.1	1.61	14.6	14.9	2.7	1.2

as collector for the flotation of pyrite from Illinois No. 6 coal. This study indicates that the reverse flotation of pyrite from Illinois No. 6 produces a separation that is inferior to that produced by the standard flotation test. This is probably due to poor selectivity of the pyrite collectors used.

10.6 References

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11.0 KINETICS OF COAL FLOTATION

Understanding the flotation kinetic behavior of coal, ash and pyrite is important in order to simulate and optimize flotation processes. The objective of this investigation was to study the effect of particle size on the separation efficiency and the flotation kinetics of coal as well as the associated mineral matter. For this purpose, the three base coals, namely Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA, were wet ground to 95% passing 28, 100 and 200 mesh and flotation tests were carried out in a 2-liter Denver cell at 6.25% solids. All other operating conditions in these flotation tests were the same as those for the standard flotation tests given in Chapter 7 and the pH of the pulp was the natural pH ($\text{pH } 4.5 \pm 0.5$ for Illinois No. 6 and $\text{pH } 3.5 \pm 0.3$ for the Upper Freeport PA and Pittsburgh No. 8 coals) of each coal. Unless specifically stated otherwise, the collector (dodecane) and frother (MIBC) dosages used in the flotation tests were those in Table 11.1.

In order to obtain reliable results and collect enough concentrates for the various analyses, four flotation tests were conducted on each ground sample by splitting the 500-gram sample into four parts after grinding to the desired top size. The flotation concentrate was collected at different time intervals, namely, 0.25, 0.5 and 1 minute for the 28-mesh grind, and 0.25, 0.5, 1, 2 and 5 minutes for the 100- and 200-mesh grinds. The corresponding flotation concentrates and tailings obtained from the four tests were then combined and wet sieved into different size fractions. In order to break agglomerates, the samples were washed with methanol before sieving. The flotation products of the 28-mesh grind were separated into four sizes: plus 48, 48 x 100, 100 x 200 and minus 200 mesh. The flotation products of the 100-mesh grind were sieved into three size fractions: plus 200, 200 x 400 and minus 400 mesh; and the products of the 200-mesh grind were sieved into the plus 400- and the minus 400-mesh fractions. Each size fraction was then dried, weighed and analyzed for its ash and total sulfur content using a LECO MAC-400

Table 11.1 - Reagent dosages used in the flotation kinetics tests.

Coal	Dodecane lb/T	MIBC lb/T
Illinois No. 6	4.92	0.98
Pittsburgh No. 8	1.92	0.30
Upper Freeport PA	0.24	0.26

Proximate Analyzer and a LECO SC-132 Sulfur Analyzer. The pyritic sulfur content of each size fraction was then calculated from its ash and total sulfur contents using the appropriate formula given in Chapter 6. The total sulfur content of each sample was analyzed at least twice and the average value was used in the calculation.

11.1 Flotation Kinetics Model Selection

In order to calculate the flotation kinetic parameters for coal flotation, a suitable model must be chosen before the kinetic results can be evaluated. Flotation kinetic models have been widely used to predict flotation time-recovery profiles that fit well with the experimental results. Numerous flotation kinetic models have been described in the literature (1) and the selection of an appropriate model depends not only on the overall fit of the model to the observed data but also on the statistical significance region of each model parameter. Klimpel et al.(2) have pointed out that when using mathematical models to describe flotation data, models having more than two curve-fitting parameters are simply not statistically warranted and the use of flotation models with three or more parameters can be misleading because the parameters of these models have very broad confidence regions.

In this investigation, eight flotation kinetic models with two fitting parameters were each tested. The first model is the classical first-order flotation kinetic model (3):

$$R = R_{\infty} (1 - e^{-K_1 t}) \quad (11.1)$$

The second model tested is a first-order flotation kinetic model with rectangular distribution of flotabilities (4-6):

$$R = R_{\infty} \left(1 - \frac{1 - e^{-K_2 t}}{K_2 t} \right) \quad (11.2)$$

where R = recovery of component at time t
 R_{∞} = ultimate recovery of component
 K_1 and K_2 = first-order rate constants (min^{-1}).

The third model is the first-order flotation kinetic model with a sine function (in the range of 0 to $\pi/2$) distribution of flotabilities. The reason for proposing this distribution is that in coal flotation, most of the material in the cell is the floatable component. Because the mineral matter content in a typical coal is significantly lower than the combustible material content, the population of floatable particles that are locked with a large portion of mineral matter is expected to be lower than that of those particles that are locked with small portion of mineral matter. Therefore, the distribution of rate constants (or flotabilities) of the floatable particles may be approximated more realistically by a sine function (in the range of 0 to $\pi/2$) than by a single rate constant or a rectangular distribution of flotabilities. To start, we assume that flotation follows first-order kinetics and that the initial distribution of rate constants (K) in the floatable particles in the cell is a sine distribution with a K having a minimum value of zero and a maximum value of K_3 , or

$$\varphi(K) = \frac{\pi}{2K_3} \sin\left(\frac{\pi}{2K_3}K\right) \quad (11.3a)$$

Flotation recovery as a function of flotation time t can be derived starting with the general equation:

$$R = R_{\infty} \left[1 - \int_0^{\infty} \varphi(K) e^{-Kt} dK \right] \quad (11.3b)$$

By substituting Equation 11.3a into Equation 11.3b, we get

$$R = R_{\infty} \left[1 - \int_0^{K_3} \frac{\pi}{2K_3} \sin\left(\frac{\pi}{2K_3}K\right) e^{-Kt} dK \right] \quad (11.3c)$$

Integration of Equation 11.3c yields

$$R = R_{\infty} \left[1 - \frac{1 - (2K_3t/\pi) e^{-K_3t}}{1 + (2K_3t/\pi)^2} \right] \quad (11.3d)$$

Table 11.2 - Comparison of the ultimate recovery, flotation rate constant and the standard error (SE) of estimation of combustible materials of 100-mesh Upper Freeport PA coal obtained by fitting different kinetic models (dodecane 0.24 lb/T, MTBC 0.26 lb/T and pH 3.3).

	<u>Model 1 (Eq. 11.1)</u>			<u>Model 2 (Eq. 11.2)</u>			<u>Model 3 (Eq. 11.3d)</u>		
<u>Size, mesh</u>	<u>R_∞, %</u>	<u>K₁, min⁻¹</u>	<u>SE, %</u>	<u>R_∞, %</u>	<u>K₂, min⁻¹</u>	<u>SE, %</u>	<u>R_∞, %</u>	<u>K₃, min⁻¹</u>	<u>SE, %</u>
+200	84.6	1.51	2.09	93.1	3.08	2.54	87.2	2.44	1.97
200x400	91.0	1.46	2.49	100.4	2.97	3.23	93.9	2.36	2.57
-400	91.3	1.19	1.65	101.6	2.36	2.47	94.5	1.90	1.80
Overall	88.7	1.36	2.01	98.0	2.74	2.58	91.6	2.19	1.97

Other two-parameter flotation kinetics models tested include a first-order kinetic model with triangular distribution of flotabilities (5), fully mixed reactor model (7), gas/solid adsorption kinetic model (8), second-order kinetic model (9) and second-order kinetic model with rectangular distribution of flotabilities (6).

In order to evaluate the suitability of the various models on the flotation time/recovery profiles of the three coals, the results for the flotation of the 100- and 200-mesh wet-ground coals were used. In particular, the various kinetic parameters were determined for the combustible material recovery in the plus 200, 200 x 400, and minus 400-mesh size fractions and the overall combustible material recovery in the flotation of both the 100-mesh and 200-mesh coal. This comparison of the models showed that the first three were able to predict flotation behavior quite well. The fourth did as well except that the equation used to describe the model are not valid at times equal to zero. The remaining four models all resulted in standard errors that were much higher than for the first three. Therefore, only the first three models were compared in detail.

Typical fitting results for the first three models are shown in Table 11.2, which gives the ultimate recovery, the flotation rate constant and the standard error (SE) of estimation for the combustible material of the 100-mesh Upper Freeport PA coal. It can be seen from this table that the prediction of the effect of particle size on the ultimate recovery and flotation rate constant is the same using all three equations, that is, the ultimate combustible material recovery of the plus 200-mesh fraction is lower than that for both the 200 x 400 mesh and the minus 400-mesh fractions while the flotation rate constant of the minus 400 mesh is lower than plus

Table 11.3 - Ranking of the standard error (SE) of estimation of the ultimate combustible material recovery of the three base coals obtained by fitting different flotation kinetic models.

<u>Flotation System</u>	<u>Model 1 (Eq. 11.1)</u>	<u>Model 2 (Eq. 11.2)</u>	<u>Model 3 (Eq. 11.3d)</u>
Illinois No. 6			
200 mesh Overall	3	1	2
100 mesh Overall	1*	3	2
+200 mesh	1	3	2
200 x 400 mesh	1*	3	2
-400 mesh	2*	3	1
Pittsburgh No. 8			
200 mesh Overall	1	3	2
100 mesh Overall	2*	3	1
+200 mesh	1*	3	2
200 x 400 mesh	2*	3	1
-400 mesh	2*	3	1
Upper Freeport PA		3	
200 mesh Overall	1	3	2
100 mesh Overall	2*	3	1
+200 mesh	2*	3	1
200 x 400 mesh	1*	3**	2
-400 mesh	1	3**	2

*The ultimate CMR is smaller than the CMR after 5 minutes of flotation.

**The ultimate CMR is greater than 100 percent.

200 mesh and 200 x 400 mesh fractions. However, the standard errors of estimation using model 2 (Eq. 11.2) are higher than those for model 1 and model 3, which have comparable values.

Table 11.3 further compares the three flotation models based on the standard error of estimation for the three base coals. A smaller standard error of estimation indicates a better fit of the model to the flotation results. It can be seen from this table that model 2, first-order kinetics with a rectangular distribution of flotabilities is not as good as either model 1 or 3 because it gives higher standard error and the ultimate recovery sometimes is over 100 percent. Although model 1 has a similar standard error of estimation as model 3, it slightly under estimates the recovery at long flotation time because in most of these systems the ultimate combustible material recovery is lower than the combustible material recovery at five minutes. These results indicate that the flotation of coal particles follows first-order kinetics with more particles having high flotabilities than low. The assumption that the flotability distribution is approximated by a sine function (model 3) seems to provide a better fit than that obtained by ascribing a single

Table 11.4 - Combustible material recovery (CMR), ash and pyritic sulfur rejection, and separation efficiency of Illinois No. 6 coal for 28-mesh grind (pH 5.5).

Particle Size mesh	Flotation Time, minutes	CMR %	Ash Rej., %	Pyr. S Rej., %	El _H
+48	0.25	57.0	64.2	65.2	22.2
	0.5	81.2	46.9	45.5	26.7
	1.0	88.2	42.1	41.5	29.7
48x100	0.25	68.4	50.4	53.2	21.6
	0.5	90.6	31.4	34.6	25.2
	1.0	97.3	26.2	30.6	27.9
100x200	0.25	66.8	50.3	59.8	26.6
	0.5	90.4	29.9	44.4	34.7
	1.0	97.6	23.5	39.2	36.9
-200	0.25	52.7	81.1	77.7	30.4
	0.5	78.3	71.8	65.2	43.5
	1.0	90.2	65.3	57.4	47.6
Overall	0.25	60.2	66.6	65.0	25.2
	0.5	84.1	51.7	48.1	32.2
	1.0	92.1	46.1	43.0	35.1

floatability (model 1) or particles having a rectangular distribution of floatabilities (model 2). Therefore, the first-order flotation kinetic model using a sine function (in the range of 0 to $\pi/2$) to predict the distribution of floatabilities (model 3, Eq.11.3d) was chosen to calculate the flotation rate constants and the ultimate recovery. The "best fit" values of these two parameters were determined by minimizing the standard error of estimation using a computer program.

11.2 Effect of Particle Size on the Flotation of 28-Mesh Ground Coal

Tables 11.4, 11.5, and 11.6 present the combustible material recovery (CMR), ash and pyritic sulfur rejection, and the separation efficiency of various size fractions as well as their combined values for Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA coals. It can be seen from these tables that for all three coals the intermediate-size fractions (48 x 100 and 100 x 200 mesh) exhibit a maximum in combustible material recovery whereas ash and pyritic sulfur rejection of the intermediate-size fractions is lower than that of both the larger and the smaller size fractions. However, the efficiency index for material in the smaller size fraction (minus 200 mesh) is higher than that of other size fractions for all three coals. This may result from increased liberation of pyrite in the finer size fraction as compared to the coarser-size fractions.

Table 11.5 - Combustible material recovery (CMR), ash and pyritic sulfur rejection, and separation efficiency of Pittsburgh No. 8 coal for 28-mesh grind (pH 3.3).

Particle Size mesh	Flotation Time, minutes	CMR %	Ash Rej., %	Pyr. S Rej., %	El _H
+48	0.25	72.9	51.0	43.4	16.3
	0.5	85.3	41.5	33.2	18.5
	1.0	93.3	36.6	27.4	20.8
48x100	0.25	75.2	40.7	35.9	11.1
	0.5	88.1	29.0	23.9	12.0
	1.0	96.3	23.2	17.9	14.1
100x200	0.25	74.3	41.8	38.3	12.6
	0.5	87.5	28.6	25.1	12.6
	1.0	96.5	22.3	18.5	15.0
-200	0.25	65.7	73.7	62.8	28.5
	0.5	81.5	66.9	53.5	35.0
	1.0	92.4	61.3	46.1	38.5
Overall	0.25	72.1	56.3	47.8	19.8
	0.5	85.4	47.0	37.4	22.6
	1.0	94.2	41.5	30.7	24.9

Table 11.6 - Combustible material recovery (CMR), ash and pyritic sulfur rejection, and separation efficiency of Upper Freeport PA coal for 28-mesh grind.

Particle Size mesh	Flotation Time minutes	CMR %	Ash Rej., %	Pyr. S Rej., %	El _H
+48	0.25	60.5	70.0	69.8	30.3
	0.5	69.8	64.8	65.1	34.9
	1.0	78.7	60.3	61.0	39.7
48x100	0.25	66.5	57.4	61.8	28.3
	0.5	77.7	48.8	54.7	32.4
	1.0	90.5	39.7	47.6	38.1
100x200	0.25	63.0	60.7	66.3	29.3
	0.5	76.8	50.2	57.3	34.1
	1.0	91.7	38.5	47.2	38.9
-200	0.25	43.9	80.0	80.9	24.8
	0.5	59.0	72.9	74.4	33.4
	1.0	81.3	61.0	63.9	47.2
Overall	0.25	58.6	68.7	70.9	29.5
	0.5	70.2	61.8	64.5	34.7
	1.0	83.9	53.8	56.8	40.7

Table 11.7 - Compositional analysis of the 28-mesh samples of Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA coals.

<u>Coal</u>	<u>Size, mesh</u>	<u>Weight, %</u>	<u>Ash, %</u>	<u>Tot. S, %</u>	<u>Pvt. S, %</u>
Illinois No. 6	+48	40.5	12.54	4.04	1.50
	48x100	22.9	12.37	4.28	1.72
	100x200	13.8	12.10	4.27	1.71
	-200	22.8	25.52	4.32	1.93
	Overall		15.40	4.19	1.68
Pittsburgh No. 8	+48	43.0	10.30	3.20	2.03
	48x100	21.1	9.76	3.50	2.17
	100x200	13.0	9.98	3.99	2.49
	-200	22.9	18.66	4.47	3.61
	Overall		12.06	3.66	2.48
Upper Freeport PA	+48	40.5	14.37	1.66	0.94
	48x100	23.1	11.28	1.88	1.11
	100x200	13.9	10.77	2.09	1.30
	-200	22.5	13.37	2.45	1.67
	Overall		12.93	1.95	1.19

Similarly, the overall efficiency indexes of the three coals are in the order Upper Freeport PA > Illinois No. 6 > Pittsburgh No. 8, and the liberation of pyrite in the 28-mesh ground samples is expected to be in the same order. This is in agreement with washability results, which show the same order of liberation.

Table 11.7 gives the weight fraction, ash, total sulfur and pyritic sulfur content of the different size fractions of the three minus 28-mesh coals. These results show that the size distribution of the three coals is similar and that the pyritic sulfur content increases with decreasing particle size. For Illinois No. 6 and Pittsburgh No. 8 coals, the ash content of the minus 200-mesh material is significantly higher than that of the other size fractions, whereas the variation of ash content between the various size fractions is insignificant for Upper Freeport PA coal.

Figures 11.1, 11.2 and 11.3 show the flotation rate constant and ultimate recovery of the combustible material as a function of particle size for the three base coals. The results presented in these figures show that for all three coals the intermediate size fractions (48 x 100 and 100 x 200 mesh) have higher ultimate combustible material recovery than the larger (plus 48 mesh) and the smaller (minus 200 mesh) size fractions. For Illinois No. 6 coal the flotation rate constant also exhibits a small maximum at the intermediate size, whereas for Pittsburgh No. 8 and

Figure 11.1 - Effect of particle size on the flotation rate constant and the ultimate recovery of combustible materials of 28-mesh Illinois No. 6 coal.

Figure 11.2 - Effect of particle size on the flotation rate constant and the ultimate recovery of combustible materials of 28-mesh Pittsburgh No. 8 coal.

Figure 11.3 - Effect of particle size on the flotation rate constant and the ultimate recovery of combustible materials of 28-mesh Upper Freeport PA coal.

Upper Freeport PA coals, the flotation rate constant increases monotonically with the increasing particle size. The results also show that the effect of particle size on the ultimate recovery and the flotation rate constant is dependent on the coal and reagent dosage. For the relatively hydrophobic Pittsburgh No. 8 coal with a large amount of dodecane added, the effect of particle size on the kinetic parameters is smaller than that for the more hydrophobic Upper Freeport PA coal with a small amount of dodecane added and also for the less hydrophobic Illinois No. 6 coal with larger amount of dodecane added. However, compared with mineral flotation systems, the effect of particle size on the flotation kinetics of coal appears to be insignificant due to the lower density and natural hydrophobicity of coal where oil is added as collector.

In order to compare the flotation kinetics of coal, ash and pyrite, the ultimate recovery and the flotation rate constants of the ash, pyritic sulfur and the maximum separation efficiency of the different particle size fractions were also calculated using Eq. 11.3d. These results are presented in Tables 11.8, 11.9 and 11.10 along with the combustible material recovery. It can be seen from these results, that for all three coals, the ultimate recovery of the combustible material is

Table 11.8 - Ultimate recoveries and flotation rate constants of combustible material, ash and pyritic sulfur of 28-mesh Illinois No. 6 coal (pH 5.5).

Size, mesh	Ultimate Recovery, %			Maximum <u>EI</u>	Flotation Rate Constant, min ⁻¹		
	<u>CM</u>	<u>Ash</u>	<u>Pyr S</u>		<u>CM</u>	<u>Ash</u>	<u>Pyr S</u>
+48	94.7	62.9	64.4	30.2	6.57	6.06	5.74
48x100	100.0	78.5	74.0	26.0	8.39	7.11	7.21
100x200	100.0	81.9	64.7	35.4	8.11	6.63	6.87
-200	98.9	38.9	48.7	50.3	5.30	4.47	4.19
Overall	98.3	58.3	62.0	36.3	6.71	5.99	5.96

Table 11.9 - Ultimate recoveries and flotation rate constant of combustible material, ash and pyritic sulfur of 28-mesh Pittsburgh No. 8 coal (pH 3.3).

Size, mesh	Ultimate Recovery, %			Maximum <u>EI</u>	Flotation Rate Constant, min ⁻¹		
	<u>CM</u>	<u>Ash</u>	<u>Pyr S</u>		<u>CM</u>	<u>Ash</u>	<u>Pyr S</u>
+48	94.5	64.7	73.7	20.8	10.40	10.02	10.34
48x100	97.5	78.4	83.6	13.9	10.38	10.00	10.34
100x200	97.9	79.8	83.5	14.4	9.94	9.19	9.45
-200	95.2	40.2	55.8	39.4	7.97	7.05	7.35
Overall	95.7	59.8	70.7	25.0	9.75	9.11	9.32

Table 11.10 - Ultimate recoveries and flotation rate constant of combustible material, ash and pyritic sulfur of 28-mesh Upper Freeport PA coal.

Size, mesh	Ultimate Recovery, %			Maximum <u>EI</u>	Flotation Rate Constant, min ⁻¹		
	<u>CM</u>	<u>Ash</u>	<u>Pyr S</u>		<u>CM</u>	<u>Ash</u>	<u>Pyr S</u>
+48	79.2	40.1	39.3	39.9	9.92	9.45	10.13
48x100	91.4	61.5	53.2	38.2	8.67	7.77	8.48
100x200	94.3	64.6	55.5	38.8	7.18	5.96	5.94
-200	92.4	46.2	41.5	50.9	3.88	3.35	3.67
Overall	85.7	47.6	44.6	41.2	7.48	6.91	6.78

Table 11.11 - Combustible material recovery (CMR), ash and pyritic sulfur rejection, and separation efficiency of 100-mesh Illinois No. 6 coal.

Particle Size, mesh	Flotation Time, minutes	CMR %	Ash Rej., %	Pyr. S Rej., %	El _H
+200	0.25	40.3	70.4	76.4	16.7
	0.5	67.5	49.9	61.0	28.5
	1.0	78.0	42.2	55.6	33.6
	2.0	80.6	40.2	54.5	35.1
	5.0	81.7	39.2	54.0	35.7
200x400	0.25	33.4	76.3	79.1	12.5
	0.5	59.5	58.4	64.8	24.3
	1.0	77.2	46.5	57.6	34.8
	2.0	83.8	41.4	54.8	38.6
	5.0	87.4	38.2	53.2	40.6
-400	0.25	29.1	87.7	83.7	12.8
	0.5	53.0	78.3	70.5	23.5
	1.0	72.5	69.9	59.6	32.1
	2.0	83.8	61.8	54.9	38.7
	5.0	90.0	54.2	48.2	38.2
Overall	0.25	35.1	81.2	80.1	15.2
	0.5	60.9	67.5	65.9	26.8
	1.0	76.0	58.8	57.7	33.7
	2.0	82.3	52.9	54.6	36.9
	5.0	85.8	47.7	51.2	36.9

significantly higher than that of the ash and pyritic sulfur, whereas the flotation rate constants of the combustible material are only slightly higher than that of ash and pyrite. The maximum separation efficiency is higher for the finest size fraction (minus 200 mesh) than the coarser size fractions. The closeness of the rate constants suggests that the flotation of the pyrite and ash are ultimately tied to that of the combustible material, either due to lack of liberation or entrainment and entrapment of free particles.

11.3 Effect of Particle Size in 100-mesh Ground Coal

Tables 11.11 through 11.13 present the CMR, ash and pyritic sulfur rejection, and the separation efficiency of various size fractions within the 100-mesh samples as well as their combined values at different flotation times for the three base coals. It can be seen from these tables that the combustible material recovery of all three coals increases with decreasing particle size at longer flotation times (2 and 5 minutes). However, the separation efficiency index of both Illinois No. 6 and Upper Freeport PA coals have a maximum for the 200 x 400 mesh

Table 11.12 - Combustible material recovery (CMR), ash and pyritic sulfur rejection, and separation efficiency of 100-mesh Pittsburgh No. 8 coal.

Particle Size mesh	Flotation Time, minutes	CMR %	Ash Rej., %	Pyr. S Rej., %	El _H
+200	0.25	33.4	78.0	76.5	9.9
	0.5	56.8	62.8	61.2	18.0
	1.0	69.5	55.0	53.2	22.7
	2.0	73.6	52.3	50.5	24.1
	5.0	76.1	49.8	48.5	24.6
200x400	0.25	29.3	81.1	81.6	11.9
	0.5	53.6	66.2	67.5	21.1
	1.0	71.5	55.8	58.2	29.7
	2.0	80.0	50.3	53.6	33.7
	5.0	86.2	44.8	49.2	35.4
-400	0.25	26.1	89.0	87.6	13.7
	0.5	48.6	80.4	77.6	26.2
	1.0	68.8	72.8	68.9	37.8
	2.0	81.0	67.0	62.9	43.9
	5.0	89.7	59.8	55.5	45.2
Overall	0.25	29.9	84.1	82.8	12.7
	0.5	53.2	72.3	70.2	23.4
	1.0	69.7	64.2	61.6	31.3
	2.0	77.6	59.4	56.9	34.6
	5.0	83.1	54.0	51.9	35.0

size fraction. The lower value of EI for the plus 200-mesh size fraction is a result of lower CMR's in the samples. On the other hand, the lower EI value for the minus 400-mesh size fraction of the Illinois No. 6 and Upper Freeport PA coals results from lower pyritic sulfur rejections. This may indicate that fine pyrite particles are more easily floated or carried over than are coarser pyrite particles. For Pittsburgh No. 8 coal, the value of EI increases significantly with decreasing particle size. This may indicate that the liberation of pyrite in the finer size fractions is higher than that in the coarser size fractions for Pittsburgh No. 8 coal and the pyrite is liberated at finer sizes compared to Illinois No. 6 and Upper Freeport PA coals. Since the overall efficiency indexes of the three coals are again in the order of Upper Freeport PA > Illinois No. 6 > Pittsburgh No. 8, the percentage of liberated pyrite in 100-mesh ground samples are expected to be in the same order.

Table 11.14 gives the weight, ash, total sulfur and pyritic sulfur content of each size fraction for the 100-mesh flotation feeds. These results show that the size distributions of these

Table 11.13 - Combustible material recovery (CMR), ash and pyritic sulfur rejection, and separation efficiency of 100-mesh Upper Freeport PA coal.

Particle Size, mesh	Flotation Time, minutes	CMR %	Ash Rej., %	Pyr. S Rej., %	El _H
+200	0.25	24.6	89.5	91.9	16.5
	0.5	46.8	79.8	83.8	30.6
	1.0	66.1	70.2	77.8	43.9
	2.0	78.5	62.0	72.0	50.6
	5.0	85.8	54.2	66.3	52.1
200x400	0.25	25.0	88.5	92.2	17.3
	0.5	49.4	77.0	85.1	34.4
	1.0	70.9	65.7	78.7	49.5
	2.0	84.1	56.1	72.9	57.1
	5.0	92.0	45.5	64.3	56.3
-400	0.25	21.4	90.4	92.0	13.4
	0.5	42.1	81.4	84.6	26.7
	1.0	64.3	71.7	77.3	41.6
	2.0	81.5	62.7	70.0	51.5
	5.0	91.5	52.8	60.4	51.9
Overall	0.25	23.4	89.7	92.0	15.4
	0.5	45.5	79.9	84.5	30.0
	1.0	66.5	69.9	77.7	44.2
	2.0	81.0	61.1	71.2	52.2
	5.0	89.5	51.8	62.9	52.4

three coals samples are similar and that the pyritic sulfur content increases with decreasing particle size. For Illinois No. 6 and Pittsburgh No. 8 coals, the ash content of each size fraction is relatively constant except for the minus 400-mesh fraction that contains significantly more ash. Again, for Upper Freeport PA coal, the variation of ash content in the various size fractions is insignificant.

Figures 11.4, 11.5 and 11.6 show the overall flotation recoveries of combustible material, ash and pyritic sulfur of the three base coals as a function of flotation time along with the fitting curves for Eq. 11.3d for the three base coals. Figures 11.7, 11.8 and 11.9 present the combustible material recovery of the plus 200, 200 x 400 and minus 400-mesh size fractions for the three base coals as a function of flotation time along with the fitting curves. It can be seen from these figures that the simulation curves fit the experimental data quite well for all three coals. The maximum separation efficiency, the ultimate recovery and flotation rate constant of the

Table 11.14 - Compositional analysis of the 100-mesh samples of Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA coals.

<u>Coal</u>	<u>Size, mesh</u>	<u>Weight, %</u>	<u>Ash, %</u>	<u>Tot. S, %</u>	<u>Pyr. S, %</u>
Illinois No. 6	+200	42.4	10.82	4.58	2.00
	200x400	19.6	11.61	5.27	2.68
	-400	38.0	24.76	5.24	2.82
	Overall		16.27	4.97	2.44
Pittsburgh No. 8	+200	41.3	7.98	3.27	1.86
	200x400	22.1	8.72	4.27	2.55
	-400	36.6	14.76	4.21	3.08
	Overall		10.63	3.84	2.46
Upper Freeport PA	+200	36.4	12.40	1.96	1.20
	200x400	23.1	11.92	2.67	1.86
	-400	40.5	14.16	2.71	1.92
	Overall		13.00	2.43	1.64

combustible material, ash and pyritic sulfur for the different size fractions of the three coals are summarized in Tables 11.15 through 11.17.

It can be seen from these data that for all three coals, the ultimate combustible material recovery is significantly higher than that of ash and pyritic sulfur, whereas the flotation rate constants of the three different components are similar. Again, as was observed with the 28-mesh material, ash and pyrite may be either locked with coal or carried over by coal particles during flotation. The data also show that separation efficiency is not improved by reducing the flotation time. Another important observation from these results is that the recoveries of ash and pyrite are almost equal at all flotation times for Illinois No. 6 and Pittsburgh No. 8 coals, whereas the pyritic sulfur recovery of Upper Freeport PA coal is significantly lower than its ash recovery. This

Table 11.15 - Ultimate recovery, flotation rate constant and the maximum separation efficiency of 100-mesh Illinois No. 6 coal.

<u>Size, mesh</u>	<u>Ultimate Recovery, %</u>			<u>Maximum</u>	<u>Flotation Rate Constant, min⁻¹</u>		
	<u>CM</u>	<u>Ash</u>	<u>Pyr S</u>		<u>CM</u>	<u>Ash</u>	<u>Pyr S</u>
+200	83.5	62.1	47.1	36.4	5.09	5.04	5.39
200x400	88.9	62.4	47.3	41.6	3.54	3.51	4.33
-400	91.2	45.9	51.2	40.0	2.78	1.93	2.71
Overall	86.7	51.6	48.6	38.1	3.85	3.10	3.83

Figure 11.4 - Flotation recovery of combustible material, ash and pyritic sulfur from 100-mesh Illinois No. 6 coal as a function of flotation time along with the fitting curves of the first-order kinetic model with sine distribution of flotabilities.

Figure 11.5 - Flotation recovery of combustible material, ash and pyritic sulfur of 100-mesh Pittsburgh No. 8 coal as a function of flotation time along with the fitting curves of the first-order kinetic model with sine distribution of flotabilities.

Figure 11.6 - Flotation recovery of combustible material, ash and pyritic sulfur of 100-mesh Upper Freeport PA coal as a function of flotation time along with the fitting curves of the first-order kinetic with sine distribution of flotabilities.

Figure 11.7 - Combustible material recovery of plus 200, 200x400 and minus 400 mesh fractions of 100 mesh Illinois No. 6 coal as a function of flotation time along with the fitting curves of the first-order kinetic model with sine distribution of flotabilities.

Figure 11.8 - Combustible material recovery of various size fractions of 100-mesh Pittsburgh No. 8 coal as a function of flotation time along with the fitting curves of the first-order kinetic model with sine distribution of flotabilities.

Figure 11.9 - Combustible material recovery of various size fractions of 100 mesh Upper Freeport PA coal as a function of flotation time along with the fitting curves of the first-order kinetic model with sine distribution of flotabilities.

Table 11.16 - Ultimate recovery, flotation rate constant and the maximum separation efficiency of 100-mesh Pittsburgh No. 8 coal.

Size, mesh	Ultimate Recovery, %			Maximum	Flotation Rate Constant, min ⁻¹		
	CM	Ash	Pyr S		CM	Ash	Pyr S
+200	77.1	50.3	51.8	25.3	4.25	4.26	4.43
200x400	86.8	54.7	50.5	36.4	3.05	3.01	3.24
-400	90.7	39.8	44.0	46.7	2.44	2.05	2.17
Overall	83.6	45.2	47.4	36.2	3.23	2.95	3.13

suggests that a higher percentage of liberated pyrite exists in Upper Freeport PA coal and that the hydrophobicity of these pyrite particles is lower than that of the coal. Because of the lower collector and frother dosages used in the flotation of Upper Freeport PA coal, the adsorption of dodecane on the pyrite particles is expected to be low although sulphide minerals have been found to be oleophilic (10). Therefore, the recovery of pyrite in this coal may be lower than that in Illinois No. 6 and Pittsburgh No. 8 coals because of a decreased adsorption of dodecane.

Comparing the fitting parameters of the different size fractions, it was found that for all three coals the ultimate CMR of the coarser (plus 200 mesh) size fraction is lower than that of the finer size fractions. However, the flotation rate constant is higher for the coarser (plus 200 mesh) size fraction and it decreases with decreasing particle size. This phenomenon can be explained in terms of the probability of collision, attachment and detachment between particles and bubbles. In general, coarser particles have a higher probability of collision with bubbles and a higher probability of detachment once attachment is made. Hydrophobic particles have a lower probability of detachment from the bubble than hydrophilic particles. Because hydrophobic

Table 11.17 - The ultimate recovery, flotation rate constant and the maximum separation efficiency of 100-mesh Upper Freeport PA coal.

Size, mesh	Ultimate Recovery, %			Maximum	Flotation Rate Constant, min ⁻¹		
	CM	Ash	Pyr S		CM	Ash	Pyr S
+200	87.2	46.6	33.8	53.4	2.44	1.73	1.90
200x400	93.9	55.6	36.1	57.9	2.36	1.60	1.51
-400	94.5	48.6	40.8	53.7	1.90	1.45	1.36
Overall	91.6	49.4	37.7	53.9	2.19	1.58	1.52

Table 11.18 - Combustible material recovery (CMR), ash and pyritic sulfur rejection, and separation efficiency of 200-mesh Illinois No. 6 coal.

<u>Particle Size,</u> <u>mesh</u>	<u>Flotation Time,</u> <u>minutes</u>	<u>CMR</u> <u>%</u>	<u>Ash</u> <u>Rej., %</u>	<u>Pyr. S</u> <u>Rej., %</u>	<u>EI_H</u>
+400	0.25	11.5	92.0	94.8	6.3
	0.5	22.1	85.2	90.3	12.4
	1.0	34.9	77.1	85.0	19.9
	2.0	45.1	70.5	81.3	26.4
	5.0	59.2	60.8	75.2	34.4
-400	0.25	15.6	91.9	92.6	8.2
	0.5	32.6	83.7	84.9	17.5
	1.0	53.4	73.6	75.6	29.0
	2.0	69.1	65.6	68.2	37.3
	5.0	84.7	55.0	57.1	41.7
Overall	0.25	14.1	91.9	93.2	7.3
	0.5	28.8	84.1	86.2	15.1
	1.0	46.6	74.4	73.0	24.6
	2.0	60.4	66.6	71.4	31.8
	5.0	75.4	56.3	61.6	37.0

particles in the coarser size fraction have a higher probability of collision with the bubbles and a lower probability of detachment, the coarser size fraction should exhibit a high flotation rate. Similarly, the coarser size fraction has lower ultimate CMR because the less hydrophobic particles in this size fraction have a higher probability of detachment. On the other hand, because fine particles have low probability of collision and low probability of detachment, the finer size fractions have low flotation rate and high ultimate CMR.

11.4 Effect of Particle Size in 200-mesh Ground Coal

Tables 11.18 through 11.20 present the CMR, ash and pyritic sulfur rejection, and the separation efficiency of the plus and minus 400-mesh size fractions as well as the cumulative values at different flotation times for the three base coals. It can be seen from these tables that the combustible material recovery of the minus 400-mesh fraction for all three coals is higher than that of the plus 400-mesh fraction at all flotation times, while the pyritic sulfur rejection of the minus 400-mesh fraction for all three coals is lower than that of plus 400-mesh. The corresponding separation efficiency index of minus 400-mesh fraction for both Illinois No. 6 and Pittsburgh No. 8 coals is higher than that of the plus 400-mesh fraction.

Table 11.19 - Combustible material recovery (CMR), ash and pyritic sulfur rejection, and separation efficiency of 200-mesh Pittsburgh No. 8 coal.

Particle Size, <u>mesh</u>	Flotation Time, <u>minutes</u>	CMR <u>%</u>	Ash <u>Rej., %</u>	Pyr. S <u>Rej., %</u>	El _H <u>—</u>
+400	0.25	5.6	96.2	96.6	2.2
	0.5	13.0	91.4	92.7	5.6
	1.0	27.1	82.8	85.2	12.3
	2.0	48.5	70.0	73.5	21.9
	5.0	68.0	57.8	61.8	29.7
-400	0.25	11.0	94.8	95.1	6.1
	0.5	25.1	88.7	89.3	14.5
	1.0	46.6	79.8	80.7	27.3
	2.0	70.3	70.4	70.6	40.9
	5.0	86.5	60.9	60.6	47.1
Overall	0.25	9.2	95.1	95.4	4.6
	0.5	21.0	89.3	90.1	11.1
	1.0	40.0	80.4	81.7	21.7
	2.0	62.9	70.3	71.3	34.2
	5.0	80.2	60.2	60.9	41.1

Table 11.20 - Combustible material recovery (CMR), ash and pyritic sulfur rejection, and separation efficiency of 200-mesh Upper Freeport PA coal.

Particle Size, <u>mesh</u>	Flotation Time, <u>minutes</u>	CMR <u>%</u>	Ash <u>Rej., %</u>	Pyr. S <u>Rej., %</u>	El _H <u>—</u>
+400	0.25	7.9	96.4	97.6	5.5
	0.5	17.7	92.1	94.8	12.5
	1.0	34.3	85.3	91.3	25.7
	2.0	56.9	75.5	86.6	43.5
	5.0	78.0	61.5	78.7	56.8
-400	0.25	13.0	94.1	95.6	13.4
	0.5	27.7	87.7	90.8	26.7
	1.0	49.0	78.4	84.7	41.6
	2.0	71.7	67.8	77.8	51.5
	5.0	88.1	56.3	69.4	51.9
Overall	0.25	11.7	94.6	95.9	7.6
	0.5	25.1	88.7	91.5	16.6
	1.0	45.1	80.1	85.9	31.0
	2.0	67.8	69.6	79.4	47.2
	5.0	85.5	57.5	71.1	56.6

Table 11.21 - Compositional analysis of the 200-mesh samples of Illinois No. 6 coal, Pittsburgh No. 8 and Upper Freeport PA coals.

<u>Coal</u>	<u>Size, mesh</u>	<u>Weight, %</u>	<u>Ash, %</u>	<u>Tot. S, %</u>	<u>Pyr. S, %</u>
Illinois No. 6	+400	34.4	9.06	4.07	1.48
	-400	65.6	16.53	4.83	2.32
	Overall		13.96	4.57	2.03
Pittsburgh No. 8	+400	32.7	6.74	3.14	1.66
	-400	67.3	11.93	4.19	2.80
	Overall		10.23	3.85	2.43
Upper Freeport PA	+400	26.0	11.26	1.86	1.09
	-400	74.0	12.88	2.54	1.75
	Overall		12.46	2.36	1.58

However, for Upper Freeport PA coal, the minus 400-mesh fraction has a lower EI than the plus 400-mesh material.

Table 11.21 gives the fractional weight, ash, total sulfur and pyritic sulfur content of the minus 200 mesh flotation feed for the three coals. These results show that the size distributions of the three coals are similar and that the pyritic sulfur content of minus 400 mesh fraction is higher than that of the plus 400 mesh. As with the 28- and 100-mesh grinds, the ash content of the minus 400-mesh fraction is significantly higher than that of plus 400-mesh size fraction. However, for Upper Freeport PA coal, the ash content of the two fractions is almost the same.

The overall flotation recoveries of combustible materials, ash and pyritic sulfur of the three base coals are plotted in Figures 11.10 through 11.12 as a function of flotation time along with the fitting curves. It can be seen from these figures that the behavior of the three components in the flotation of 200-mesh ground coals is similar to that of the 100-mesh ground coals. The ultimate combustible material recovery is significantly higher than that of ash and pyritic sulfur, whereas the flotation rate constants of the three different components are almost the same. Again the same conclusion may be drawn regarding the float portion of ash and pyrite. The recoveries of ash and pyrite for Illinois No. 6 and Pittsburgh No. 8 coals are very close at all flotation times, whereas the pyritic sulfur recovery of Upper Freeport PA coal is significantly lower than its ash recovery. This may be indicative of a better pyrite liberation in Upper Freeport PA coal and/or the lower hydrophobicity of pyrite particles.

Figure 11.10 - Flotation recovery of combustible material, ash and pyritic sulfur of 200-mesh Illinois No. 6 coal as a function of flotation time along with the fitting curves of the first-order kinetic model with sine distribution of flotabilities.

Figure 11.11 - Flotation recovery of combustible materials, ash and pyritic sulfur of 200-mesh Pittsburgh No. 8 coal as a function of flotation time along with the fitting curves of the first-order kinetic model with sine distribution of flotabilities.

Figure 11.12 - Flotation recovery of combustible materials, ash and pyritic sulfur of 200-mesh Upper Freeport PA coal as a function of flotation time along with the fitting curves of the first-order kinetic model with sine distribution of flotabilities.

The effect of particle size on the separation efficiency index of the 200-mesh coals can be explained in terms of the composition of the two size fractions and carry over by the froth during flotation. In general, for a given coal, high ash and pyritic sulfur contents, and better liberation in the fine size feeds can lead to increases in both the CMR and pyritic sulfur rejection and subsequently the separation efficiency index. On the other hand, fine ash and pyrite particles are easily carried over by the froth and results in a decrease in the pyritic sulfur rejection and EI. Therefore, the change in the EI indicates the relative magnitude of the two effects. For Illinois No. 6 and Pittsburgh No. 8 coals, the effect of the higher ash and pyritic sulfur, and better liberation in the finer size fraction is greater and therefore the value of EI for minus 400-mesh size fraction is higher than that of the coarser size fraction. On the other hand, for Upper Freeport PA coal, the ash content of the two size fractions is similar and the ash and pyrite in this coal appear to be liberated at coarser sizes. Therefore, the effect of carry over of fine ash and pyrite particles is larger and therefore, the EI value for the minus 400-mesh size fraction is lower than that of plus 400-mesh fraction. The overall separation efficiency for the three coals

Figure 11.13 - Combustible material recovery of the plus and minus 400-mesh size fractions of 200-mesh Illinois No. 6 coal as a function of flotation time along with the fitting curves of the first-order kinetic model with sine distribution of flotabilities.

given in Tables 11.18 through 11.20 shows that Upper Freeport PA is easier to clean (higher EI value) than Illinois No. 6 and Pittsburgh No. 8 coals.

Figures 11.13 through 11.15 present the combustible material recovery of the plus and minus 400-mesh size fractions for the three base coals as a function of flotation time along with the fitting curves and the two fitting parameters. It was found that for all three coals the ultimate CMR of the minus 400-mesh fraction is higher than that of the plus 400-mesh fraction. The flotation rate constant of the finer particles is also slightly higher than that of the coarser. Further, it can be seen from these figures that the ultimate CMR's of the minus 400-mesh size fraction of all three coals are nearly equal. Therefore, the magnitude of the ultimate CMR of the minus 400-mesh fraction does not correlate with the hydrophobicity of the coals.

Figure 11.16 shows the difference between the CMR values of the plus and minus 400-mesh size fractions as a function of flotation time for the three coals. This figure clearly shows that at flotation time longer than two minutes, the difference between the CMR of the minus and plus 400-mesh size fractions is much larger for Illinois No. 6 coal followed by

Figure 11.14 - Combustible material recovery of various size fractions of 200-mesh Pittsburgh No. 8 coal as a function of flotation time along with the fitting curves of the first-order kinetic model with sine distribution of flotabilities.

Figure 11.15 - Combustible material recovery of various size fractions of 200-mesh Upper Freeport PA coal as a function of flotation time along with the fitting curves of the first-order kinetic model with sine distribution of flotabilities.

Figure 11.16 - The difference between combustible material recovery of the plus and minus 400 mesh size fractions as a function of flotation time for the three base coals.

Pittsburgh No. 8 and Upper Freeport PA coals. This trend follows the hydrophilicity of the untreated surfaces of the three base coals. This observation, taken with the flotation behavior of the minus 400-mesh material, indicates these flotation experiments were made under collector starvation conditions in which the collector had very little chance to adsorb on the surfaces of the plus 400-mesh size fraction due to the large surface area associated with the smaller particles. Since larger particles normally need to be more hydrophobic than smaller particles in order to float, in collector starvation conditions, the plus 400-mesh size fraction (larger particles) will have a lower recovery than the minus 400-mesh material (smaller particles).

In addition, the rate constant of the fine particles will be enhanced because most of the collector will adsorb on the minus 400-mesh particles. This phenomenon is not observed in the flotation of the 100-mesh grind because the minus 400-mesh particles only represent 40 percent of the total sample weight whereas they comprise about 70 weight percent of the 200-mesh grind and these oil-coated particles tend to form agglomerates. This phenomenon is shown clearly in flotation of minus 200-mesh Pittsburgh No. 8 and Upper Freeport PA coal. Because of its higher

Figure 11.17 - The effect of reagent dosage on the difference between the combustible material recovery of the plus and minus 400 mesh size fractions as a function of flotation time for 200-mesh Illinois No. 6 coal.

hydrophilicity, the tendency for Illinois No. 6 coal to form agglomerates will be reduced and therefore the flotation rate constants are about the same for both the minus and plus 400-mesh size fractions.

In order to test this hypothesis, similar flotation experiments with minus 200-mesh coal with double the amount of collector or frother. Figures 11.17 through 11.19 show the difference between the CMR of the plus and minus 400-mesh size fractions as a function of flotation time for the double collector, double frother and standard reagent additions. The ultimate recovery and flotation rate constant of the combustible material, ash and pyritic sulfur, and the maximum efficiency index of the three base coals are summarized in Tables 11.22 through 11.30. The results in these figures and tables show clearly that the difference between the CMR of plus and minus 400-mesh fractions decreases as the reagent dosage increases confirming that the low flotation yield of the plus 400-mesh size fraction is a result of collector starvation. Another important observation from Tables 11.22 through 11.30 is that for all three coals the maximum EI decreases when double the collector or frother dosages are used due to high pyritic sulfur

Figure 11.18 - The effect of reagent dosage on the difference between the combustible material recovery of the plus and minus 400 mesh size fractions as a function of flotation time for 200-mesh Pittsburgh No. 8 coal.

Figure 11.19 - The effect of reagent dosage on the difference between combustible material recovery of the plus and minus 400 mesh size fractions as a function of flotation time for Upper Freeport PA coal.

Table 11.22 - The ultimate recovery, flotation rate constant and the maximum separation efficiency of 200-mesh Illinois No. 6 coal (Dodecane 4.92 lb/T, MIBC 0.98 lb/T).

<u>Size, mesh</u>	Ultimate Recovery, %				Maximum Flotation Rate Constant, min ⁻¹		
	<u>CM</u>	<u>Ash</u>	<u>Pyr S</u>	<u>EI</u>	<u>CM</u>	<u>Ash</u>	<u>Pyr S</u>
+400	61.2	40.4	25.3	35.9	1.36	1.36	1.45
-400	88.2	47.0	45.0	43.1	1.47	1.30	1.22
Overall	78.3	45.5	40.1	38.2	1.44	1.32	1.26

recoveries. It is expected that at higher reagent dosages pyrite will adsorb the collector, thus becoming hydrophobic.

11.5 Effect of Particle Topsize on Flotation Kinetics

In order to determine the effect of particle top size on the flotation performance of the three coals, Tables 11.31 through 11.33 were compiled in order to summarize the CMR, ash and pyritic sulfur rejection and separation efficiency at 28-, 100- and 200-mesh grinds. These results show that for all three coals the CMR decreases and the ash and pyritic sulfur rejections increase as the feed becomes finer. Because the same amount of dodecane and MIBC was used for each grind size, the collector coverage on the coal surface decreases with decreasing grind size and, therefore, the CMR decreases accordingly. On the other hand, the increase in ash and pyritic sulfur rejection with decreasing grind size is due to both a decrease in entrainment (less material

Table 11.23 - The ultimate recovery, flotation rate constant and the maximum separation efficiency of 200-mesh Illinois No. 6 coal (Dodecane 9.84 lb/T, MIBC 0.98 lb/T).

<u>Size, mesh</u>	Ultimate Recovery, %				Maximum Flotation Rate Constant, min ⁻¹		
	<u>CM</u>	<u>Ash</u>	<u>Pyr S</u>	<u>EI</u>	<u>CM</u>	<u>Ash</u>	<u>Pyr S</u>
+400	72.2	51.0	35.6	36.6	1.72	1.71	2.00
-400	93.0	48.6	53.6	39.4	1.72	1.51	1.51
Overall	85.0	49.1	49.2	35.2	1.72	1.55	1.59

Table 11.24 - The ultimate recovery, flotation rate constant and the maximum separation efficiency of 200-mesh Illinois No. 6 coal (Dodecane 4.92 lb/T, MIBC 1.95 lb/T).

Size, mesh	Ultimate Recovery, %			Maximum E_{IH}	Flotation Rate Constant, min ⁻¹		
	CM	Ash	Pyr S		CM	Ash	Pyr S
+400	83.7	61.8	40.9	42.8	1.57	1.65	1.84
-400	97.3	63.2	64.8	33.3	2.01	1.50	1.48
Overall	92.0	62.8	58.4	33.6	1.92	1.54	1.55

Table 11.25 - The ultimate recovery, flotation rate constant and the maximum separation efficiency of 200-mesh Pittsburgh No. 8 coal (Dodecane 1.92 lb/T, MIBC 0.30 lb/T and pH 3.71).

Size, mesh	Ultimate Recovery, %			Maximum E_{IH}	Flotation Rate Constant, min ⁻¹		
	CM	Ash	Pyr S		CM	Ash	Pyr S
+400	83.2	51.0	47.3	35.8	0.66	0.68	0.63
-400	95.2	43.0	44.1	51.1	1.08	1.01	0.92
Overall	90.0	45.4	44.0	46.0	0.95	0.93	0.89

Table 11.26 - The ultimate recovery, flotation rate constant and the maximum separation efficiency of 200-mesh Pittsburgh No. 8 coal (Dodecane 3.84 lb/T, MIBC 0.30 lb/T).

Size, mesh	Ultimate Recovery, %			Maximum E_{IH}	Flotation Rate Constant, min ⁻¹		
	CM	Ash	Pyr S		CM	Ash	Pyr S
+400	86.4	57.0	57.4	29.0	1.15	1.02	1.05
-400	94.1	45.2	46.2	47.9	1.56	1.24	1.21
Overall	91.7	47.4	48.1	43.6	1.44	1.19	1.17

Table 11.27 - The ultimate recovery, flotation rate constant and the maximum separation efficiency of 200-mesh Pittsburgh No. 8 coal (Dodecane 1.92 lb/T, MIBC 0.60 lb/T).

Size, mesh	Ultimate Recovery, %			Maximum E_{IH}	Flotation Rate Constant, min ⁻¹		
	CM	Ash	Pyr S		CM	Ash	Pyr S
+400	100.00	85.2	77.6	24.7	1.23	0.90	0.93
-400	100.00	57.5	61.6	40.3	1.78	1.29	1.16
Overall	100.00	62.8	64.4	36.9	1.58	1.18	1.11

Table 11.28 - The ultimate recovery, flotation rate constant and the maximum separation efficiency of 200-mesh Upper Freeport PA coal (Dodecane 0.24 lb/T, MIBC 0.26 lb/T).

<u>Size, mesh</u>	Ultimate Recovery, %			Maximum E_{IH}	Flotation Rate Constant, min ⁻¹		
	<u>CM</u>	<u>Ash</u>	<u>Pyr S</u>		<u>CM</u>	<u>Ash</u>	<u>Pyr. S</u>
+400	91.2	48.6	25.6	65.6	0.77	0.58	0.65
-400	95.5	48.5	33.5	62.0	1.16	0.94	0.98
Overall	93.8	48.0	31.9	61.9	1.06	0.86	0.94

Table 11.29 - The ultimate recovery, flotation rate constant and the maximum separation efficiency of 200-mesh Upper Freeport PA coal (Dodecane 0.48 lb/T, MIBC 0.26 lb/T).

<u>Size, mesh</u>	Ultimate Recovery, %			Maximum E_{IH}	Flotation Rate Constant, min ⁻¹		
	<u>CM</u>	<u>Ash</u>	<u>Pyr S</u>		<u>CM</u>	<u>Ash</u>	<u>Pyr. S</u>
+400	88.4	48.2	27.8	60.6	0.94	0.71	0.73
-400	94.7	50.7	37.5	57.1	1.29	1.02	0.97
Overall	92.6	49.8	35.5	57.1	1.19	0.94	0.93

Table 11.30 - The ultimate recovery, flotation rate constant and the maximum separation efficiency of 200-mesh Upper Freeport PA coal (Dodecane 0.24 lb/T, MIBC 0.52 lb/T).

<u>Size, mesh</u>	Ultimate Recovery, %			Maximum E_{IH}	Flotation Rate Constant, min ⁻¹		
	<u>CM</u>	<u>Ash</u>	<u>Pyr S</u>		<u>CM</u>	<u>Ash</u>	<u>Pyr S</u>
+400	100.0	87.0	67.4	42.6	2.13	1.20	1.00
-400	100.0	70.5	65.3	41.4	2.24	1.41	1.18
Overall	100.0	74.5	65.6	41.6	2.21	1.35	1.15

Table 11.31 - Combustible material recovery (CMR), ash and pyritic sulfur rejection, and separation efficiency of Illinois No. 6 coal for different grind sizes.

Grind Size, mesh	Flotation Time, minutes	CMR %	Ash Rej. %	Pyr. S Rej. %	El _H —
28	0.25	60.2	66.6	65.0	25.2
	0.5	84.1	51.7	48.1	32.3
	1.0	92.1	46.1	43.0	35.1
100	0.25	35.1	81.2	80.1	15.2
	0.5	60.9	67.5	65.9	26.8
	1.0	76.0	58.8	57.7	33.7
	2.0	82.3	52.9	54.6	37.0
	5.0	85.8	47.7	51.2	37.0
200	0.25	14.1	91.9	93.2	7.3
	0.5	28.8	84.1	86.2	15.1
	1.0	46.6	74.4	78.0	24.6
	2.0	60.4	66.6	71.4	31.8
	5.0	75.4	56.3	61.6	37.0

floating), lower CMR and better liberation at finer sizes. For Pittsburgh No. 8 and Upper Freeport PA coals, the decrease in CMR is smaller than the increase in pyritic sulfur rejection, which results in an increase in EI with decreasing grind size. However, for Illinois No. 6 coal, the decrease in CMR almost match the increase in pyritic sulfur rejection and therefore the change in EI is insignificant.

Table 11.32 - Combustible material recovery (CMR), ash and pyritic sulfur rejection, and separation efficiency of Pittsburgh No. 8 coal for different grind sizes.

Grind Size, mesh	Flotation Time, minutes	CMR %	Ash Rej. %	Pyr. S Rej. %	El _H —
28	0.25	72.1	56.3	47.8	19.8
	0.5	85.4	47.0	37.1	22.6
	1.0	94.2	41.5	30.7	24.9
100	0.25	30.0	84.1	82.8	12.7
	0.5	53.2	72.3	70.2	23.4
	1.0	69.7	64.2	61.6	31.3
	2.0	77.6	59.4	56.9	34.6
	5.0	83.1	54.0	51.9	35.0
200	0.25	9.2	95.1	95.4	4.6
	0.5	21.0	89.3	90.1	11.1
	1.0	40.0	80.4	81.7	21.7
	2.0	62.9	70.3	71.3	34.2
	5.0	80.2	60.2	60.9	41.1

Table 11.33 - Combustible material recovery (CMR), ash and pyritic sulfur rejection, and separation efficiency of Upper Freeport PA coal for different grind sizes.

<u>Grind Size,</u> <u>mesh</u>	<u>Flotation Time,</u> <u>minutes</u>	<u>CMR</u> <u>%</u>	<u>Ash</u> <u>Rej., %</u>	<u>Pyr. S</u> <u>Rej., %</u>	<u>El_H</u>
28	0.25	58.6	68.7	70.9	29.5
	0.5	70.2	61.8	64.5	34.7
	1.0	83.9	53.8	56.8	40.7
100	0.25	23.4	89.7	92.0	15.4
	0.5	45.5	79.9	84.5	30.0
	1.0	66.5	69.9	77.7	44.2
	2.0	81.0	61.1	71.2	52.3
	5.0	89.5	51.8	62.9	52.4
200	0.25	11.7	94.6	95.9	7.6
	0.5	25.1	88.7	91.5	16.6
	1.0	45.1	80.1	85.9	31.0
	2.0	67.8	69.6	79.4	47.2
	5.0	85.5	57.5	71.1	56.6

Figures 11.20 through 11.22 present the combustible material recovery of 28-, 100- and 200-mesh grinds for the three coals as a function of flotation time along with the fitting curves. The flotation rate constant and the ultimate recovery of combustible material, ash and pyritic sulfur, and the separation efficiency of the three coals at 28-, 100- and 200-mesh grinds are summarized in Tables 11.34 through 11.36. The results presented in these figures and tables clearly show that flotation rate constant for all three coals decreases significantly as the top size of the coal feed decreases. This agrees with the results reported in the literature (11). The lower collector coverage on the coal surface for the smaller top size (and hence decreased hydrophobicity) is considered to be the main reason for the decrease in the flotation rate constant because the effect of particle size is not as significant as it is in mineral flotation system (due to the low

Table 11.34 - Effect of grind size on the efficiency index, ultimate recovery and flotation rate constants of the combustible material, ash and pyritic sulfur of Illinois No. 6 coal.

<u>Grind size,</u> <u>mesh</u>	<u>Ultimate Recovery, %</u>			<u>Maximum</u> <u>El_H</u>	<u>Flotation Rate Constant, min⁻¹</u>		
	<u>CM</u>	<u>Ash</u>	<u>Pyr. S</u>		<u>CM</u>	<u>Ash</u>	<u>Pyr. S</u>
28	98.3	58.3	62.0	36.3	6.71	5.99	5.96
100	86.7	51.6	48.6	38.1	3.85	3.10	3.83
200	78.3	45.5	40.1	38.2	1.44	1.32	1.26

Table 11.35 - Effect of grind size on the efficiency index, ultimate recovery and flotation rate constants of the combustible material, ash and pyritic sulfur of Pittsburgh No. 8 coal.

Grind size, <u>mesh</u>	Ultimate Recovery, %			Maximum <u>El_H</u>	Flotation Rate Constant, min ⁻¹		
	<u>CMR</u>	<u>Ash</u>	<u>Pyr. S</u>		<u>CMR</u>	<u>Ash</u>	<u>Pyr. S</u>
28	95.7	59.8	70.7	25.0	9.75	9.11	9.32
100	83.6	45.2	47.4	36.2	3.23	2.95	3.13
200	90.0	45.4	44.0	46.0	0.95	0.93	0.89

specific gravity of coal) as was shown for the flotation of the 28-mesh coals. Furthermore, the flotation rate constant of the combustible materials is only slightly higher than that of ash and pyrite for all three coals, which may indicate that the floated mineral matter is either locked with coal particles and carried over by the froth during flotation.

These results also show that the ultimate recovery of pyritic sulfur decreases as the feed is ground to finer sizes, which may be a result of better liberation and less entrainment of pyrite at finer sizes. The ultimate CMR of the coals behave differently with the change in the feed size depending on the hydrophobicity of the coal and the reagent dosage. As discussed before, for the most hydrophobic coal, Upper Freeport PA, the hydrodynamics of flotation (bubble/particle interaction) are such that the flotation rate constant of small particles will be smaller but the ultimate CMR higher than that for larger particles. The flotation behavior of Upper Freeport PA exhibits this behavior in that the flotation rate constants decrease with particle size while the ultimate recovery of the CMR increases. The ultimate recovery of the ash does not change significantly while that of pyritic sulfur decreases from 47.6% at 28 mesh to 31.9% at 200 mesh.

Table 11.36 - Effect of grind size on the efficiency index, ultimate recovery and flotation rate constants of the combustible material, ash and pyritic sulfur of Upper Freeport PA coal.

Grind size, <u>mesh</u>	Ultimate Recovery, %			Maximum <u>El_H</u>	Flotation Rate Constant, min ⁻¹		
	<u>CMR</u>	<u>Ash</u>	<u>Pyr. S</u>		<u>CMR</u>	<u>Ash</u>	<u>Pyr. S</u>
28	85.7	47.6	44.5	41.2	7.48	6.91	6.78
100	91.6	49.4	37.7	53.9	2.19	1.58	1.52
200	93.8	48.0	31.9	61.9	1.06	0.86	0.94

Figure 11.20 - Combustible material recovery of 28, 100 and 200-mesh Illinois No. 6 coal as a function of flotation time along with the fitting curves of the first-order kinetic model with sine distribution of flotabilities.

Figure 11.21 - Combustible material recovery of 28, 100 and 200-mesh Pittsburgh No. 8 coal as a function of flotation time along with the fitting curves of the first-order kinetic model with sine distribution of flotabilities.

Figure 11.22 - Combustible material recovery of 28, 100 and 200-mesh Upper Freeport PA coal as a function of flotation time along with the fitting curves of the first-order kinetic model with sine distribution of flotabilities.

The decrease in particle size has a large influence on the CMR, due to a decrease on collector coverage. For a hydrophilic coal, the collector coverage is important to ensure bubble/particle attachment. This clearly does not occur under collector starvation conditions to the same extent as the more hydrophobic coals.

11.6 Flotation Kinetics of Coal at Long Flotation Times

Understanding kinetic behavior of coal flotation is important in order to simulate and optimize flotation processes. Flotation rates are determined by collecting flotation concentrates at different flotation times, and the flotation recovery - time profile is then fitted to a flotation kinetic model. For some systems, the kinetic relation obtained from short flotation times may not provide the whole picture of the flotation kinetic behavior over extended flotation times. For that reason, we conducted a kinetic study of coal flotation at long flotation times.

Table 11.37 - Collector and frother dosages used in the flotation tests.

<u>Coal</u>	<u>Dodecane, lb/T</u>	<u>MIBC, lb/T</u>
Illinois No.6	5.76	1.17
Pittsburgh No. 8	1.92	0.30
Upper Freeport PA	0.48	0.21

In this study, samples of Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA coal were wet ground in a rod mill to 95% passing 200 mesh. Flotation tests were carried out in a 2-liter Denver cell. All operating conditions in these flotation tests were identical to those of the standard flotation tests. The solids concentration in the pulp was 6.25% and flotation was carried out at the natural pH of each coal. The collector (dodecane) and frother (MIBC) dosages used in the flotation tests are given in Table 11.37. Flotation concentrates were collected at the following time intervals: 0.5, 1, 2, 4, 8, 16, 32 and 64 minutes. The concentrates and tailings were then filtered, dried and weighed. The flotation yield was calculated based on the reconstituted weight of the feed.

Table 11.38 gives the flotation yield of minus 200-mesh Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA coals after various flotation times. In order to determine the ultimate recovery and the flotation rate constant, these results were fitted with the proportionality law of flotation kinetics proposed by Dr. Ralph Lai of DOE Pittsburgh Energy Technology Center (12).

Table 11.38 - Flotation yield of minus 200-mesh Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA coals at different flotation times.

<u>Flotation Time (Minutes)</u>	<u>Illinois No. 6 % Yield</u>	<u>Pittsburgh No. 8 % Yield</u>	<u>Upper Freeport PA % Yield</u>
0.5	24.6	33.7	27.9
1	44.5	58.5	47.6
2	64.7	73.6	66.5
4	73.1	79.0	76.8
8	78.3	82.5	82.6
16	81.4	86.9	85.7
32	83.7	89.8	87.3
64	85.7	91.9	88.9

Table 11.39 - The fitting parameters of equation 11.4c for the flotation data of the three base coals.

<u>Coal</u>	<u>R_∞ %</u>	<u>K</u>	<u>R(1) %</u>	<u>SE %</u>
Illinois No.6	88.2	0.671	47.6	1.79
Pittsburgh No. 8	95.5	0.630	56.7	1.65
Upper Freeport PA	90.0	0.718	51.3	1.71

The mathematical form of this flotation kinetic law is

$$dR / dt = K(R_{\infty} - R)/t \quad (11.4a)$$

which states that the rate of recovery R of floatable material is proportional to the amount of floatable material remaining to be recovered ($R_{\infty} - R$), and is inversely proportional to flotation time t . R_{∞} is the ultimate recovery at infinite time and K is the flotation rate constant. In the integration form, the equation becomes:

$$\ln[1/(R_{\infty} - R)] = K \ln(t) + \ln(c) \quad (11.4b)$$

or
$$R = R_{\infty} - [R_{\infty} - R(1)]/t^K \quad (11.4c)$$

where c is equal to $1/[R_{\infty} - R(1)]$ and $R(1)$ is cumulative recovery after one minute of flotation. If the flotation process follows the proportionality law, a log-log plot of $1/(R_{\infty} - R)$ versus t should be a straight line with a slope of K .

Figures 11.23a, 11.24a and 11.25a show the yield as a function of flotation time on a linear-linear scale for Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA coals, along with the fitting curves of Equation 11.4c. Figures 11.23b, 11.24b and 11.25b plot $1/(R_{\infty} - R)$ versus t on a log-log scale for the three base coals. Table 11.39 summarizes the values of all fitting parameters in Equation 11.4c and the standard error of the fitting equation for these coals. These parameters were determined using a non-linear fitting computer program. It can be seen from Figures 11.23b, 11.24b and 11.25b that a log-log plot of $1/(R_{\infty} - R)$ versus t yields

a nearly straight line for all three coals at the tested conditions. The standard errors of fitting in Figures 11.23a, 11.24a and 11.25a are each within two percentage points.

11.7 References

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Figure 11.23 - Flotation results of minus 200-mesh Illinois No. 6 coal fitted with Lai's proportionality law of flotation kinetics.

Figure 11.24 - Flotation results of 200-mesh Pittsburgh No. 8 coal fitted with Lai's proportionality law of flotation kinetics.

Figure 11.25 - Flotation results of 200-mesh Upper Freeport PA coal fitted with Lai's proportionality law of flotation kinetics.

12.0 FLOTATION OPTIMIZATION AND CIRCUITRY

12.1 Optimization of Dodecane and MIBC

The standard flotation test used to evaluate the effect of various grinding environments and surface modifiers on coal flotation was not an optimized test. The standard collector and frother addition were selected such that any effect of conditioning or the reagent being investigated might have had on the flotation separation efficiency could be identified. It was felt that if the dodecane and MIBC additions were optimized then any effect, either beneficial or detrimental, could be obscured. However, since oily collectors and MIBC are the most common coal flotation reagents, the best flotation separation obtainable with only dodecane and MIBC was determined for the most hydrophilic and hydrophobic of the base coals, Illinois No. 6 and Upper Freeport PA, respectively.

12.1.1 Illinois No. 6

In order to determine the maximum pyrite rejection obtainable with only dodecane and MIBC as flotation reagents, optimization of the collector and frother dosages was undertaken as part of the research under Task 7. A two-level two-factor factorial design was used for the experiments with Illinois No. 6 coal. In this series of flotation tests, operating conditions were the same as those of the standard flotation test. In order to validate experimental results, all tests were duplicated, and the proximate analysis and total sulfur content of the flotation products from each test were determined separately. The CMR, ash rejection, pyritic sulfur rejection and Hancock efficiency index (EI_H) of each test were calculated based on the analyses of the feed and the concentrate or of the concentrate and the tailing. The final results reported here are the average of the two tests. In order to calculate flotation rate constants, the flotation concentrate was collected at regular intervals, namely, after 1, 3 and 5 minutes of flotation.

12.1.1.1 Effect of reagent dosage on the separation efficiency index

A two-level factorial design was used to determine the optimal flotation reagent dosages. Based on the reagent dosages used in the standard flotation test, 3.60 lb/T and 0.72 lb/T were chosen as the low-level dosages for dodecane and MIBC, respectively, and 9.36 lb/T and 1.89 lb/T

Table 12.1 - Combustible material recovery (CMR), ash and pyritic sulfur rejection, and separation efficiency obtained in the first step flotation optimization tests with 200-mesh wet-ground Illinois No. 6 coal.

Dodecane lb/T	MIBC lb/T	Flotation time, min.	CMR %	Ash Rej. %	Pyr. S Rej., %	El _H —
3.60	0.72	1	23.9	89.3	91.1	15.0
		3	49.5	77.4	80.8	30.3
		5	56.2	73.9	77.5	33.8
9.36	0.72	1	51.3	76.4	78.6	29.8
		3	73.2	65.1	67.9	41.1
		5	76.7	62.9	65.8	42.5
5.76	1.17	1	53.0	73.5	75.2	28.2
		3	79.1	59.3	61.7	40.8
		5	83.6	56.1	58.4	42.0
3.60	1.89	1	52.8	72.4	75.8	28.6
		3	82.0	55.9	60.5	42.5
		5	87.3	51.7	56.5	43.8
9.36	1.89	1	68.0	64.8	66.9	35.0
		3	88.6	51.8	54.8	43.4
		5	91.5	49.1	52.0	43.5

as the high-level dosages. Table 12.1 shows the combustible material recovery, ash rejection, pyritic sulfur rejection and the El_H, all of which were obtained from the first stage of these optimization tests. For comparison, the results obtained with the standard flotation dosage are also given in this table. As expected, the results in this table show that the CMR increases with an increase in either the dodecane or MIBC dosage, whereas ash rejection and pyritic sulfur rejection both decrease. The efficiency index, which is used as the optimization criterion, however, does not change significantly when either reagent is added at the high level. Based on the efficiency index, three minutes of flotation results in a product with an efficiency index nearly equivalent to that after five minutes of flotation.

Table 12.2 summarizes the effect of dodecane and MIBC dosages on El_H in the first step optimization tests. These values were calculated following standard statistical analysis procedures. It can be seen from this table that an increase in both dodecane and MIBC dosages increases the efficiency index for all three flotation times. However, the amount by which it increases is found to decrease with flotation time. The negative values of the combined effect of MIBC and dodecane indicate that the effect of the dosage of the two reagents on El_H is not additive in the

Table 12.2 - Effect of collector (dodecane) and frother dosage (MIBC) on the separation efficiency index of Illinois No. 6 in the first step optimization tests.

Factor	FLOTATION TIME		
	1 min.	3 min.	5 min.
Average EI_H	27.1	39.3	40.9
Dodecane (3.6 to 9.36 lb/T)	10.6	5.9	4.2
MIBC (0.72 to 1.88 lb/T)	9.4	7.2	5.5
Dodecane x MIBC	-4.2	-4.9	-4.4

range studied. Since results of QA/QC tests have shown that a two-point increase in EI_H is significant, the results in this table confirm the improvement in the separation efficiency obtained with the high-level dosages.

Since the results in Table 12.1 show that EI_H decreases when both dodecane and MIBC were added at low dosage, and it does not change much when one reagent (dodecane or MIBC) is added at the high level, the optimal dosages of dodecane and MIBC should be between the low and high dosages. Therefore, in the second series of optimization tests, an optimization factor 0.618 was chosen to determine the reagent dosage level, that is, 4.92 lb/T and 0.98 lb/T as the low-level dosages of dodecane and MIBC, respectively; and 7.20 lb/T and 1.43 lb/T as the high-level dosages. Table 12.3 presents the CMR, ash rejection, pyritic sulfur rejection, and EI_H which were obtained from the second set of optimization tests. As in the first set of optimization tests, the CMR increases while the ash and pyritic sulfur rejection both decrease with increasing reagent dosage and flotation time.

Table 12.4 summarizes the statistical analysis of the effect of dodecane and MIBC on the EI_H in this series of tests. Although the results show that the reagent dosage increases the separation efficiency obtained for all three flotation times, the improvement is not significant (the increase in EI_H is less than two points) when the flotation time is longer than three minutes. By considering both the reagent consumption and the magnitude of the increase in EI_H , we chose 4.92 lb/T of dodecane and 0.98 lb/T of MIBC as the optimal flotation reagent dosages. It can be seen from Table 12.4 that the values of EI_H obtained with these optimal dosages at the three different flotation times are the same as that obtained with the standard flotation dosages.

Table 12.3 - Combustible material recovery (CMR), ash and pyritic sulfur rejection, and separation efficiency obtained in the second step flotation optimization tests with 200-mesh wet-ground Illinois No. 6 coal.

Dodecane lb/T	MIBC lb/T	Flotation time, min.	CMR %	Ash Rej. %	Pyr. S Rej. %	El _H
4.92	0.98	1	52.1	74.1	76.7	28.8
		3	80.5	58.1	61.3	41.9
		5	85.7	53.9	57.1	42.7
7.20	0.98	1	59.4	69.7	71.6	31.1
		3	82.7	56.6	58.9	41.6
		5	86.9	53.3	55.6	42.5
4.92	1.43	1	54.8	71.0	74.6	29.4
		3	84.7	53.2	57.8	42.4
		5	89.7	48.4	53.3	43.1
7.20	1.43	1	62.9	68.8	71.7	34.5
		3	87.3	54.1	57.1	44.4
		5	90.8	50.5	53.5	44.3

12.1.1.2 Effect of Reagent Dosage on Flotation Kinetics

In order to determine the effect of dodecane and MIBC on flotation kinetics, the flotation recovery as a function of time was fitted with a first-order flotation kinetic model with a sine function (in the range of 0 to $\pi/2$) distribution of flotabilities (Equation 11.3). Table 12.5 gives the flotation rate constants and ultimate recoveries of combustible materials, ash and pyritic sulfur of Illinois No. 6 obtained from the second step optimization tests. As can be seen from the

Table 12.4 - Effect of collector (dodecane) and frother dosage (MIBC) on the separation efficiency index obtained in the second step optimization tests with Illinois No. 6 coal.

Factor	FLOTATION TIME		
	1 min.	3 min.	5 min.
Average El _H	30.9	42.6	43.1
Dodecane (4.92 to 7.2 lb/T)	3.7	0.9	0.5
MIBC (0.98 to 1.43 lb/T)	2.0	1.7	1.1
Dodecane x MIBC	1.4	1.2	0.8
Standard Flotation El _H (dodecane 5.76 lb/T, MIBC 1.17 lb/T)	28.2	40.8	42.0
Optimal flotation El _H (dodecane 4.92 lb/T, MIBC 0.98 lb/T)	28.8	41.8	42.7

Table 12.5 - Flotation rate constant and ultimate recoveries of combustible materials, ash and pyritic sulfur of 200-mesh wet-ground Illinois No. 6 coal obtained from the second set of optimization tests.

Dodecane lb/T	MIBC lb/T	Rate Constant, min ⁻¹			Ultimate Recovery, %		
		CMR	Ash	Pyr. S	CMR	Ash	Pyr. S
4.92	0.98	1.47	1.26	1.18	89.9	48.9	45.9
7.20	0.98	1.89	1.69	1.64	89.2	48.0	45.7
4.92	1.43	1.48	1.26	1.18	94.2	54.7	50.0
7.20	1.43	1.94	1.59	1.48	93.4	51.2	48.4

results given in Table 12.5 the flotation rate constant and ultimate recovery of combustible materials are always higher than those of either the ash or pyritic sulfur (which are almost the same in all the tests). Table 12.6 shows the effect of dodecane and MIBC dosage on the flotation rate constant and the ultimate recovery calculated from the statistical analysis of the results from the second step of the optimization tests, along with the flotation rate constants and ultimate flotation recovery obtained from the standard flotation and optimal flotation reagent dosages. These results indicate that the dodecane dosage affects the flotation rate constants much more than does MIBC; whereas the MIBC dosage affects the ultimate recovery more significantly than does dodecane. The same conclusion can be drawn from the results of the first set of optimization tests.

Table 12.6 - Statistical analysis of the effect of collector (dodecane) and frother dosage (MIBC) on the flotation rate constant and ultimate recovery of combustible materials, ash and pyritic sulfur of Illinois No. 6 coal in the second set of optimization tests.

Factor	Rate Constant, min ⁻¹			Ultimate recovery, %		
	CMR	Ash	Pyr. S	CMR	Ash	Pyr. S
Average value	1.70	1.45	1.37	91.7	50.7	47.5
Dodecane (4.92 to 7.2 lb/T)	0.44	0.38	0.38	-0.3	-2.2	-0.9
MIBC (0.98 to 1.43 lb/T)	0.03	-0.05	-0.08	4.3	4.5	3.4
Dodecane x MIBC	0.02	-0.05	-0.08	-0.1	-1.3	-0.7
Standard flotation value (dodecane 5.76, MIBC 1.17 lb/T)	1.60	1.45	1.41	87.0	45.9	43.5
Optimal flotation value (dodecane 4.92, MIBC 0.98 lb/T)	1.47	1.26	1.18	89.9	48.9	45.9

Table 12.7 - Flotation reagent optimization results for 200-mesh wet-ground Upper Freeport PA coal.

<u>REAGENT DOSAGE</u>		<u>PRODUCT ANALYSIS</u>				<u>REJECTION</u>		<u>El_H</u>
Dodecane	MIBC	Yield	CMR	Ash	Pyr. S	Ash	Pyr. S	
<u>lb/T</u>	<u>lb/T</u>	<u>%</u>	<u>%</u>	<u>%</u>	<u>%</u>	<u>%</u>	<u>%</u>	
<u>First-Stage Optimization</u>								
0.33	0.14	34.7	37.6	4.93	0.35	86.0	92.2	29.8
0.86	0.14	56.0	60.4	5.32	0.42	75.7	86.9	45.3
0.33	0.37	78.0	83.9	5.62	0.40	64.0	80.0	63.9
0.86	0.37	85.6	91.0	6.65	0.50	53.5	72.6	63.6
<u>Second-Stage Optimization</u>								
1.39	0.23	76.7	82.5	6.37	0.48	62.2	76.4	58.9
1.39	0.60	88.5	93.8	7.16	0.52	49.1	70.5	64.3
0.53	0.60	91.4	96.1	7.19	0.59	41.7	65.4	61.5
0.20	0.60	89.6	94.7	7.42	0.52	46.5	70.1	64.8

12.1.2 Upper Freeport PA

The optimization of reagent dosage for Upper Freeport PA coal was also accomplished using a two-factor, two-level factorial design. The results of the first step are given in Table 12.7. These results indicate that with sufficient frother addition, an increase in collector does not increase the separation efficiency. Results from the second step further confirm that the addition of more collector has detrimental effects on the flotation separation. These results show that the separation efficiency is most sensitive to the frother dosage. Therefore, the final optimization step was run with no collector and a high dosage of frother (1.0 lb/T MIBC). These results are reported in Table 12.8 and represent the optimized reagent conditions for 200-mesh Upper Freeport PA coal.

Optimization tests were also conducted for wet-ground 100- and 325-mesh Upper Freeport PA coal. The test results are shown in Tables 12.9 and 12.10. It can be seen from Table 12.9

Table 12.8 - Optimization of standard flotation test results for 200-mesh wet-ground Upper Freeport PA coal.

REAGENT DOSAGE			PRODUCT ANALYSIS				REJECTION		El _H
Coll. lb/T	Frot. lb/T	Mod. lb/T	Yield %	CMR %	Ash %	Pyr. S %	Ash %	Pyr. S %	
0	1.0	-	87.0	92.4	6.91	0.48	51.3	73.2	65.6
0	1.0	-	90.2	95.2	7.42	0.54	45.5	68.8	64.0

Table 12.9 - Optimization flotation test results for 100-mesh wet-ground Upper Freeport PA coal.

REAGENT DOSAGE			PRODUCT ANALYSIS				REJECTION		
Coll.	Frot.	Mod.	Yield	CMR	Ash	Pyr. S	Ash	Pyr. S	El _H
lb/T	lb/T	lb/T	%	%	%	%	%	%	
0.26	0.70	--	88.7	93.7	7.15	0.50	47.7	71.6	65.3
0.52	0.70	--	91.0	96.3	7.71	0.57	40.9	66.8	62.1
0.84	0.70	--	91.6	96.0	8.21	0.66	39.5	61.2	57.2
0.84	0.43	--	87.6	92.4	7.28	0.56	47.4	68.6	61.0

that an optimized separation efficiency of 65.3 was obtained for 100-mesh feed when the collector (dodecane) dosage was 0.26 lb/T and the frother (MIBC) was 0.70 lb/T. The optimized separation efficiency for the 325-mesh coal was 66.4 when the collector added was 0.30 lb/T and frother added was 1.30 lb/T.

Flotation kinetic tests were also carried out for wet-ground minus 100-, 200- and 325-mesh Upper Freeport PA coal at the optimized reagent dosages. The test results are given in Tables 12.11 through 12.13 and Figures 12.1 and 12.2. These results show that for all three grinding sizes the combustible material recovery continues to increase with flotation time, but their separation efficiency decreases when flotation time increases beyond 5 minutes. The maximum separation efficiency of Upper Freeport PA coal obtained at the optimal dodecane and MIBC dosages for the three grinding sizes does not vary significantly.

Table 12.10 - Optimization flotation test results for 325-mesh wet-ground Upper Freeport PA coal.

REAGENT DOSAGE			PRODUCT ANALYSIS				REJECTION		
Coll.	Frot.	Mod.	Yield	CMR	Ash	Pyr. S	Ash	Pyr. S	El _H
lb/T	lb/T	lb/T	%	%	%	%	%	%	
0.30	1.30	--	90.5	95.4	7.35	0.50	45.1	71.0	66.4
0.60	1.30	--	93.1	97.2	7.88	0.55	37.8	67.2	64.4
1.00	1.30	--	92.4	96.9	7.68	0.57	40.8	66.2	63.1
1.00	0.80	--	91.1	95.7	7.76	0.50	42.1	71.0	66.7

Figure 12.1 - Combustible material recovery as a function of time for Upper Freeport PA coal ground to minus 100-mesh, minus 200-mesh and minus 325-mesh.

Figure 12.2 - Separation efficiency as a function of time for Upper Freeport PA coal ground to minus 100-mesh, minus 200-mesh and minus 325-mesh.

Table 12.11 - The test results of flotation kinetics using dodecane 0.25 lb/T and MIBC 0.70 lb/T for 100-mesh wet-ground Upper Freeport PA coal.

Flot. time (min.)	Prod. Name	PRODUCT ANALYSIS				REJECTION		EI _H %
		Yield %	CMR %	Ash %	Pyr. S %	Ash %	Pyr. S %	
0.5	Conc. 1	39.8	42.7	6.02	0.47	80.6	88.0	30.7
1.0	Conc. 2	20.5	21.7	7.41	0.53	68.3	81.0	45.4
2.0	Conc. 3	12.9	13.4	8.66	0.57	59.3	76.3	54.1
3.0	Conc. 4	8.7	9.0	8.89	0.60	53.0	73.0	59.8
5.0	Conc. 5	8.3	8.3	12.61	0.97	44.5	67.8	62.3
10.0	Conc. 6	3.0	2.3	32.1	3.34	36.7	61.4	58.8
20.0	Conc. 7	1.0	0.6	47.73	5.55	32.8	57.8	55.8

Table 12.12 - The results of flotation kinetics using MIBC 1.0 lb/T for wet-ground minus 200-mesh Upper Freeport PA coal.

Flot. time (min.)	Prod. Name	PRODUCT ANALYSIS				REJECTION		EI _H %
		Yield %	CMR %	Ash %	Pyr. S %	Ash %	Pyr. S %	
0.5	Conc. 1	22.3	24.2	4.58	0.31	91.5	95.6	19.8
1.0	Conc. 2	21.6	23.2	4.97	0.31	82.5	91.3	38.7
2.0	Conc. 3	23.2	24.8	5.93	0.50	71.1	83.4	56.0
3.0	Conc. 4	8.3	8.7	7.53	0.65	65.9	80.4	61.3
5.0	Conc. 5	9.8	9.7	13.09	0.98	55.2	74.2	64.8
10.0	Conc. 6	6.5	5.6	23.39	1.64	42.5	67.4	63.6
20.0	Conc. 7	2.5	1.7	39.34	2.59	34.3	63.2	61.1

Table 12.13 - The test results of flotation kinetics using dodecane 0.30 lb/T and MIBC 1.30 lb/T for 325-mesh wet-ground Upper Freeport PA coal.

Flot. time (min.)	Prod. Name	PRODUCT ANALYSIS				REJECTION		EI _H %
		Yield %	CMR %	Ash %	Pyr. S %	Ash %	Pyr. S %	
0.5	Conc. 1	22.5	24.1	6.37	0.47	88.7	93.2	17.3
1.0	Conc. 2	29.6	31.6	6.73	0.47	73.0	84.3	40.0
2.0	Conc. 3	23.4	24.6	8.05	0.56	58.2	75.9	56.2
3.0	Conc. 4	9.0	9.2	10.89	0.78	50.5	71.4	60.9
5.0	Conc. 5	5.7	5.7	12.62	0.93	44.8	68.0	63.2
10.0	Conc. 6	3.6	2.8	31.99	3.63	35.7	59.6	57.6
20.0	Conc. 7	1.6	0.8	57.84	7.77	28.4	51.6	50.4

12.2 Effect of Frother Conditioning Time on Separation Efficiency and Flotation Kinetics

In a continued effort to optimize the flotation procedure, we studied the effect of frother conditioning time on the flotation separation efficiency and flotation kinetics of the three base coals. In this series of flotation tests, all operating conditions were the same as the standard flotation procedures except the frother conditioning time. For all three coals, 200-mesh grind samples were used in the tests and the pH of the pulp was the natural pH of each coal sample tested. The frother conditioning times were changed by adjusting the time interval between the addition of collector (dodecane) and frother (MIBC), so that the total conditioning time (sum of pulp, collector and frother conditioning time) was constant (7 minutes). In order to achieve reliable results, duplicates were run for every test, and the proximate and total sulfur of flotation products of each test were analyzed separately. The combustible material recovery, ash rejection, pyritic sulfur rejection and Hancock efficiency index of each test were calculated based on the analyses of the concentrates and the tailings. The final results reported here are the average of the two tests. In order to calculate flotation rate constants, the flotation concentrate was collected at regular intervals, 0.5, 1, 3 and 5 minutes.

Tables 12.14, 12.15, and 12.16 give the combustible material recovery, ash rejection, pyritic sulfur rejection and efficiency index of Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA coals as a function of frother conditioning time (0.5, 1, 2 and 3 minutes) at various flotation times. The reagent dosage and flotation pH for each coal are also given in these tables. These results show that for all three coals the frother conditioning time does not affect the efficiency index at long flotation time (3 and 5 minutes). However, at short flotation time (0.5 and 1 minute), increasing frother conditioning time decreases the efficiency index.

Figures 12.3, 12.4 and 12.5 show the effect of frother conditioning time on the flotation rate constant and ultimate recovery of combustible materials for the three coals. These values were calculated by fitting the classical first-order kinetic model with a sine function distribution of flotability (Equation 11.3). It can be seen from the plots that for all three coals the ultimate CMR decreases only slightly (1 to 2 percent) with increasing frother conditioning time between 0.5 to 3 minutes. However, for the same frother conditioning time period, the flotation rate

Figure 12.3 - Effect of frother conditioning time on the flotation rate constant and ultimate recovery of combustible material of Illinois No. 6 coal.

Figure 12.4 - Effect of frother conditioning time on the flotation rate constant and ultimate recovery of combustible material of Pittsburgh No. 8 coal.

Figure 12.5 - Effect of frother conditioning time on the flotation rate constant and ultimate recovery of combustible material of Upper Freeport PA coal.

constant decreases quite significantly. This may be due to adsorption of MIBC on coal surfaces which results in a decrease in its frothing power during the flotation operation (1,2).

12.3 Flotation Circuitry

Industrial application of flotation to the separation of solids is rarely limited to a single-stage process. Generally, flotation products are re-floated one or more times to yield a cleaner product. The material that floats in the first step but not in the second step, called middlings, can be subjected to a number of different treatment methods. Among these are regrinding step followed by recharging back to the first flotation stage. In any case, the treatment of the middlings can have a profound effect on the final product. Part of the research associated with this experimental study was designed to determine the effect of various flotation circuits on the final product. These results are reported in this section.

Table 12.14 - Effect of frother conditioning time on flotation of Illinois No. 6 coal (200-mesh grind) at collector (dodecane) dosage 4.92 lb/T, frother (MIBC) dosage 0.98 lb/T and pH 7.2.

Frother Cond. Time, min.	Flotation Time, min.	CMR %	Ash Rej. %	Pyr S Rej. %	El _H —
0.5	0.5	37.5	81.2	83.1	20.6
	1.0	61.2	69.5	72.2	33.5
	3.0	85.4	54.6	57.9	43.3
	5.0	89.2	51.0	54.3	43.5
1.0	0.5	37.5	81.3	83.1	20.6
	1.0	61.5	69.7	71.3	32.8
	3.0	85.8	55.0	56.3	42.1
	5.0	89.9	51.1	52.3	42.2
2.0	0.5	33.0	83.9	84.9	17.9
	1.0	55.2	73.2	75.4	30.7
	3.0	82.4	57.2	59.9	42.3
	5.0	86.9	53.4	56.3	43.2
3.0	0.5	26.6	87.2	88.4	15.0
	1.0	47.5	77.5	79.5	27.0
	3.0	79.5	60.6	63.0	42.5
	5.0	84.5	56.9	59.3	43.8

Table 12.15 - Effect of frother conditioning time on flotation of Pittsburgh No. 8 coal (200-mesh grind at collector (dodecane) dosage 1.92 lb/T, frother (MIBC) dosage 0.39 lb/T and pH 3.9.

Frother Cond. Time, min.	Flotation Time, min.	CMR %	Ash Rej. %	Pyr S Rej. %	El _H —
0.5	0.5	27.7	87.2	87.4	15.1
	1.0	51.3	76.1	76.3	27.6
	3.0	79.4	61.2	61.1	40.4
	5.0	84.9	56.6	56.4	41.2
1.0	0.5	25.9	87.8	87.8	13.7
	1.0	49.5	76.9	77.5	27.0
	3.0	78.5	62.0	62.2	40.7
	5.0	83.6	58.2	58.6	42.1
2.0	0.5	22.2	89.8	90.2	12.4
	1.0	44.1	79.9	80.4	24.4
	3.0	75.8	64.2	64.5	40.3
	5.0	82.7	59.1	59.2	41.9
3.0	0.5	23.7	89.2	89.3	13.0
	1.0	46.4	78.8	78.8	25.1
	3.0	76.6	63.0	63.0	39.6
	5.0	82.9	58.1	58.2	41.1

Table 12.16 - Effect of frother conditioning time on flotation of Upper Freeport PA coal (200-mesh grind) at collector (dodecane) dosage 0.48 lb/T, frother (MIBC) dosage 0.21 lb/T and pH 3.4.

Frother Cond. Time, min.	Flotation Time, min.	CMR %	Ash Rej. %	Pyr S Rej. %	El _H —
0.5	0.5	31.1	85.1	87.1	18.2
	1.0	52.4	75.0	79.6	32.0
	3.0	80.0	60.0	68.3	48.2
	5.0	86.5	54.8	64.5	51.0
1.0	0.5	29.9	86.3	88.2	18.2
	1.0	51.5	76.2	80.4	31.9
	3.0	79.4	61.0	69.6	49.0
	5.0	85.8	55.5	65.5	51.3
2.0	0.5	26.2	87.9	90.2	16.4
	1.0	46.1	78.5	82.7	28.8
	3.0	76.6	62.5	71.2	47.8
	5.0	83.9	56.7	66.9	50.8
3.0	0.5	23.8	88.8	92.4	16.2
	1.0	43.0	79.8	86.3	29.3
	3.0	74.0	63.9	75.5	49.5
	5.0	82.1	58.2	71.6	53.8

12.3.1 Closed-Circuit Flotation

The beneficial effects of butanol on the flotation of Upper Freeport PA coal were investigated in more detail through closed-circuit flotation. In continuous plant operation, the middlings are often fed back to the feed-conditioning tank or roughing cell to mix with the new feed. In laboratory tests, the simulation of this flowsheet is called "closed-circuit" or "cyclic circuit", as shown in Figure 12.6. The middlings of the first batch are combined with the feed for the second batch, the middlings of the second batch are combined with the feed for the third batch, and so on. The results of a five-cycle flotation circuit, using 1.0 lb/T MIBC, given in Table 12.17, show that after three cycles the CMR attained was 85.8% with a 90.6% pyritic sulfur rejection, resulting in a separation efficiency of 71.0. This set of tests represents the base-line, to which further tests with butanol or other modifiers can be evaluated.

Results for five-cycle closed circuit flotation of Upper Freeport PA coal ground with 4 lb/T butanol (no other reagents added) are given in Table 12.18. These results show that after three cycles equilibrium has been reached resulting in a CMR of close to 90% with a pyritic sulfur

Figure 12.6-Closed circuit flotation

rejection of about 85% was obtained. The efficiency index associated with this separation is about 75. This represents an increase of 5 points in the efficiency index over that when MIBC is used. These results are very close to the initial goal of 90% Btu recovery with 90% pyritic sulfur rejection. From experimental observation, it appears that if the cell volume was changed for the fourth and fifth cycles to accommodate the increased froth volume and the reagent dosage optimized the results may be improved even more.

Figure 12.7 - Cleaner-Scavenger Circuit

Table 12.17 - Flotation results of five cycles using MIBC 1.0 lb/T for wet-ground 200-mesh Upper Freeport PA coal.

Product name	PRODUCT ANALYSIS				REJECTION		EI _H
	Yield %	CMR %	Ash %	Pyr. S %	Ash %	Pyr. S %	
Concentrate #1	53.3	58.7	3.51	0.21	84.9	92.8	51.5
Tail #1	18.2		38.62				
Feed #1			12.42	1.56			
Concentrate #2	66.3	72.5	4.25	0.25	77.3	89.4	61.9
Tail #2	15.9		42.57				
Feed #2			12.42	1.56			
Concentrate #3	79.5	85.8	4.56	0.29	68.7	85.2	71.0
Tail #3	20.5		38.86				
Feed #3			11.59	1.56			
Concentrate #4	83.7	89.7	5.1	0.34	63.9	81.8	71.5
Tail #4	16.6		43.4				
Feed #4	100.3		11.50	1.56			
Concentrate #5	80.4	86.9	4.79	0.31	67.7	84.0	70.9
Tail #5	19.6		41.15				
Feed #5	100.0		11.92	1.56			
Middling	46.1	45.4	13.79	1.20	51.9	35.9	

Table 12.18 - Flotation results of 200-mesh wet-ground Upper Freeport PA coal for five cycles with 4.0 lb/T butanol added to the mill.

Product	FLOTATION PRODUCT ANALYSIS					REJECTION			
	Yield %	Ash %	Pyr. S %	CMR %	HVR %	Ash %	Pyr. S %	El _H —	El _H * —
Con. 1	54.6	3.40	0.20	60.2	61.6	84.1	93.0	53.2	54.6
Con. 2	79.8	4.44	0.25	87.1	88.6	71.5	87.2	74.3	75.8
Con. 3	83.9	4.87	0.28	89.9	91.5	63.6	84.9	74.8	76.4
Con. 4	82.6	5.10	0.28	89.5	91.1	65.9	85.2	74.7	76.3
Con. 5	82.0	4.95	0.26	88.9	91.5	67.1	86.3	75.2	76.5
Middling	34.8	16.39	1.48						

* El_H based on Btu recovery (HVR)

12.3.2 Cleaning and Scavenging

In order to increase the pyritic-sulfur rejection and maintain the high recovery of combustible materials (or the recovery of heating value), tests with circuits that include cleaning and scavenging operations were also performed in addition to the closed-circuit studies.

The test flowsheet with a cleaning and scavenging circuit is shown in Figure 12.7 and the results for Upper Freeport PA coal ground with 6.0 lb/T ethanol are summarized in Table 12.19. It can be seen that the cleaning operation is beneficial with an overall pyritic-sulfur rejection of close to 80%. In the first cleaning stage, half of the material recovered in the roughing stage is rejected and sent to the scavenging stage to help increase the recovery. After two stages of scavenging, the CMR increased to 88.8% and pyritic-sulfur rejection decreased to 79.8% giving a separation efficiency of 68.6. Without the cleaning circuit, the CMR was 92.2% and the pyritic sulfur rejection was 73.7%, giving a separation efficiency of 65.9.

Table 12.19 - Flotation results with cleaning and scavenging for 200-mesh wet-ground Upper Freeport PA coal ground with 6.0 lb/T ethanol.

Product	Coll. lb/T	Froth. lb/T	PRODUCT ANALYSIS				REJECTION		El _H —
			Yield %	CMR %	Ash %	Pyr. S %	Ash %	Pyr. S %	
Rougher	0.5	0.3	86.6	92.2	--	--	55.8	73.7	65.9
Conc. 1	--	--	50.2	55.0	3.3	0.22	85.9	93.2	48.2
Conc. 2	0.1	0.1	24.7	81.3	6.0	0.57	73.3	84.5	65.8
Conc. 3	--	0.2	7.4	88.8	10.5	1.03	66.7	79.8	68.6
Middlings			4.3	92.2	29.5	2.30	55.8	73.7	65.9

Table 12.20 - Flotation results with cleaning and scavenging for 200-mesh wet-ground Upper Freeport PA coal ground with 4.0 lb/T butanol.

Product	Coll. lb/T	Froth. lb/T	PRODUCT ANALYSIS				REJECTION		E _{I_H}
			Yield %	CMR %	Ash %	Pyr. S %	Ash %	Pyr. S %	
Rougher	0.3	--	84.6	90.4	--	--	59.2	77.8	68.2
Conc. 1	--	--	54.5	59.8	3.2	0.19	85.3	93.6	53.4
Conc. 2	0.1	--	13.2	73.9	5.2	0.39	78.8	90.4	64.3
Middling			16.9	90.4	13.7	1.21	59.2	77.8	68.2

The flowsheet used for the tests with butanol with cleaning and scavenging stages is identical to that shown in Figure 12.7 except that the last two scavenger operations were combined into one (total flotation time of 2 minutes). The test results summarized in Table 12.20 indicate that a cleaning operation is very effective for pyritic sulfur rejection (93.6%) but, as with ethanol, the yield is reduced. However, the scavenging cell helps to minimize the decrease in yield and a separation efficiency of 64.3 is achieved. This is 4 points less than the efficiency index of 68.2 obtained without cleaning.

12.3.3 Staged Reagent Addition

Results of flotation reagent tests on Upper Freeport PA coal showed the addition of either ethanol or butanol to the grinding mill to have a beneficial effect on the flotation separation. High-levels of collector (n-dodecane) and frother (MIBC) increase the combustible material recovery (>90%) but decreases the pyritic-sulfur rejection (<77%); the reverse trend occurs when low levels are used. Therefore, in an attempt to increase the pyritic sulfur rejection and maintain a high combustible material recovery, the effect of incremental addition (staged addition) of collector and frother on the separation efficiency was investigated.

For these tests, the optimum collector and frother dosage was used in three stages while the ethanol (6 lb/T) was added to the grinding mill. The flowsheet is shown in Figure 12.8 and the results obtained are summarized in Table 12.21. It can be seen that in the first stage, the high pyritic sulfur rejection (91.6%) was offset by a corresponding low combustible material recovery (49.6%). The cumulative results of all three stages resulted in a CMR of 93% and an overall pyritic-sulfur rejection of 69.4% yielding an efficiency of 62.4.

Figure 12.8 - Flow sheet for staged reagent addition.

Since the previous optimization tests using butanol added to the mill, collector to the cell, and no frother showed promising results, tests with butanol added to the mill and staged collector addition (at a reduced level) were also performed. The flowsheet used is identical to that shown in Figure 12.8 except that the flotation time for the last two steps was halved.

Flotation results with coal ground with 3 lb/T butanol and 0.2 lb/T collector (Table 12.22) show that neither the recovery nor pyritic sulfur rejection is better than results obtained with ethanol.

However, by increasing the butanol to 4 lb/T and the collector to 0.3 lb/T (Table 12.23) the recovery increased slightly to 94.9% while the pyritic sulfur rejection dropped to 72.1%. This

Table 12.21 - Flotation results of 200-mesh wet-ground Upper Freeport PA coal with 6 lb/T ethanol added to the mill with staged collector and frother addition.

Product	Coll. lb/T	Froth. lb/T	PRODUCT ANALYSIS				REJECTION		El _H
			Yield %	CMR %	Ash %	Pyr. S %	Ash %	Pyr. S %	
Conc. 1	0.3	0.1	45.6	49.6	4.07	0.30	84.4	91.6	41.2
Conc. 2	0.1	0.1	31.0	82.4	6.88	0.77	82.4	66.1	76.8
Conc. 3	0.1	0.1	10.9	93.0	14.11	1.10	93.0	53.1	69.4

Table 12.22 - Flotation results using 3 lb/T butanol in the mill and staged collector addition in the cell for 200-mesh wet-ground Upper Freeport PA coal.

Product	PRODUCT ANALYSIS				REJECTION		EI _H
	Yield %	CMR %	Ash %	Pyr. S %	Ash %	Pyr. S %	
Concentrate 1	53.0	58.7	4.19	0.29	83.6	90.5	49.2
Concentrate 2	13.3	82.2	9.29	0.92	68.1	77.8	60.0
Concentrate 3	6.0	88.2	15.21	1.20	61.4	73.3	61.5

is marginally better than the previous tests with ethanol and butanol or the closed cycle tests showing that staged frother and collector addition do not produce better flotation separation.

12.4 Column flotation

Flotation tests were carried out using a column to compare its performance with that of the mechanically agitated cell. Wet-ground 200-mesh Pittsburgh No. 8 coal was used as the flotation feed and the tests were conducted at pH 8, adjusted by adding sodium hydroxide in the mill. The column was assembled by joining together seven 3.5 cm x 3.5 cm x 15 cm sections made of plexiglass. In this design, the height of the column can be varied by attaching or detaching the desired number of sections.

Experiments were conducted in a batch mode using 31 grams of coal at 6.25 wt% solids concentration, the same as that used in the mechanically agitated cell. Air flow rate was maintained in the range of 0.65 to 1.55 liters per minute since preliminary experimental results showed that selectivity is not significantly affected by air flow rate in this range. The froth was allowed to accumulate for 4 minutes following which the samples were drawn from a range of different fixed depths of the froth column using a 0.4 cm ID glass tube.

Table 12.23 - Flotation results using 4 lb/T butanol in the mill and staged collector addition in the cell for 200-mesh wet-ground Upper Freeport PA coal.

Product	Coll. lb/T	Froth. lb/T	PRODUCT ANALYSIS				REJECTION		EI _H
			Yield %	CMR %	Ash %	Pyr. S %	Ash %	Pyr. S %	
Conc. 1	--	--	70.6	76.6	4.38	0.33	73.9	85.6	62.2
Conc. 2	0.1	--	13.3	89.9	11.83	0.97	60.6	77.7	67.6
Conc. 3	0.2	--	5.6	94.9	20.75	1.62	50.8	72.1	67.0

12.4.1 Comparison of Performances of Mechanically Agitated Cell and Flotation Column

Results of the flotation tests conducted using the column are plotted in terms of selectivity curves for non-pyritic minerals and pyrite rejections in Figures 12.9 and 12.10 respectively. Results obtained using the mechanically agitated cell are also shown in these figures for comparison. In the case of the column, different points along the selectivity curve represent cumulative values of combustible material recovery versus pyrite or non-pyritic minerals rejection. These values were calculated using the analysis of froth products sampled at different depths of the froth column. In the curves generated for the mechanically agitated cell, different points along the curve were obtained by varying the frother dosage. Thus, in the case of the column, selectivity curve was generated using data obtained from a single experiment whereas the curve for the cell was generated using data obtained from several experiments.

Since the second stage of the cell is used for cleaning the first stage concentrate, the results discussed above are in accord with the view that the froth phase of the flotation column functions as a cleaning stage.

12.4.2 Effect of bubble size and wash water

The effect of bubble size on the performance of the column was tested by using frits of different porosities. Figure 12.11 shows that the pyrite rejection obtained using the coarse frit (40-60 microns; coarser bubbles) is higher than that obtained with the medium frit (10-15 microns). This observation can be explained in the following manner: Finer bubbles offer larger resistance to flow of liquid past its surface due to greater specific surface area and, therefore, smaller equivalent hydraulic radius. Due to this increased resistance, drainage velocity of the liquid past the finer bubbles will be lower. As a consequence, the residence time of the trapped solids (impurity) in the froth will increase and this will result in decreased selectivity. In the case of non-pyritic minerals, bubble size has no effect on the selectivity (Figure 12.12). This can be explained by the fact that the selectivity curve obtained using the medium frit (finer bubbles) has already reached the limit of physical separation as it overlaps the washability curve.

Figures 12.13 and 12.14 show the effect of wash water on pyrite rejection in the tests carried out using the coarse frit and the medium frit respectively. It can be seen from these figures that, in the range tested, there is no effect of wash water on selectivity.

Figure 12.9 - Comparison of performances of mechanically agitated cell and flotation column for the rejection of non-pyritic minerals from Pittsburgh No. 8 coal.

Figure 12.10 - Comparison of performances of mechanically agitated cell and flotation column for the rejection of pyrite from Pittsburgh No. 8 coal.

Figure 12.11 - Effect of bubble size on the rejection of pyrite from Pittsburgh No. 8 coal.

Figure 12.12 - Effect of bubble size on the rejection of non-pyritic minerals from Pittsburgh No. 8 coal.

Figure 12.13 - Effect of wash water on the rejection of pyrite from Pittsburgh No. 8 coal - tests with coarse frit.

Figure 12.14 - Effect of wash water on the rejection of pyrite from Pittsburgh No. 8 coal - tests with medium frit.

Table 12.24 - Results of combined tabling and flotation for pyritic sulfur removal from the base coals.

<u>Coal</u>	<u>Test Type*</u>	<u>CMR</u> <u>%</u>	<u>Ash</u> <u>Rej. %</u>	<u>Pyr. S</u> <u>Rej. %</u>	<u>El_H</u>
Illinois No. 6	T (28 M)	93.2	33.5	62.2	55.3
	T (28 M) + F (200 M)	74.6	68.8	76.4	51.0
	F (28 M)	80.7	54.7	62.9	43.6
	F (28 M) + F (200 M)	76.7	69.0	74.4	51.1
	F (200 M) standard	81.6	54.7	58.9	40.5
Pittsburgh No. 8	T (28 M)	93.4	38.0	49.4	42.8
	T (28 M) + F (200 M)	79.9	71.0	76.3	56.2
	F (28 M)	95.0	39.4	29.8	24.9
	F (28 M) + F (200 M)	89.2	58.5	61.0	50.2
	F (200 M) standard	76.6	65.7	66.0	42.6
Upper Freeport PA	T (28 M)	89.5	45.3	63.1	52.6
	T (28 M) + F (200 M)	77.6	70.1	86.6	64.2
	F (28 M)	86.0	52.7	60.3	46.3
	F (28 M) + F (200 M)	75.9	72.7	85.2	61.1
	F (200 M) standard	73.8	71.1	78.5	52.3

* T = Tabling, F = Standard Flotation, M = Mesh

In summary, the selectivity obtained using two stages in a mechanically agitated contactor can be matched by a single stage of the column; this indicates that the deep froth bed usually achieved in a column acts as a cleaning stage. Also, the selectivity obtained using the coarse frit (larger bubbles) is higher than that achieved with the medium frit (smaller bubbles); this is due to the higher resistance offered by the finer bubbles to the drainage of liquid and a consequent increase in the residence time of the solids in the froth.

12.5 Combination of Tabling and Flotation

In order to try to take advantage of alternative methods for the removal of free pyrite particles, a combination of gravity separation and flotation (shaking tabling and flotation), tests were conducted. Tabling experiments were performed using a laboratory Wilfley shaking table (1.5 x 3.25 feet deck). In carrying out a tabling test, 500 grams of a given coal was wet ground to minus 28 mesh and the pulp was fed to the shaking table at a feed rate of 200 g/minute. After tabling, the cleaned coal was reground to 200 mesh and then floated using the standard flotation

test procedure. In order to compare the results, similar tests were conducted with two-stage flotation. Stage 1 involved floating the material at 28 mesh and stage 2 involved floating the concentrate from stage 1 after it had been reground to minus 200 mesh. Table 12.24 presents the results of these tests along with the values obtained in the standard flotation tests for 200-mesh coal. Clearly, for 28-mesh coal, the efficiency index for coarse pyrite rejection by tabling is significantly higher than that obtained in the first-stage flotation. By pre-cleaning 28-mesh coal with a shaking table and floating the tabling concentrates after regrinding them to 200 mesh, the separation efficiency can be improved significantly over single-stage flotation of 200-mesh coal. Furthermore, the separation efficiency is equivalent or better than that obtained by two-stage grinding together with two-stage flotation.

12.6 References

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2. N. T. Moxon, C. N. Bensley, R. Keast-jones and S. K. Nicol, "Insoluble Oils in Coal Flotation: The effects of Surface Spreading and Pore Penetration," Int. J. of Mineral Processing, Vol. 21, pp. 261-274 (1987).

13.0 WEATHERING STUDIES OF THE BASE COALS

The overall objective of the weathering study was to determine and understand the effect of storage environment on the surface properties of coal and, hence, on its flotation behavior. Weathering phenomena are important in coal handling and processing. The impact of weathering on the behavior of coal ranges from the spontaneous combustion of coal in storage to alteration of the physical, chemical and surface characteristics. For example, weathering has been reported to decrease the surface area of coal (1), increase the moisture adsorption capacity (2,3), decrease the calorific value (4), increase the heat of wetting (5), decrease the flotability (6), and reduce the coking power (7).

When exposed to atmospheric air, coals absorb and desorb moisture depending on the heat and humidity of the environment and can react chemically with oxygen. While absorption of moisture leads to swelling, desorption leads to shrinkage. Repeated wetting and drying can result in fissuring and spalling. To what extent a given coal decrepitates depends on its rank and composition and the vagaries of the climatic conditions.

Chemisorption of oxygen on coal results in the formation of surface oxygen functional groups such as carboxyl, hydroxyl and carbonyl radicals. These oxygen functional groups reduce the hydrophobicity of the coal surface and thereby have a deleterious effect on surface-based separation processes, such as flotation and oil agglomeration. In some instances weathering or oxidation may have beneficial effects. For example, when coal particles are to be heated on grates or under fluidized conditions (as in gasification processes), agglomeration of the particles is undesirable. Since oxidation decreases the agglomerability, a pre-oxidation step is often included in the process scheme to prevent particles from adhering to each other. Most of the literature pertaining to the weathering aspects of coal research is limited to coal samples oxidized under controlled laboratory conditions, and little is known about the changes in the behavior of coal exposed to natural weathering, which has been the impetus for this research effort.

For each of the three base coals, three size fractions, namely, +1 inch, 1 x 1/4-inch, 1/4-inch x 0, were weathered under "inert" (argon), covered and open conditions and samples were collected at scheduled intervals. In all, a total of thirteen increments of weathered samples were collected during the period starting from November 1988 and extending into December

1989. The schedule provided for the collection of six biweekly samples for the first three months, followed by monthly sampling for the next four months and bimonthly sample collection for the final six months. After collection of the sample, the minus 1/4-inch material was dry-screened at 28 mesh, and both size fractions (that is, the 1/4-inch x 28 mesh and minus 28-mesh material) were re-inerted prior to shipping.

Experimental work involved with the testing of these weathered samples was substantial during the first year of this research program. The test work consisted of surface and bulk characterization of the weathered samples, including proximate and sieve analyses. The surface hydrophobicity of some of these samples was assessed using film flotation, induction time measurements, microflotation studies and standard flotation testing. Zeta potential measurements and DRIFT spectra were also obtained for some selected samples. Since the weathering effect is more pronounced on smaller particles, the majority of the laboratory-scale standard flotation tests, as well as surface characterization, was carried out on the finest size fraction, namely the minus 28-mesh material (natural fines obtained by screening). Flotation testing was also performed on ground samples of the next size interval (1/4 inch x 28 mesh). The results are discussed in paragraphs that follow in this section.

13.1 Weathering Site Preparation and Sampling

Each weathering sample consisted of about 14 tons of minus 4-inch coal. The material to be used for the weathering study was screened on site to three size fractions:

- Coarse (+1 inch)
- Medium (1 inch x 1/4 inch)
- Fine (1/4 inch x 0)

Each of these fractions was weathered separately by storing them under three different modes:

- Inert (in drums stored under argon atmosphere)
- Covered (in drums without lids but with plywood boards on top)
- Open (in a stockpile completely exposed to the atmosphere)

The splitting scheme used in preparing the material for this purpose is given in Figure 13.1.

The 14-ton sample set aside for weathering was screened using two Sweco 30-inch diameter screens, one having one-inch openings and the other 1/4-inch openings. The screening was done at the mine site using a portable diesel generator to supply power to the screens and for flood

Once filled, the 55-gallon drums containing the two coarse fractions and the 5-gallon buckets containing the fine sizes were separated into two categories. Half of the drums and half of the buckets were "inerted" by purging argon into the bags, which were then tied with a special tape. The drums and buckets were then inerted around the bags and the lids were closed and clamps replaced. The other half, designated as covered samples, was covered with plywood boards allowing a gap between the top of the containers and the ply boards. The three open stockpiles were dressed on the top to prevent rain water accumulation. The site was fenced in

Figure 13.2 - Coal weathering stockpiles of Upper Freeport PA coal at the Indiana County, Pennsylvania mine site.

with wooden posts and ropes with relevant information of the project and emergency telephone numbers posted at the entry posts. Figure 13.2 is a photograph of the Upper Freeport PA weathering piles located in Indiana County, Pennsylvania.

13.2 Periodic Collection of Weathering Samples

According to the Project Work Plan, coal weathering was to be conducted over a period of 12 months, taking a maximum of 15 increments. The sample collection schedule was revised and the new one shown in Table 13.1, indicating the six biweekly increments taken during the first three months, followed by monthly increments for the next four months. Subsequently, three more samples were collected on a bi-monthly basis, thus covering a total of thirteen months instead of twelve.

Periodic sample collection at each site was carried out by trained laboratory technicians from the same coal testing laboratories who assisted during the bulk coal sample collection and preparation effort. At every scheduled collection of a weathering sample, one drum from the

Table 13.1 - Coal weathering sampling schedule for the three base coals.

<u>Increment</u>	<u>Weathering Time, months</u>	<u>Pittsburgh No. 8 Belmont Co. Warnock, OH</u>	<u>Upper Freeport PA Indiana Co. Blacklick, PA</u>	<u>Illinois No. 6 Randolph Co. Marissa, IL</u>
1	0.5	11/22/88	11/30/88	12/07/88
2	1.0	12/07/88	12/15/88	12/20/88
3	1.5	12/22/88	1/04/89	1/04/89
4	2.0	1/05/89	1/16/89	1/17/89
5	2.5	1/23/89	1/27/89	1/31/89
6	3.0	2/10/89	2/10/89	2/14/89
7	4.0	3/07/89	3/08/89	3/13/89
8	5.0	4/07/89	4/06/89	4/10/89
9	6.0	5/09/89	5/08/89	5/11/89
10	7.0	6/07/89	6/06/89	6/09/89
11	9.0	8/08/89	8/07/89	8/10/89
12	11.0	10/06/89	10/05/89	10/09/89
13	13.0	12/06/89	12/05/89	12/08/89

+1 inch and 1-inch x 1/4-inch fractions stored in the covered and inert modes, as an individual discrete increment, was collected. Also, one 5-gallon bucket each from the 1/4-inch x 0 covered and inert modes was collected.

The sampling procedure for the samples stored in the open stockpiles first involved carefully scraping the top surface (about 1/4-inch) prior to actual sampling. This was necessary because the stockpiles became contaminated with dirt, leaves, and other wind-borne debris, in the case of the fine and medium size fractions. The stockpiles were then carefully sampled, taking small increments from different positions along the pile; and the samples were placed in polyethylene bags and then sealed. Following sampling, the stockpiles were smoothed over on all sides. The approximate amount of material collected from the stockpiles was the following:

- Coarse, +1-inch (about 120 lb)
- Medium, 1-inch x 1/4-inch (about 100 lb)
- Fine, 1/4-inch x 0 (about 80 lb)

The collected samples were then taken to the designated laboratory (Geochemical Testing, Somerset, PA for Pittsburgh No. 8 coal; Gould Energy-Warner Division Labs, Cresson, PA for

Upper Freeport PA coal; and Commercial Testing and Engineering, Henderson, KY for Illinois No. 6 coal) where they were inerted individually and packed in drums such that one drum held all the sizes belonging to each of the three weathering modes. The minus 1/4-inch material from each of the three storage modes was dry-screened at 28 mesh to form two fractions each: 1/4-inch x 28 mesh and 28 mesh x 0. The samples were then placed in separate plastic bags (one corresponding to each of the three storage modes), inerted and packed in three drums (corresponding to the three storage modes), each of which contained four size fractions of the coal: +1-inch, 1-inch x 1/4-inch, 1/4-inch x 28 mesh, and 28 mesh x 0. Each drum was then inerted with argon gas and sealed tightly to maintain the inert atmosphere and labelled for shipping.

13.2.1 QA/QC Analysis of Coal Weathering Samples

The thirteen increments of the weathered coal samples received by the University of California and the University of Pittsburgh as a part of the weathering program can be used as a QA/QC index for the uniformity of the material in this particular program. As will be discussed in sections that follow, the minus 1/4-inch samples of all three weathering modes were screened at 28 mesh at the laboratories, the samples reinerted in plastic bags, and shipped along with the other samples. The minus 28-mesh material was then used to conduct experiments to delineate the effect of weathering on the flotation behavior of the coal.

The reconstituted feed ash and sulfur values from these flotation tests were calculated as a part of the QA/QC program, analyzed statistically, and are reported in Table 13.2 for Pittsburgh No. 8 coal. The consistency of ash and sulfur values among all inert, covered and open weathering increments show that we are able to reproduce the sampling and splitting procedures. Similar results for the Illinois No. 6 and Upper Freeport PA coal are reported in Tables 13.3 and 13.4, respectively. The screened minus 28-mesh fines from the Illinois No. 6 coal weathered samples have considerably higher ash contents as compared to the total ash of the parent sample. This is probably due to the concentration of liberated mineral matter, particularly clays, into the natural fines. It is also for this reason that the variability of ash between increments is higher for this coal as compared to the other two coals.

Table 13.2 - Ash and total sulfur of the minus 28-mesh screened fraction of Pittsburgh No. 8 weathering samples reconstituted from flotation product analysis.

Increment No.	Weathering Time, mths.	INERT		COVERED		OPEN	
		Ash %	Tot. S %	Ash %	Tot. S %	Ash %	Tot. S %
1	0.5	17.19	6.42	17.14	6.37	17.13	6.21
2	1.0	16.92	6.04	--	--	17.91	5.92
4	2.0	17.12	6.65	17.18	6.81	18.06	6.63
6	3.0	17.63	6.53	16.99	5.96	17.70	6.30
8	5.0	17.42	5.86	17.10	6.55	17.31	6.43
Mean		17.26	6.30	17.09	6.42	17.62	6.30
Standard Deviation		0.25	0.30	0.07	0.31	0.35	0.24

Table 13.3 - Ash and total sulfur of the minus 28-mesh screened fraction of Illinois No. 6 weathering samples reconstituted from flotation product analysis.

Increment No.	Weathering Time, mths.	INERT		OPEN	
		Ash %	Tot. S %	Ash %	Tot. S %
1	0.5	26.93	4.98	26.72	4.84
2	1.0	28.11	4.48	30.48	4.90
3	1.5	27.67	4.95	30.02	4.62
4	2.0	27.65	5.02	26.87	5.14
5	2.5	28.05	4.56	28.07	4.74
6	3.0	28.48	4.94	--	--
7	4.0	24.17	4.97	29.36	4.79
Mean		27.3	4.84	28.6	4.84
Standard Deviation		1.35	0.21	1.46	0.16

Table 13.4 - Ash and total sulfur of the minus 28-mesh screened fraction of Upper Freeport PA weathering samples reconstituted from flotation product analysis.

Increment No.	Weathering Time, mths.	INERT		COVERED		OPEN	
		Ash %	Tot. S %	Ash %	Tot. S %	Ash %	Tot. S %
1	0.5	9.10	3.25	9.82	3.45	9.46	3.44
4	2.0	9.36	2.83	9.48	2.90	9.85	2.82
8	5.0	9.43	2.88	9.36	3.11	9.68	2.85
Mean		9.29	2.94	9.54	3.15	9.67	3.04
Standard Deviation		0.14	0.23	0.21	0.23	0.17	0.29

13.3 Collection of Weather Data

In order to complete the weathering study of the coal samples stored under three different storage modes, information on the prevailing weather conditions at the three base coal sites was collected and compiled for use in interpreting the effects of weathering.

Weather data, obtained from the National Climatic Data Center in Asheville, North Carolina, a division of the U.S. Department of Commerce, were available for three locations in close proximity to the three base coal storage sites:

- St. Louis, Missouri, about 30 miles west of Marissa, Illinois where the Illinois No. 6 coal was stored,
- Wheeling, West Virginia, about 10 miles east of Warnock, Ohio where the Pittsburgh No. 8 coal was stored, and
- Indiana, Pennsylvania, about 15 miles northeast of Blacklick, Pennsylvania, where the Upper Freeport PA coal was stored.

The "local climatological data" includes temperature, number of degree days (base 65°F), precipitation (rain, snow, ice), wind and duration of sunshine. For this general part of the country, the main variable was considered to be the seasonal temperature, and this was used to help interpret the results. The weathering study took place over the course of a year, beginning in late Fall. Figures 13.3, 13.4 and 13.5 plot the maximum and minimum temperatures over this period for the weathering site of the Pittsburgh No. 8, Illinois No. 6, and Upper Freeport PA coals, respectively. In each figure, the time for the start of the weathering study is marked. As can be seen from these plots, the weather near the Pittsburgh No. 8 and Upper Freeport PA weathering sites remained cold until about April (the first four months of the study). Near St. Louis, the site of the Illinois No. 6 weathering study, the temperature began to warm up in March and was about 10 degrees warmer during the summer months in comparison to the other two sites. How this seasonal temperature variation affected the weathering rate of the three coals will become apparent in the sections that follow.

Figure 13.3 - The maximum and minimum temperatures from October 1988 through November 1989 in Ohio County, West Virginia, located 15 miles from the Pittsburgh No. 8 coal weathering site in Belmont County, Ohio.

Figure 13.4 - The maximum and minimum temperatures of St. Louis, Missouri from October 1988 through November 1989.

Figure 13.5 - The maximum and minimum temperatures at the Upper Freeport PA coal weathering site in Indiana County, Pennsylvania from October 1988 through November 1989.

13.4 Proximate analysis of weathered samples

As a first step in characterizing the weathered samples, the proximate analysis (volatile matter, fixed carbon and ash) of the 28-mesh x 0 and 1/4-inch x 28-mesh fractions of the samples stored under the inert (argon), covered and open modes was carried out using a LECO MAC 400 analyzer. The samples analyzed are those which were tested for flotation response. The results are tabulated in Table 13.5 for Illinois No. 6 coal, Table 13.6 for Pittsburgh No. 8 coal, and Table 13.7 for Upper Freeport PA coal.

The data given in Table 13.5 show that the weathered samples of Illinois No. 6 coal (minus 28 mesh) from Increments 1 to 13 analyze between 28% and 32% ash. The ash content of the natural minus 28-mesh material is considerably higher than the ash content of the representative research sample (about 17 percent ash). The higher ash in this weathered screened fraction is probably due to the concentration of clays and mineral matter in the natural fines. The next higher size interval, 1/4 inch x 28 mesh (that is, 3 x 28 mesh), screened from weathering

Table 13.5 - Proximate analysis of weathered samples of Illinois No. 6 coal.

Incr.	Weathering Time (mths)	Size	Storage Mode	(ON DRY BASIS, %)			
				Moist. %	Vol. Matter	Fixed Carbon	Ash
1	0.5	28 M x 0	Inert	2.83	34.0	37.2	28.8
		28 M x 0	Covered	3.10	33.7	37.6	28.7
		28 M x 0	Open	3.11	33.8	38.0	28.2
		1/4" x 28 M	Inert	5.73	35.2	45.0	19.8
		1/4" x 28 M	Open	5.90	35.1	44.5	20.4
2	1.0	28 M x 0	Inert	3.11	33.1	37.1	29.8
		28 M x 0	Covered	3.26	33.1	37.4	29.5
		28 M x 0	Open	3.16	32.1	36.0	31.9
3	1.5	28 M x 0	Inert	3.16	33.6	37.6	28.8
		28 M x 0	Covered	3.32	33.5	37.3	29.2
		28 M x 0	Open	2.97	32.0	35.0	33.0
4	2.0	28 M x 0	Inert	3.06	33.8	37.3	28.9
		28 M x 0	Covered	3.14	34.0	37.6	28.4
		28 M x 0	Open	3.04	33.7	36.7	29.6
5	2.5	28 M x 0	Inert	3.34	33.5	37.3	29.2
		28 M x 0	Covered	3.86	33.9	37.3	28.8
		28 M x 0	Open	3.40	33.4	37.5	29.1
6	3.0	28 M x 0	Inert	5.58	33.2	36.9	29.9
		28 M x 0	Covered	5.68	33.7	37.9	28.4
		28 M x 0	Open	5.33	32.3	36.5	31.2
7	4.0	28 M x 0	Inert	4.54	33.9	38.2	27.9
		28 M x 0	Covered	4.63	33.6	37.8	28.6
		28 M x 0	Open	4.24	32.7	36.9	30.4
		1/4" x 28 M	Open	6.61	35.3	45.7	18.9
9	6.0	28 M x 0	Inert	2.80	34.3	37.5	28.2
		28 M x 0	Covered	3.30	34.8	37.1	28.1
		28 M x 0	Open	3.00	33.9	38.3	27.8
11	9.0	28 M x 0	Inert	7.65	33.2	39.3	27.6
		28 M x 0	Covered	7.55	32.9	34.9	32.2
		28 M x 0	Open	7.68	31.6	37.9	30.5
12	11.0	28 M x 0	Inert	3.83	33.9	35.1	31.0
		28 M x 0	Covered	4.13	34.2	35.3	30.5
		28 M x 0	Open	5.57	33.7	33.6	33.8
13	13.0	28 M x 0	Inert	5.39	33.9	34.2	31.9
		28 M x 0	Covered	6.37	34.0	35.4	30.6
		28 M x 0	Open	7.50	33.7	32.9	33.4

Increments 1 and 7 contained about 19 to 20 percent ash, which is closer to the ash content of the research sample.

Table 13.6 - Proximate analyses of weathered samples of Pittsburgh No. 8 coal.

Incr.	Weathering Time (mths)	Size	Storage Mode	(ON DRY BASIS, %)			
				Moist. %	Vol. Matter	Fixed Carbon	Ash
1	0.5	28 M x 0	Inert	1.81	30.2	52.1	17.7
		28 M x 0	Covered	1.86	30.0	52.0	18.0
		28 M x 0	Open	1.86	30.2	51.9	17.9
		1/4" x 28 M	Inert	1.98	35.1	52.4	11.3
		1/4" x 28 M	Open	2.00	36.13	52.7	11.2
2	1.0	28 M x 0	Inert	1.82	30.3	53.0	16.7
		28 M x 0	Covered	1.90	30.4	51.9	17.7
		28 M x 0	Open	1.95	30.4	51.9	18.6
4	2.0	28 M x 0	Inert	1.82	30.9	51.1	18.0
		28 M x 0	Covered	1.98	30.6	51.4	18.0
		28 M x 0	Open	2.0	31.2	50.0	18.8
6	3.0	28 M x 0	Inert	2.00	30.6	51.5	17.9
		28 M x 0	Covered	2.16	30.7	51.3	18.0
		28 M x 0	Open	2.35	30.6	50.5	18.9
8	5.0	28 M x 0	Inert	1.96	30.5	51.4	18.1
		28 M x 0	Covered	2.26	30.5	51.7	17.7
		28 M x 0	Open	2.15	30.3	51.8	17.9
		1/4" x 28 M	Inert	2.4	35.5	53.5	11.0
		1/4" x 28 M	Open	2.4	36.2	53.0	10.8
9	6.0	28 M x 0	Inert	2.15	30.1	51.9	18.0
		28 M x 0	Covered	2.67	30.4	51.9	17.7
		28 M x 0	Open	2.62	30.4	51.9	17.7
10	7.0	28 M x 0	Inert	2.26	30.9	50.9	18.2
		28 M x 0	Covered	2.79	31.6	51.1	17.3
		28 M x 0	Open	2.57	31.9	51.7	16.3
11	9.0	28 M x 0	Inert	3.24	31.9	49.8	18.3
		28 M x 0	Covered	4.87	32.9	49.4	17.7
		28 M x 0	Open	5.06	34.4	51.1	14.5
		1/4" x 28 M	Open	2.58	37.7	52.1	10.2
12	11.0	28 M x 0	Inert	2.08	30.9	50.2	18.9
		28 M x 0	Covered	3.23	31.6	50.1	18.3
		28 M x 0	Open	4.08	33.2	51.2	15.7
13	13.0	28 M x 0	Inert	2.06	31.2	50.1	18.7
		28 M x 0	Covered	3.78	32.5	49.9	17.6
		28 M x 0	Open	3.25	33.0	52.7	14.3

Table 13.6 gives the proximate analysis of the Pittsburgh No. 8 weathered samples for the 28 mesh x 0 size fraction from Increments 1 to 13 (inert, covered and open modes) and for the 1/4-inch x 28-mesh size fraction for Increments 1, 8 and 11 for the inert and open modes of

Table 13.7 - Proximate analyses of weathered samples of Upper Freeport PA coal.

Incr.	Weathering Time (mths)	Size	Storage Mode	(ON DRY BASIS, %)			
				Moist. %	Vol. Matter	Fixed Carbon	Ash
1	0.5	28 M x 0	Inert	0.88	25.7	64.4	9.9
		28 M x 0	Covered	0.86	25.6	64.6	9.8
		28 M x 0	Open	0.91	25.9	64.5	9.6
		1/4" x 28 M	Inert	0.91	26.5	64.6	8.9
		1/4" x 28 M	Open	0.86	26.6	64.7	8.7
4	2.0	28 M x 0	Inert	0.94	25.3	65.1	9.6
		28 M x 0	Covered	1.06	25.2	64.8	10.0
		28 M x 0	Open	1.17	25.4	64.8	9.8
8	5.0	28 M x 0	Inert	1.00	25.8	64.5	9.7
		28 M x 0	Covered	0.99	25.8	64.5	9.7
		28 M x 0	Open	0.97	26.0	64.6	9.4
		1/4" x 28 M	Inert	0.94	27.3	63.7	9.1
		1/4" x 28 M	Open	0.82	26.7	63.4	9.9
10	7.0	28 M x 0	Inert	1.02	25.3	64.9	9.8
		28 M x 0	Covered	1.06	25.5	64.7	9.8
		28 M x 0	Open	1.20	26.0	65.3	8.7
11	9.0	28 M x 0	Inert	1.39	26.4	62.7	10.9
		28 M x 0	Covered	1.44	26.5	64.5	9.4
		28 M x 0	Open	1.42	27.5	65.0	7.5
		1/4" x 28 M	Inert	0.98	25.2	64.6	10.3
		1/4" x 28 M	Open	0.93	25.9	66.0	8.1
12	11.0	28 M x 0	Inert	1.27	26.9	62.6	10.5
		28 M x 0	Covered	1.31	26.3	62.4	11.3
		28 M x 0	Open	1.11	26.0	65.4	8.5
13	13.0	28 M x 0	Inert	1.34	26.1	64.1	9.8
		28 M x 0	Covered	1.30	26.1	62.8	11.2
		28 M x 0	Open	1.05	26.2	65.1	8.7

storage. The ash contents of coal stored under each of the three storage modes of storage are very close to each other. The higher ash content, 17 to 18%, of the 28-mesh x 0 size fraction as compared to 11% ash in the parent sample is again due to the concentration of mineral matter (clays) in the natural fines, although this effect is not as pronounced as that observed for Illinois No. 6 coal. The ash content of the 1/4-inch x 28-mesh fraction of Pittsburgh coal analyzed 11%, which is close to that of the research sample.

In the case of Upper Freeport PA coal (Table 13.7), the ash content of the weathered samples (about 10 percent) is slightly lower than that of the research sample (about 12 percent). No difference between the ash contents of the 28-mesh x 0 and the 1/4-inch x 28-mesh size fractions of the weathered samples was observed.

Even though weathering or oxidation is expected to release some of the volatile components from coal (4), no appreciable decrease in the volatile matter of the weathered samples was observed for any of the three coals irrespective of the weathering mode or the weathering time.

13.5 Size Consistency of the Weathered Samples

Weathering can occur either by mechanical (due to swelling and shrinking means) or by chemical (due to oxidation) action. While chemical weathering alters the composition of the material, mechanical weathering can contribute to size degradation. Since natural weathering is a combination of chemical and mechanical processes, the susceptibility of coal to weathering may be monitored by observing the size distribution of the weathered samples, provided that the effect of weathering exceeds typical experimental or sampling variability or the alteration of the material which occurs during handling. Sieve analyses of the 28-mesh x 0 and 1/4-inch x 28-mesh fractions of the samples weathered under inert (argon), covered and open modes were obtained for each of the increments of the three base coals.

Figure 13.6 is representative of the size distributions of the weathered samples of Illinois No. 6 coal, for Increment 7 (after 4 months of weathering). In the same plot, the size distribution of the standard grind of the research sample of Illinois No. 6 coal is also presented for comparison. One of the notable characteristics of the weathered samples is that the fines resulting from natural breakage and/or degradation are much coarser than those of the research sample ground in a rod mill even though the top sizes are nearly identical. The effect of the difference in the size distributions must be considered when comparing the flotation results of the weathered samples with the research samples.

The effect of the size degradation due to weathering can be elucidated from plots of the size distribution of the minus 28-mesh samples (Increments 1 through 7) stored under open

Figure 13.6 - Comparison of the particle size distribution of the rod milled research sample (95 percent passing 28 mesh) and Increment 7 of the weathered samples (inert, covered and open) for Illinois No. 6 coal.

conditions. As shown by the plots presented in the 1st Annual Report (8), even though there appears to be no consistent size degradation, there may be a tendency for the breakdown of coarser particles, which would contribute to an increase in the amount of fine particles. In addition, samples stored under the open environment may lose some very fine material due to rain and wind. In order to minimize the error introduced by such losses on the interpretation of the size distributions of the weathered samples, the data were recalculated to exclude the minus 150-mesh material. These results are plotted in Figure 13.7 as the weight percent of material in a given size interval (28 x 35 mesh, 48 x 65 mesh, 65 x 100 mesh and 100 x 150 mesh) as a function of weathering time. It appears that there is a decrease in the amount of 28 x 35-mesh material as weathering progresses but an increase in the amount of finer material. This may be ascribed to an increase in the friability of the weathered samples due to the propagation of cracks and cleats in the coal when the coal swells, resulting in a reduction of the mechanical strength of the coal and hence in the generation of fine material. The probability of this type of behavior should be more pronounced in the case of large particles.

Figure 13.7 - Effect of weathering time on the weight percent of material in different size intervals obtained by sieving the minus 28-mesh material of Illinois No. 6 coal weathered under open mode.

As a second step in understanding the effect of weathering on size degradation, sieve analyses of representative samples of the next larger size interval, that is 1/4-inch x 28-mesh material, were carried out. In Figure 13.8 the cumulative size distributions of the samples taken from Increments 1 (inert and open), 7 (open) and 9 (open) are presented. It is interesting to observe that in comparison to the inerted sample of the first increment (15 days of weathering), there is a progressive increase in the amount of fine particles as the weathering time progresses. Because the minus 28-mesh material in this sample had been sieved out by the sampling laboratory, this effect probably results from the increased friability of the coal, which manifests itself in the spalling off of material during sieving.

The size distributions of the Pittsburgh No. 8 and Upper Freeport PA coals weathered under similar conditions, but at different locations, were also determined. As an example, the size distributions of Pittsburgh No. 8 coal samples from Increments 1 and 8 weathered under the inert (argon), covered and open modes are presented in Figures 13.9. Unlike Illinois No. 6 coal, the size distributions of the weathered samples of Pittsburgh No. 8 coal are not appreciably different

Figure 13.8 - Effect of weathering on the size distribution of the 1/4-inch x 28-mesh Illinois No. 6 weathered coal samples (inert and open samples of Increment 1, and open samples of Increments 7 and 9).

Figure 13.9 - Comparison of the particle size distribution of rod mill ground research sample (95 percent passing 28 mesh) and Increment 8 of the weathered samples (inert, covered and open) for Pittsburgh No. 8 coal.

Figure 13.10 - Effect of weathering on the size distribution of the 1/4-inch x 28-mesh weathered samples (inert and open samples of Increment 1, and open samples of Increments 8 and 9) for Pittsburgh No. 8 coal.

from that of the corresponding standard grind sample, with the weathered samples being only slightly finer. Even in the case of the next size interval (1/4 inch x 28 mesh), no size degradation due to increased friability of the coal is observed with weathering time (Figure 13.10). The weight percent of the material in various size intervals (recalculated after excluding the minus 150-mesh material) was plotted as a function of the weathering time and since the plots were essentially horizontal, they are not shown here. Thus, it appears that Pittsburgh No. 8 coal does not physically degrade because of weathering during the period studied.

Similar particle size analyses were also carried out with the weathered samples of Upper Freeport PA coal. A comparison of the size distributions shows that the weathered samples are coarser than those of the corresponding research samples ground in the rod mill, as exemplified by the results for Increment 8 given in Figure 13.11. With respect to weathering degradation of Upper Freeport PA coal, there was no observable particle size change for material that had been weathered under open environment.

Figure 13.11 - Comparison of the particle size distribution of rod mill ground research sample (95 percent passing 28 mesh) and Increment 8 of the weathered samples (inert, covered and open) for Upper Freeport PA coal.

In summary, size degradation due to weathering affects Illinois No. 6 more than either Pittsburgh No. 8 or Upper Freeport PA coal. This effect, even in the extreme, is slight and is probably not as significant as the associated surface chemical effects due to weathering, as reflected in the flotation behavior of these coals.

13.6 Assessing the Hydrophobicity of Weathered Samples

The nature of the surface of different kinds of particles is of utmost importance in surface-based separation processes. This part of the report presents the results of assessing the effect of weathering on the hydrophobicity of coal particles by film flotation response, induction time measurements, Hallimond tube flotation and standard flotation behavior.

Because film flotation, induction time measurement and Hallimond tube flotation testing are all performed on small samples, the minus 28-mesh material was sieved in an inert atmosphere in a glove box to obtain a 100 x 150-mesh fraction of coal particles for these tests. The 100 x 150-mesh size fraction of each sample studied was first dried under vacuum at room

Figure 13.12 - Film flotation partition curves of as-received samples of Increment 3 for Illinois No. 6 weathered under inert, covered and open conditions.

temperature to reduce any effect of moisture on film flotation and then stored in glass bottles in an argon-filled glove box.

13.6.1 Film Flotation

Figure 13.12 shows the film flotation partition curves of Increment 3 for Illinois No. 6 coal stored under the inert, covered and open modes. It appears that both the inerted and covered samples have similar hydrophobic character, as compared with the sample weathered under open conditions. We were concerned with the effect of fine particles adhering to larger particles, the so-called slime coating phenomenon. Since in flotation processes, particulates are conditioned under turbulent conditions, slime coatings on particles may be removed from the solid surface during processing. However, there is no turbulence in film flotation, and hence, any small particles attached to the 100 x 150-mesh particles will probably remain attached. Therefore, in order to correlate the film flotation results more closely with actual laboratory flotation, the coal samples used for film flotation were deslimed by washing with running distilled water and then

Figure 13.13 - Film flotation partition curves of deslimed samples of Increment 3 for Illinois No. 6 coal samples weathered under inert, covered and open conditions.

dried under inert conditions to prevent further oxidation. The film flotation partition curves of the deslimed sample (Increment 3) of Illinois No. 6 coal given in Figure 13.13 indicate that the inert sample is more hydrophobic than either the covered or the open samples. A comparison of the proximate analysis of the 100 x 150 mesh sieved and deslimed samples indicate that the ash content of the sieved samples is higher than that of the deslimed sample. The higher ash content of the as-received sample is indicative of the potential for slime coating by the ash-forming minerals, with its effect on the apparent hydrophobicity of the coal particles as shown in Figure 13.12. Therefore, subsequent testing by the film flotation technique was carried out using deslimed samples.

Further delineation of the effects of the time of weathering on the hydrophobicity of the coal was obtained by performing additional film flotation tests with Illinois No. 6 weathered under open conditions. Figure 13.14 presents the film flotation partition curves of the samples weathered for 0.5, 1.5 and 4 months under open conditions. The shift in the partition curve is not as significant between Increment 1 (15 days of weathering) and Increment 3 (1.5 months of

Figure 13.14 - Film flotation partition curves of deslimed Illinois No. 6 coal samples weathered under open mode for 0.5, 1.5 and 4 months.

weathering) as between Increment 3 and Increment 7 (4 months of weathering). The coal surface became appreciably more hydrophilic, as reflected by the shift in the partition curve towards higher surface tensions of wetting.

Similar experiments were completed with Pittsburgh No. 8 and Upper Freeport PA coals weathered under open conditions for 0.5, 2 and 5 months, respectively. For both coals, the film flotation partition curves (Figures 13.15 and 13.16, respectively) show that the longer the coal samples were weathered, the more hydrophilic they became. The hydrophobicity of the coals did not change with only 2 months of weathering, but when the same coals were weathered for five months, there was a significant decrease in their hydrophobicities. This reduction in hydrophobicity is attributed to surface oxidation of these coals.

Table 13.8 summarizes the proximate analyses of the open-storage mode coals for various periods of weathering. Perhaps the best indication of the coal becoming more hydrophilic is the increase in contained moisture as the coals weathered, that is, became more oxidized. Film

Figure 13.15 - Film flotation partition curves of deslimed Pittsburgh No. 8 coal weathered under open mode for 0.5, 2 and 5 months.

Figure 13.16 - Film flotation partition curves of deslimed Upper Freeport PA coal weathered under open mode for 0.5, 2 and 5 months.

Table 13.8 - Proximate analysis of deslimed 100 x 150 mesh weathered samples stored in the open mode, their film flotation response, induction time for the three weathering modes, and the standard flotation of minus 28-mesh weathered coal.

Incr.	Weathering time, months	(Open) Moisture %	Dry Basis % (Open)			Film Flot. Yield* (Open)	Induction Time, ms			Std. Flot. Yield (Open)
			Fixed Carbon	Volatile Matter	Ash		Inert	Covered	Open	
Illinois No. 6										
1	0.5	1.61	37.12	36.31	26.57	56.3	28	60	60	56.7
3	1.5	0.75	34.42	34.64	30.94	51.2	50	175	200	56.3
7	4	1.72	37.54	35.37	27.09	45.1	--	--	250	36.4
9	6	0.72	37.69	36.51	25.80	--	--	--	250	29.1
11	9	5.17	34.05	35.83	30.12	22.3	--	--	1000	30.0
12	11	6.25	36.32	36.36	27.32	12.0	--	--	1520	23.6
13	13	5.33	34.94	36.14	28.94	17.5	825	--	1750	25.4
Pittsburgh No. 8										
1	0.5	0.51	50.91	33.09	16.00	85.6	80	--	40	55.3
4	2	0.55	49.03	32.22	18.75	83.7	--	--	40	55.5
8	5	0.68	50.60	32.57	16.83	70.9	--	--	40	51.8
10	7	0.68	53.55	33.85	12.60	--	--	--	100	12.8
11	9	2.55	56.55	34.17	9.28	89.5	--	--	375	10.0
12	11	2.47	56.31	34.75	8.94	92.8	--	--	500	19.0
13	13	2.51	55.55	34.51	9.94	91.6	--	--	200	29.7
Upper Freeport PA										
1	0.5	0.58	65.47	25.43	9.10	98.6	6.0	--	7.5	84.7
4	2	0.40	64.57	25.18	10.25	97.8	--	--	7.5	83.3
8	5	0.45	64.54	25.31	10.15	90.0	--	--	7.8	70.2
10	7	0.31	65.85	26.58	7.57	--	--	--	8.0	39.8
11	9	0.96	66.57	26.91	6.52	94.5	--	--	13.5	10.5
12	11	0.80	65.76	27.81	6.43	95.6	--	--	14.1	19.3
13	13	0.89	66.08	27.29	6.63	93.3	--	2.8	10.0	25.8

* Film flotation in 3-D water (72.7 mN/m)

flotation in water shows that Illinois No. 6 coal is more susceptible to oxidation than the other two coals. Similar experiments indicated that significant surface oxidation of the 100 x 150-mesh particles of Pittsburgh No. 8 coal and Upper Freeport PA coals did not occur during the course of the weathering tests. These measurements follow the general order of hydrophobicity of the three coals.

13.6.2 Induction Time Measurements of Weathered Samples

Because of the sensitivity of induction times (the time required for bubble-particle contact) to changes in hydrophobicity, this technique is ideally suited for characterizing small changes in hydrophobicity, such as those due to weathering. Detailed induction time measurements were conducted for all three base coals stored in the three modes, inert, covered and open, that had been weathered for various lengths of time. The experimental data are plotted such that the percentage of fruitful contacts is given as a function of the bubble-particle contact time. Figure 13.17 is such a plot for two different sample preparation procedures for Illinois No. 6 coal which had been weathered for 1.5 months under the open mode. As can be seen from these two plots, the induction time decreases when the sample is deslimed. It appears that the slime coating on the coal particles renders the particle surface more hydrophilic, thereby increasing the time for a bubble to make contact with the particle. Analogous behavior was found in the film flotation studies.

The proximate analysis results of the 100 x 150-mesh fraction of the selected increments of deslimed weathered samples used in both induction time measurements and film flotation studies are given in Table 13.8. From this table, it can be seen that the ash content of the 100 x 150-mesh deslimed fraction is lower than that of the entire minus 28-mesh fraction of the weathering samples. This supports the concept that slime coatings (fine ash particles adhering to the large coal particles) could be responsible for the difference in the induction times observed for the deslimed and undeslimed samples. Consequently, to correlate induction times with the results of laboratory flotation testing where a turbulent environment removes the slime coating from particles, the coal samples used for induction time measurements were then always deslimed.

The induction time distribution curves for Illinois No. 6, after 0.5 month of weathering (Increment 1), stored under inert, covered and open modes, is shown in Figure 13.18. Coal stored in the inert mode exhibits a shorter induction time than coal stored in either the covered or open modes. However, it appears that the difference between the induction times of coal stored in the covered and open modes is not as large as that between coal stored in the inert mode and that stored in the other two modes. The induction time results obtained for Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA weathered samples up to Increment 13

Figure 13.17 - Induction time curves indicating the percentage of fruitful contacts as a function of contact time in milliseconds for 100 x 150 M Illinois No. 6 weathered samples stored under open mode. Two different sample preparation procedures were used.

Figure 13.18 - Induction Time Curves indicating the percentage of fruitful contacts as a function of contact time for 100 x 150 M Illinois No. 6 weathered samples Incr. 1 stored under inert, covered and open modes.

Figure 13.19 - Induction Time Curve indicating the percentage of fruitful contacts as a function of contact time for 100 x 150 M Illinois No. 6 weathered sample Incr. 13 stored under open mode.

(13 months of weathering) are presented in Table 13.8. The induction time measurements clearly show that the hydrophobicity of the weathered samples is a function of the storage environment. Specifically, the hydrophobicity of the coals stored in inert mode is higher than that for coals stored in the covered and open modes, increasing in the following order: open < covered < inert.

At long contact times, a leveling off of the induction time curve occurs, as is evident from the plot (Figure 13.19) for the Illinois No. 6 sample (Increment 13) that had been weathered for 13 months in the open mode. This behavior may be related not only to the hydrophilic nature of the coal surface but also to deformation of the bubble as it is pushed onto the particle bed. This behavior is only observed for Illinois No. 6 coal for which the induction time is approaching the upper limit that the apparatus is capable of measuring.

13.7 Flotation Behavior of the Weathered Samples

The main emphasis in studying the surface characteristics of the weathered samples was to delineate their behavior in surface-based separation processes, and particularly in flotation.

The flotation response of coal is a function of its rank, particle size distribution, mineral matter content and the state of oxidation. To determine how weathering affects flotation under the more usual conditions, resulting from either the weathering mode or the weathering time, an extensive series of tests was carried out on all increments using the standard flotation test procedure. The collector and frother dosages used correspond to those used for the respective standard dry-ground (95 percent passing 28 mesh) research samples of the three base coals. For this study, the minus 28-mesh screened fraction from the weathered samples was used as the feed so as to have coal particles with their original weathered surface and not a fresh surface that have resulted from grinding. Flotation of coal samples weathered in the covered mode was conducted but the results were not considered to be meaningful because the integrity of the samples is questionable. During the extended weathering period, some of the plywood covers blew off and the drum filled with rain water. Thus, the chemistry of those systems was completely altered and the flotation results of those samples may have also been altered drastically.

Figure 13.20 presents the flotation test results for Illinois No. 6 coal that had been weathered for 13 months (Increment 13). The results clearly show that the samples stored under an inert environment gave higher yields than those that had been stored under either the open or covered conditions. The flotation yield of the "inerted" sample was found to decrease from 68.5 percent after 15 days of weathering (Increment 1) to about 48 percent after 9 months of weathering. This decrease is probably due to inefficiencies of purging and to oxygen trapped in the pores of the coal, which could not have been eliminated by purging the sample in a drum with inert gas. The corresponding yields for the samples that had been stored under open conditions decrease from about 56 percent to about 30 percent after thirteen months of weathering.

The tendency for Illinois No. 6 to oxidize is rather great, as shown by the decrease in flotability of coal under both the inert and open modes of storage. The daily temperatures of St. Louis, Missouri, located about 60 miles from the Illinois No. 6 weathering site, are shown in Figure 13.3. During the month of April, the maximum temperature began to climb above 60°F and continued climbing for the next month up into the 80's. Oxidation processes that led to the decrease in the hydrophobicity of Illinois No. 6 coal started to take place even at winter temperatures.

Figure 13.20 - Effect of weathering time on the flotation yield for Illinois No. 6 coal stored under inert and open modes.

The samples stored in the "inert" environment also showed signs of oxidation. The inerting process used for these samples was to purge the coal drum with three volumes (empty volume) of argon. Obviously, because of the decrease in flotation yield, some oxidation had taken place in the inerted sample, indicating that either this type of inerting process is not completely effective or air managed to diffuse into the closed drums. Moreover, because of the porous nature of coal, it may well be that entrapped air is responsible for the oxidation of the samples that had previously been purged with argon.

In comparison to the standard flotation yield obtained for the research sample (about 75 percent yield for the minus 28-mesh sample), the yields for the inerted weathered samples are less for the first increment (68.5 percent). This difference may be attributed to the higher ash content in the weathered samples (about 30 percent) in comparison to that of the research sample (17.3 percent) and the difference between the particle size distribution of the standard ground sample and the weathered samples. The effect of differences in the ash content on the flotation behavior can be minimized by comparing the combustible recoveries of the research sample (about 80

percent) with that of the first increment of the inerted weathered sample (about 83 percent). However, since the ash contents of the weathered samples collected at different times are reasonably consistent, the effect of weathering can be delineated by comparing the flotation response of the weathered samples to the inert sample of Increment 1. Such a comparison shows that weathering is, as expected, more obvious with the open sample than with the inert samples. The decrease in flotation yields can be attributed to an increase in hydrophilic oxygen functional groups present on the weathered coal surface as seen in the shifts of the film flotation partition curves (Figure 13.12) and the induction time measurements (Figure 13.17).

To determine the effect of the size at which the coal particles were weathered on their flotation response, samples of the 1/4-inch x 28-mesh weathered coal from Increments 1 and 7 (open and inert) and Increment 11 (open) of Illinois No. 6 were dry-ground under the same conditions as those of the standard grinding procedure to produce the usual minus 28-mesh flotation feed. The results of these experiments are also given in Figure 13.20. The flotation yield decreased from about 82 percent for the first increment (15 days of weathering) to about 72 percent for coal that had been weathered for 11 months. While the coarser material had undergone the same intensity of weathering, grinding produced sufficient new surface to nullify the increase in hydrophilicity of the original surface such that its flotation response was better than that of the finer weathered fractions. Prolonged weathering does appear to have had some influence on the flotation behavior of ground coarser material, though this effect is much less pronounced than for un-ground weathered minus 28-mesh coal.

Flotation yields of the weathered samples for Pittsburgh No. 8 coal are presented in Figure 13.21. As in the case of Illinois No. 6 coal, the inerted samples exhibit uniformly higher yields than the samples from the open modes. The effect of weathering time is more pronounced with the open samples than for the inerted samples. The yields of the inerted material decrease slightly from 62 percent for Increment 1 (0.5 month of weathering) to 55 percent after 13 months of weathering (Increment 13), whereas for the samples stored in the open, the yield decreases from 55 to 10.7 percent over a nine month period. Unlike Illinois No. 6 coal, Pittsburgh No. 8 did not oxidize during the initial months of weathering, corresponding to winter and early spring, but required the onset of warmer temperatures (see Figure 13.4) before the flotation yield began

Figure 13.21 - Effect of weathering time on the flotation yield for Pittsburgh No. 8 coal stored under inert and open modes.

to drop off significantly. With this coal, the oxidation of the hydrophobic material is activated by higher temperatures. The induction time measurements presented earlier highlight this effect as well. It is interesting to note that the oxidation of pyrite and of the coal matrix seem to be activated at different temperatures with that of pyrite being much lower than that of the coal.

As shown in Figure 13.21, the flotation yields of the weathered 1/4-inch x 28-mesh coal that had been ground to minus 28 mesh are always much higher than the unground 28-mesh x 0 fraction, even after thirteen months of weathering. The observed decrease in the flotation yield of the ground 1/4-inch x 28-mesh material can again be explained in terms of internal oxidation as in the case of Illinois No. 6 coal. The increase in the recovery of the coal stored in the open mode, Increment 13 is not fully understood. The flotation recovery, after grinding the sample of 1/4-inch x 28-mesh coal that had been stored under the inert mode, did not decrease at all with time.

Similar experiments were completed with Upper Freeport PA weathered samples. The flotation results of minus 28-mesh samples of Increments 1, 4, 8, 10, 11, 12 and 13 (inert and

Figure 13.22 - Effect of weathering time on the flotation yields for Upper Freeport PA coal stored under inert and open modes.

open) and ground 1/4-inch x 28-mesh samples of Increments 1, 8, 10 and 12 (inert and open) are presented in Figure 13.22. The effect of weathering on the flotability of the open samples is greater than that on the inerted samples. There is also a large decrease in the flotation yield of the open sample after April or May, again probably due to the onset of warmer temperatures (Figure 13.5). The flotation behavior of weathered Upper Freeport PA coal readily correlates with the temperature of the storage site.

A better comparison of the flotation behavior of the three coals weathered under similar conditions but at different locations might be obtained from plots of combustible matter recovered as a function of weathering time (Figure 13.23). For Illinois No. 6 coal, the combustible matter recovery decreases during the first five months of weathering and then stabilizes at about 30 percent during the later months. This leveling off may indicate the presence of some material that is somewhat inert and does not readily oxidize. In the case of both Pittsburgh No. 8 and Upper Freeport coals, the coal surface does not oxidize appreciably during the initial months (up to about 5 months) of weathering. Instead, a sudden decrease in

Figure 13.23 - Comparison of the effect of weathering on the combustible matter recovery for Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA coals weathered under open condition.

the recovery of combustible matter was observed only after about five months of weathering, corresponding to the onset of warm weather. Apparently the kinetics of oxidation are coal specific.

In summary, weathering has a definite effect on the flotabilities of all three coals tested. The susceptibility of each coal to oxidation seems to be strongly influenced by the mode and duration of storage, and the thermal conditions of the storage environment. As was expected, the weathering effect is more pronounced on the flotabilities of the minus 28-mesh fractions that had not been reground than on the coarser 1/4-inch x 28-mesh material. Grinding of the 1/4-inch x 28-mesh material apparently generates enough fresh unoxidized surfaces that better flotation results after grinding. However, since coal is porous, oxidation may progress into the internal surface of the coal after long exposure to the environment. This was reflected in slight decreases in the flotation yields when the 1/4-inch x 28-mesh material was ground and floated.

Figure 13.24 - Induction time, standard flotation and film flotation yields for Illinois No. 6 weathered samples stored under open mode as a function of weathering time.

13.8 Correlation of Wettability Criteria With Weathering

For correlating wettability criteria with the weathering behavior of the base coals, plots have been prepared to present induction time, film flotation response and standard flotation yield for each of the base coals weathered in the open mode. Figure 13.24 gives such plots for Illinois No. 6. Both the film flotation response and the standard flotation yield decrease, right from the beginning of the weathering process. Also, the hydrophobicity of the coal, as measured by the increase in induction time, decreases with weathering time, following the same general trend as that for the standard flotation tests and the film flotation studies. However, it is important to recognize that for the induction time and film flotation studies, only 100 x 150-mesh samples of coal particles were used, with neither collector nor frother present. In the standard flotation tests, not only was the entire minus 28-mesh fraction used, but also the hydrophobicity of the weathered samples was enhanced by the addition of both frother and collector. Even though the trend indicated by the three techniques is the same, the magnitude of the changes in the standard flotation yield do not necessarily correspond to similar changes in the induction

times due to the differences in the test procedures. For example, the rather sharp increase in induction time for Illinois No. 6 coal after 6 months of weathering (Figure 13.24) indicates a sharp decrease in the hydrophobicity of the coal surface, although the flotation yield of the standard flotation tests levels off at about this point. Since the natural oxidation of coal depends on particle size, it appears that for very hydrophilic coal samples the standard flotation test is not able to show small differences in hydrophobicity of the different coal particles due to the broad particle size range used (minus 28-mesh fraction) and the presence of the collector. Bubble-particle induction time measurements are, however, sensitive enough to show small changes in hydrophobicity.

Induction times, standard flotation and film flotation yields for Pittsburgh No. 8 weathered samples stored under the open mode as a function of weathering time are presented in Figure 13.25. As can be seen, the induction time and film flotation exhibit the same general trend as that of the standard flotation tests. However, these plots suggest that induction times are more sensitive to changes in the hydrophobicity of the coal surface than the film flotation response. In the case of the standard flotation tests, a sharp decrease in hydrophobicity of the coal surface was observed after 5 or 6 months of weathering, resulting in a minimum of a 10% yield at 9 months of weathering. Beyond nine months of weathering, the coal surface appears to become slightly more hydrophobic (remember that collector and frother are present in these experiments). Even though induction times follow the same trend, the sharp decrease in the hydrophobicity appears at 7 months of weathering, instead of 5, with a maximum in the induction time at 11 months of weathering followed by a slight increase in the hydrophobicity of the final coal increment (13 months of weathering). Although the trends are the same, the induction time and standard flotation test results are displaced by about two months. Again, these differences are probably due to differences in the experimental techniques of the two experimental procedures. The increase in hydrophobicity of the coal surface after 13 months of weathering, detected by both induction measurements and the standard flotation tests, seems to be real and must be related to the nature of the coal itself and the weathering conditions in the storage site.

The induction times, standard flotation and film flotation yields for weathered Upper Freeport PA coal samples stored under the open mode are plotted as a function of weathering

Figure 13.25 - Induction time, standard flotation and film flotation yields for Pittsburgh No. 8 weathered samples stored under open mode as a function of weathering time.

time in Figure 13.26. The same general trend as that found for Pittsburgh No. 8 weathered coal samples was also observed for Upper Freeport PA weathered samples, including the increase in hydrophobicity of the final weathered coal increment. The induction time measurements and standard flotation yields correlate quite well. The film flotation measurements, however, do not mirror the hydrophobicity of the samples very well.

Illinois No. 6 coal is susceptible to weathering right from the start, but both Pittsburgh No. 8 and Upper Freeport PA coals do not begin to weather (oxidize) until the temperature warms up in the spring. From the results given in Table 13.8 and Figures 13.24, 13.25 and 13.26, it can be seen that the hydrophobicity of weathered samples, even after open weathering, follows the following order:

$$\text{Illinois No. 6} < \text{Pittsburgh No. 8} < \text{Upper Freeport PA.}$$

13.9 pH of Weathered Coal Samples

The pH of the coal pulp during flotation provides a good deal of information on the state of oxidation of the coal and pyrite. The pH of the pulp is influenced by a number of oxidation-

Figure 13.26 - Induction time, standard flotation and film flotation yields for Upper Freeport PA weathered samples stored under open mode as a function of weathering time.

related phenomena. Generation of oxygen functional groups on the carbonaceous material occurs when coal is oxidized, while sulfuric acid is produced when pyrite degrades. Because of the relative strengths of the organic acid functional groups present due to coal oxidation and that of sulfuric acid, the pH of the pulp will be influenced more by the oxidation state of the pyrite than by that of the coal. However, the presence of basic cations in the mineral matter can partially offset the influence of oxidation on the pH by neutralizing some of the acid produced. The pH of the minus 28-mesh weathered samples (open mode) is shown in Figure 13.27 for the three base coals. This figure shows that the pH of the Illinois No. 6 coal was the highest of the three and did not start to drop until the fourth or fifth month of weathering which corresponds to the onset of spring (and warmer temperatures). The high pH associated with this coal probably is a result of the presence of calcite. The lack of change in the pH after five months of weathering does not signify that oxidation has not taken place, but just that any acid produced by oxidation was compensated for by the calcite present in these coals. Indeed, the decrease in

Figure 13.27 - Flotation pulp pH of the 28 mesh x 0 weathered samples as a function of weathering time.

flotation yield of Illinois No. 6 weathered coal (Figure 13.20) after short weathering times suggests that oxidation had taken place.

The pH of the other two coals both started at about 5 with the Upper Freeport dropping rapidly with time initially and then levelling off at long times, whereas the Pittsburgh No. 8 sample began to decrease less rapidly but dropped steadily for a long period of time. Both of these coals contain essentially no calcite so that any acid produced by oxidation immediately influenced the pulp pH. Additionally, pulp pH measurements and flotation evaluations show that the oxidation of pyrite alone is not sufficient to decrease the flotation yield since that is much more dependent on the oxidation of the coal itself. Both oxidation reactions are temperature controlled and the activation energy for the reaction is coal-dependent. In conclusion, the pH of the pulp is influenced most dramatically by the oxidation of pyrite and therefore the pH decreases of the coal slurries is related to the rate at which the pyrite oxidized. The rate of the oxidation reaction is a function of temperature and moisture, which is site-dependent, and the activation energy of the reaction, which is sample-dependent.

13.10 References

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14.0 DEGRADATION OF STORED COAL SAMPLES

During the latter half of the second year of this research effort, a study of an experimental procedure to assess the flotation of coal, the tree analysis of Pratten, Bensley and Nicol (1), was undertaken in order to compare the results with those obtained from the more common Release Analysis procedure (2). At the onset of this work it was felt that the two experimental procedures should give the same results. However, after the first tree analysis was completed, the measured flotability was considerably less than that shown by the release analysis that had been completed six months earlier. The reasons for this inconsistency were thoroughly investigated and is the genesis of this discussion on the degradation of stored coal samples.

Just prior to the start of this study, the original drums of coal with which the majority of the prior experimental work had been conducted, were depleted and new drums of the samples were used. The new drums had been filled at the same time, with the same coal, and inerted just as the original drums of coal had been. The results presented here put into question the integrity of coal samples that have been stored, albeit in "inerted drums", for long periods of time.

14.1 Tree Analysis of Base Coals

The flotability curves of the three base coals, Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA, were obtained using the tree analysis procedure as outlined by Pratten et al. (1). Figure 14.1 illustrates the flotation scheme for the tree analysis procedure used to determine the flotability curves of the three base coals. The aims of these tests were two-fold: i) to compare the flotability results obtained with those from the release analysis procedure and ii) to establish the effect of two years of storage under argon on the flotability of the three base coals. The tree analysis test methodology was applied to a set of samples taken from different drums (Illinois No. 6 B1-04, Pittsburgh No. 8 B2-04 and Upper Freeport PA B3-04) than had been used for the release analysis procedure tests (B1-03, B2-03 and B3-03). Because both procedures attempt to fractionate the coal based on the relative hydrophobicity of the coal, it was thought at the onset of this work that the results from the two techniques would be nearly identical. The flotation schemes for the release analysis procedure were presented in Chapter 7 of this report.

Figure 14.2 - Release analysis and tree analysis results showing ash rejection as a function of CMR for Illinois No. 6 coal.

Figure 14.3 - Release analysis and tree analysis showing pyritic sulfur rejection as a function of CMR for Illinois No. 6 coal.

Table 14.1 - Flotation results for Illinois No. 6 coal using the Tree Analysis Procedure.
Standard flotation conditions, 8.21 lb/T dodecane and 2.63 lb/T were used.
The flotation pH was 3.7.

Yield %	Cum. %	PRODUCT ANALYSIS				REJECTION		EI _H
		1-Yield %	CMR %	Ash %	Pyr. S %	Ash %	Pyr. S %	
5.4	5.4	94.6	6.0	4.3	0.59	98.3	98.4	4.4
21.1	26.5	73.5	29.4	4.7	0.79	91.3	90.1	13.5
3.5	30.0	70.0	33.2	6.5	0.93	89.6	88.4	21.6
12.9	42.9	57.1	47.0	8.1	1.15	82.1	81.0	28.0
0.9	48.8	51.2	47.9	8.1	1.19	81.6	80.5	28.4
9.4	53.2	46.8	57.9	8.5	1.24	75.8	74.7	32.6
27.4	80.6	19.4	86.8	9.1	1.39	57.9	55.7	42.5
0.4	81.0	19.0	87.2	10.9	1.57	57.6	55.4	42.6
0.7	81.7	18.3	87.9	19.1	2.33	56.7	54.6	42.4
0.4	82.1	17.9	88.2	21.0	2.73	56.0	54.0	42.2
0.1	82.2	17.8	88.4	28.0	3.61	55.8	53.8	42.1
6.6	88.8	11.2	94.0	26.0	3.80	43.4	41.2	35.2
1.8	90.6	9.4	95.4	34.5	4.02	39.0	37.6	33.0
3.2	93.8	6.2	97.9	31.2	4.07	31.9	31.2	29.1
0.4	94.2	5.8	98.1	58.4	6.42	30.1	29.8	27.9
5.8	100.0	0.0	100.0	72.3	10.28	--	--	--
FEED				13.9	2.00			

had taken place was the flotation pH of the two samples, pH 7 for B1-03 coal (release analysis) and pH 3.7 for B1-04 coal (tree analysis). This pH change is indicative of pyrite oxidation and does not preclude the oxidation of the carbonaceous material. The decrease in the flotability of the coal may be due to decreased hydrophobicity of oxidized coal and/or interaction of soluble iron complexes with the coal and mineral matter.

Even though an increase in the dosage of collector and frother slightly moves the flotability curves for the B1-04 sample upward, the curves obtained with this sample are significantly lower than those obtained in the original release analysis procedure.

For identical samples and the same collector and frother dosage, the tree analysis procedure gives slightly better results at high yield (low 100 - Yield) than those obtained in the release analysis. This difference in this part of the flotability curves is again probably related to the number of flotation stages in each of the methods.

For Pittsburgh No. 8 coal, the results obtained by using the two tree analysis procedures are presented in Table 14.4. The ash and pyritic sulfur rejection as a function of (100 - Yield) are presented in Figures 14.4 and 14.5, respectively. As can be seen from these curves, the

Figure 14.4 - Release analysis and tree analysis results showing ash rejection as a function of CMR for Pittsburgh No. 8 coal.

Figure 14.5 - Release analysis and tree analysis results showing pyritic sulfur rejection as a function of CMR for Pittsburgh No. 8 coal.

Table 14.2 - Flotation results for Illinois No. 6 coal using Tree Analysis Procedure. Standard flotation conditions, 7.44 lb/T dodecane and 1.76 lb/T MIBC were used. The flotation pH was 3.5.

Yield %	Cum. %	PRODUCT ANALYSIS				REJECTION		EI _H
		1-Yield %	CMR %	Ash %	Pyr. S %	Ash %	Pyr. S %	
0.7	0.7	99.3	0.7	3.5	0.46	99.8	99.8	0.6
0.8	1.5	98.5	1.6	4.8	0.62	99.6	99.5	1.2
2.0	3.5	96.5	3.9	4.7	0.63	98.9	98.8	2.7
7.2	10.7	89.3	11.9	4.3	0.69	96.6	95.9	7.7
0.7	11.4	88.6	12.6	7.6	0.70	96.3	95.6	8.2
14.1	25.5	74.5	28.0	6.3	0.81	89.9	88.9	16.9
14.9	40.5	59.5	44.1	7.4	0.84	82.0	81.5	25.6
14.4	54.9	45.1	59.8	6.4	0.86	75.4	74.2	34.0
5.1	60.0	40.0	65.4	6.5	0.93	73.0	71.5	36.9
1.4	61.4	38.6	66.8	8.4	0.96	72.2	70.7	37.5
0.4	61.8	38.2	67.2	13.3	1.46	71.8	70.3	37.6
10.0	71.8	28.2	77.2	13.8	1.65	61.8	60.6	37.9
8.2	80.0	20.0	85.3	15.1	1.83	53.0	51.9	37.2
8.6	88.6	11.4	93.6	16.3	1.97	42.9	42.0	35.6
2.5	91.1	8.9	95.9	22.4	2.32	38.9	38.6	34.5
8.9	100.0	0.0	100.0	60.3	7.35	--	--	--
FEED				13.9	1.71			

flotation results using tree analysis indicate a slight shift downward in the flotability curves with respect to those obtained from release analysis. Since the results obtained for Illinois No. 6 (and Upper Freeport PA coal) showed that the flotability curves should be nearly identical, this difference is probably a result of degradation of the coal surface.

Table 14.3 - Flotation results for Illinois No. 6 coal using Release Analysis Procedure. Standard flotation conditions, 7.83 lb/T dodecane and 1.54 lb/T MIBC were used. The flotation pH was 3.8.

Yield %	Cum. %	PRODUCT ANALYSIS				REJECTION		EI _H
		1-Yield %	CMR %	Ash %	Pyr. S %	Ash %	Pyr. S %	
4.9	4.9	95.1	5.4	4.6	0.64	98.4	98.2	3.6
17.6	22.5	77.5	24.7	5.9	0.74	90.9	90.9	15.6
19.6	42.1	57.9	46.2	5.7	0.76	82.9	82.5	28.7
1.1	43.2	56.8	47.3	5.5	0.77	82.5	82.1	29.4
5.7	48.9	51.1	53.5	5.7	0.79	81.1	79.5	33.1
16.6	65.5	34.5	71.2	8.5	1.04	69.9	69.8	41.0
10.2	75.7	24.3	81.4	14.4	1.63	59.3	60.4	41.8
5.0	80.7	19.3	85.4	30.1	3.27	48.5	51.3	36.7
19.3	100.0	--	100.0	34.8	4.72	--	--	--
FEED				13.9	1.78			

Table 14.4 - Flotation results for Pittsburgh No. 8 coal using the Tree Analysis Procedure. Standard flotation conditions, 2.52 lb/T dodecane and 0.6 lb/T MIBC were used. The flotation pH was 3.7.

Yield %	Cum. %	PRODUCT ANALYSIS				REJECTION		El _H
		1-Yield %	CMR %	Ash %	Pyr. S %	Ash %	Pyr. S %	
0.8	0.8	99.2	0.9	1.9	0.35	99.9	99.9	0.8
5.7	6.5	93.5	7.2	2.0	0.41	98.8	98.8	6.0
1.8	8.4	91.6	9.2	2.5	0.46	98.4	98.5	7.6
1.6	10.0	90.0	10.9	2.8	0.51	97.9	98.1	9.0
8.6	18.5	81.5	20.2	3.0	0.54	95.5	96.0	16.2
13.3	31.8	68.2	34.5	3.8	0.75	90.8	91.6	26.1
8.1	39.9	60.1	43.1	4.0	0.81	87.7	88.7	31.8
13.9	53.8	46.2	58.1	4.1	0.83	82.3	83.5	41.6
1.1	54.9	45.1	59.2	8.2	1.53	81.5	82.8	42.0
1.5	56.3	43.7	60.7	8.6	1.77	80.3	81.7	42.3
1.9	58.2	41.8	62.5	9.2	1.99	78.7	80.0	42.6
8.9	67.1	32.9	71.5	10.4	2.18	69.9	71.4	42.9
14.1	81.2	18.8	85.6	10.8	2.37	55.5	56.5	42.1
6.5	87.8	12.2	92.0	11.6	2.47	48.4	49.3	41.3
11.8	99.5	0.5	99.8	40.9	8.96	2.8	2.4	2.2
0.5	100.0	0.0	100.0	60.6	11.21	--	--	--
FEED				10.6	2.25			

As was the case with Illinois No. 6, the pH of the second drum of coal, B2-04, from which the tree analyses samples were taken, was lower (pH 3.7) than that of the primary drum, B2-03, from which the release analysis sample was taken (pH 4.2). This drop again suggests that oxidation of the coal sample has taken place.

The flotation results using the tree analysis procedure for Upper Freeport PA are presented in Table 14.5. Figures 14.6 and 14.7 show the ash and pyritic sulfur rejection as a function of unfloated material (100 - Yield), respectively. The washability and release analysis results are also shown for comparison. As was expected, because this coal has the lowest pyritic sulfur content and the highest hydrophobicity of the three base coals, there is little difference between the locus of points comprising the tree analysis curve with that of the release analysis curve. Moreover, the natural pH of the flotation pulp (pH 3.5) was identical for the two samples. Therefore, it seems the Upper Freeport PA sample has not oxidized appreciably during storage.

The results from the tree analysis procedure for Upper Freeport PA coal are slightly better at lower yields than those obtained from the release analysis procedure. This may be related to the fact that the release analysis procedure was performed using only three flotation stages,

Figure 14.6 - Release analysis and tree analysis results showing ash rejection as a function of CMR for Upper Freeport PA coal.

Figure 14.7 - Release analysis and tree analysis results showing pyritic sulfur rejection as a function of CMR for Upper Freeport PA coal.

Table 14.5 - Flotation results for Upper Freeport PA coal using the Tree Analysis Procedure. Standard flotation conditions, 0.57 lb/T dodecane and 0.36 lb/T MIBC were used. The flotation pH was 3.5.

Yield %	Cum. %	PRODUCT ANALYSIS				REJECTION		EI _H
		1-Yield %	CMR %	Ash %	Pyr. S %	Ash %	Pyr. S %	
13.4	13.4	86.6	15.0	2.0	0.06	97.9	99.3	14.3
9.7	23.1	76.9	25.7	3.0	0.10	95.5	98.5	24.2
9.6	32.7	67.3	36.4	2.6	0.11	93.5	97.6	33.9
4.9	37.6	62.4	41.8	3.0	0.14	92.2	96.9	38.7
2.7	40.3	59.7	44.8	3.3	0.20	91.4	96.5	41.3
8.3	48.6	51.4	53.7	5.9	0.32	87.4	94.1	47.8
6.6	55.2	44.8	60.7	6.2	0.33	84.1	92.3	53.0
3.9	59.1	40.9	64.8	6.3	0.37	82.1	91.0	55.8
2.8	61.9	38.1	67.8	7.5	0.42	80.4	90.0	57.8
2.2	64.1	35.9	70.1	7.9	0.51	79.0	89.0	59.1
1.3	65.4	34.6	71.4	10.9	0.81	77.8	88.1	59.5
8.0	73.4	26.6	79.1	15.9	1.34	67.4	78.7	57.8
10.9	84.3	15.7	89.5	16.7	1.39	52.6	65.5	55.0
3.0	87.3	12.7	92.3	17.1	1.90	48.4	60.6	52.9
1.8	89.1	10.9	93.8	26.9	2.48	44.5	56.7	50.5
10.9	100.0	0.0	100.0	50.1	5.97	--	--	--
FEED				12.3	1.15			

whereas the tree analysis procedure comprised four flotation stages. Thus, the products from the tree analysis were submitted to more cleaning steps than those of the release analysis tests.

14.2 Effect of Storage Time on the Behavior of Illinois No. 6 Coal

As shown before, the maximum separation efficiencies of Illinois No. 6 coal stored in Drum 3 (B1-03) and in Drum 4 (B1-04) obtained from the release and tree analyses was 65 for sample B1-03 and 42 for sample B1-04 when using dodecane and MIBC. The difference between these samples is larger than the experimental error and therefore must be attributed to changes in coal properties. In order to investigate this further, the data in Tables 14.6 and 14.7 were compiled in order to compare the results of standard flotation tests and the natural pH of Illinois No. 6 coal stored in Drum 3 (B1-03) with that stored in Drum 4 (B1-04). The letters A, B and C in the sample identification indicate different subplots of the samples contained within Drum 4. The results presented in Tables 14.6 and 14.7 clearly show that both the initial pH and the final separation efficiency of coal stored in Drum 4 are lower than that stored in Drum 3. This may indicate that oxidation of both coal and pyrite takes place even though "inert" conditions

Table 14.6 - Comparison of flotation behavior of Illinois No. 6 coal (200 mesh wet grind) in drum 3 (B1-03) and drum 4 (B1-04) using standard flotation conditions (4.92 lb/T dodecane and 0.98 lb/T MIBC.)

Sample ID/ Flotation Date	Initial pH/ Final pH	Flot.time, minutes	CMR %	REJECTION		EI
				Ash %	Pyr. S %	
438-B1-03-W200D/ June 29, 1990	6.50/7.20	0.5	27.1	87.1	88.6	15.7
		1.0	47.3	78.1	80.7	28.1
		3.0	79.5	61.3	64.7	44.2
		5.0	84.7	57.3	60.8	45.5
439-B1-03-W200D/ June 29, 1990	6.70/7.40	0.5	26.1	87.3	88.2	14.3
		1.0	47.7	76.9	78.2	26.0
		3.0	79.5	59.9	61.2	40.7
		5.0	84.3	56.5	57.8	42.1
460-B1-04-W200B/ August 31, 1990	4.40/4.63*	0.5	36.2	80.4	80.4	16.7
		1.0	55.9	70.1	69.5	25.4
		3.0	80.2	55.0	53.7	33.9
		5.0	86.7	48.9	47.4	34.0
471-B1-04-W200/ October 25, 1990	4.01/4.07	0.25	14.1	91.9	93.2	7.3
		0.5	28.8	84.1	86.2	15.1
		1.0	46.6	74.4	78.0	24.6
		2.0	60.4	66.6	71.4	31.8
		5.0	75.4	56.3	61.6	37.0

* Frother conditioning time is one minute.

were maintained for both coal samples. The reason for this significant change in pH and the flotation behavior of sample B1-04 is not clear at present. However, one explanation may be that the oxygen in the pores of coal is not completely displaced by argon when purging the samples. Over time, the remaining oxygen will oxidize the coal and pyrite surfaces, resulting in a decrease in both the flotation separation efficiency and the pH of the coal slurry. This effect was seen for Pittsburgh No. 8 coal in the initial months of the project.

In summary, differences in oxidation behavior of the coals are coal-specific and dictate how fast the samples will degrade even when stored in an "inert" environment. These results strongly suggest that in any research effort extending over a period of time, it is absolutely necessary to regularly collect newly mined samples from a source that is as close to the original mine location as possible. Without this regeneration of coal samples, the data obtained from the latter part of the research project must be viewed with caution.

Table 14.7 - Comparison of the average initial flotation pH of Illinois No. 6 coal samples stored in Drum 3 (B1-03) and Drum 4 (B1-04).

<u>Coal Sample ID</u>	<u>Initial pH</u>	<u>Working Period</u>
B1-03	6.2	3/89 - 7/90
B1-04A	3.8	8/90 - 1/91
B1-04B	3.9	1/91 - 3/91
B1-04C	3.5	3/91 - present

14.3 References

1. S.J. Pratten, C.N. Bensley and S.K. Nicol, "An Evaluation of the Flotation Response of Coals", International Journal of Mineral Processing, Vol. 27, pp. 243-262, (1989).
2. C.C. Dell, "Determination of Froth-flotation-washability Data", Journal of the Institute of Fuel, Vol. 30, pp. 523-526, (1957).

15.0 SUMMARY AND CONCLUSIONS

15.1 Project Team and Objective

This final report for the DOE project entitled "Coal Surface Control for Advanced Fine Coal Flotation" contains a comprehensive summary of work completed during the contract period, October 1, 1988 through December 31, 1991. The project team consisted of research and engineering groups at the University of California at Berkeley, Columbia University, the University of Utah, and Praxis Engineers, Inc., with the University of California acting as the prime contractor with DOE.

The initial goal of the research project was to develop methods of coal surface control in advanced froth flotation to achieve 90% pyritic sulfur rejection, while operating at Btu recoveries above 90% based on run-of-mine quality coal. Moreover, such technology is to concomitantly reduce the ash content significantly (to six percent or less) to provide a high-quality fuel to the boiler. The sections that follow summarize the findings reported in each chapter of this report.

15.2 Coal Procurement

Upon the award of the contract, the initial effort was focused on the collection of samples from six coal seams, three designated as base coals and three as additional coals, to provide samples for research work, coal weathering studies and washability testing. The three base coals, which were selected as representative of important high-pyritic-sulfur bituminous coals, were Illinois No. 6 from Randolph County, Illinois; Pittsburgh No. 8 from Belmont County, Ohio; and Upper Freeport PA from Indiana County, Pennsylvania. The additional coal samples consisted of two eastern high-sulfur bituminous, Kentucky No. 9 from Hopkins County, Kentucky, and Upper Freeport WV from Grant County, West Virginia; and one western low-sulfur sub-bituminous coal, Wyodak from Campbell County, Wyoming. In order to ensure uniformity of samples for the duration of this project, as well as for the selective oil agglomeration project being conducted at the University of Pittsburgh, DOE consolidated the sample procurement task into a single effort referred to as Coal Procurement and Weathering (Task 2). This aspect of the

program was carried out by the team from Praxis Engineers. About 23 tons of each base coal and five tons of each additional coal were collected from six operating open-pit mines, taking care to remove overburden and floor shale material. Thus, the coal samples were equivalent to a washed raw coal. Each base coal sample was reduced to a topsize of 4 to 6 inches, homogenized and split into two parts, one for use in the weathering studies and the other for research and washability studies. The research/washability sample was split into 400-lb lots and packed into 55 gallon drums, inerted with argon and then distributed. Approximately 9 tons of coal (research and weathering samples) were shipped to the University of California, 6 tons to the University of Pittsburgh, and 6 tons of coal to commercial testing laboratories for washability testing. Over 42 tons of coal were used to set up the weathering studies of the three base coals at the three mine sites. Another 21 tons were stored in inerted drums as a reserve sample for future use on this project or on other projects funded by DOE.

15.3 Bulk Characterization of the Coal Samples

The three base coals were characterized by a variety of different techniques. Bulk characterization studies included the determination of chemical composition by proximate, ultimate and sulfur-forms analyses. The ash content of Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA coals is 17.5%, 11.8% and 12.0%, respectively, and their pyritic sulfur content is 2.50%, 2.82%, and 1.62%, respectively. The pyritic sulfur as a percentage of the total sulfur is the highest in Upper Freeport PA (69 wt%), followed by Pittsburgh No. 8 (60 wt%) and Illinois No. 6 coal (48 wt%). This implies that the application of physical separation techniques, such as froth flotation and selective agglomeration, should potentially be most effective for lowering the sulfur content of Upper Freeport PA coal, followed by Pittsburgh No. 8 and Illinois No. 6 coals, assuming equal liberation and rejection of the pyritic sulfur.

Examination of the ultimate (elemental) analysis results shows that the bulk carbon-to-oxygen ratio (number in the parentheses) follows the order:

Upper Freeport PA (19.9) > Pittsburgh No. 8 (11.1) > Illinois No. 6 (10.5).

The dry mineral matter free (dmmf) carbon content of the three coals is:

Upper Freeport PA (85.9% dmmf C) > Pittsburgh No. 8 (80.5%) > Illinois No. 6 (77.3%).

This order agrees with the hydrophobicity and the flotability of the three base coals.

Qualitative analysis of the mineralogical content of the ash minerals contained in the coal was conducted using diffuse reflectance infrared Fourier transform (DRIFT) spectroscopy. It was concluded that all three samples contain kaolinite and quartz. In addition, peaks characteristic of calcite were present in the spectra of the ash of Illinois No. 6 coal but were not present in the other two base coals. Neutron activation analysis shows that the Illinois No. 6 sample contains 0.7% calcium whereas Pittsburgh No. 8 and Upper Freeport PA samples contain less than 0.1%.

As part of the bulk characterization studies, photomicrographic studies using SEM/EDXRF (scanning electron microscopy/energy dispersive x-ray fluorescence) and optical image analyses were also conducted to determine pyrite and non-pyritic mineral association in the coal. The SEM/EDXRF study consisted of a qualitative analysis of the distribution of pyrite and non-pyritic mineral matter in narrow particle size ranges, finer than 28 mesh. The results of this study indicated that there was almost no liberated pyrite in the 28-mesh samples of Illinois No. 6 or Pittsburgh No. 8 coal, whereas free particles of pyrite did exist in the 200-mesh samples. Optical image analysis was carried out to quantitatively analyze the distribution of locked, semi-locked and free pyrite grains in different size fractions within the minus 200-mesh grind. This analysis showed that the majority of the pyrite present in Illinois No. 6 and Upper Freeport PA coal was liberated, whereas that in the Pittsburgh No. 8 coal was not. Petrographic analysis was also conducted to determine the maceral composition of the three base coals. In all three cases, vitrinite was the major component. About 11 to 15 percent inertinite was present in all three coals and 10% liptinite in Pittsburgh No. 8 and Illinois No. 6 coal. In the case of Upper Freeport PA coal, only 0.2% liptinite was present.

15.4 Washability Studies

Washability studies were conducted on the original 4-inch topsize samples as well as on representative samples that had been comminuted to pass 1-1/2 inches, 1/8 inch, 28 mesh and 200 mesh. The results show that when the top size of the crushed material is 28 mesh and above, none of the three base coals exhibits significant pyrite liberation, and the pyritic sulfur rejection

is the least for Pittsburgh No. 8 coal at 90% Btu recovery, indicating a greater degree of pyrite dissemination throughout the coal in this material. However, all of the samples show that more than 90% of the pyritic sulfur can be rejected by sink-float techniques at specific gravity of 1.6 if they are ground to minus 200 mesh (74 microns). Similar behavior was observed in the washability tests of the additional coals (Upper Freeport WV, Kentucky No. 9 and Wyodak).

Because liberation occurs at fine sizes and in view of heightened interest in fine coal beneficiation, more detailed washability studies of the three base coals ground to finer sizes were undertaken. Reserve samples of the coals, which had been used in the original washability studies and had been stored in sealed "inerted" 55-gallon drums, were comminuted to minus 100, 200, 325 and 400 mesh for these tests. For all three base coals, there was enough liberation of pyrite at the 100-mesh grind size to achieve 90% pyritic sulfur rejection with 90% Btu recovery by sink-float methods. There were only slight differences in the washability results between the coals comminuted to 100, 200, 325 and 400-mesh grind sizes. These results indicate that by grinding the coals to minus 100 mesh (150 microns) it is possible to achieve 90% pyritic sulfur rejection at 90% Btu recovery. However, it is important to note that these separations are based only on the specific gravity of the particles and provide an indication of mineral liberation, but not on pyrite separability by processes that utilize other phenomena, such as surface behavior.

15.5 Wettability and Surface Characterization

The second part of the general characterization studies was directed at delineating the nature of surface properties and included analysis of both macroscopic and microscopic properties. Macroscopic properties evaluated included the wettability behavior (by film flotation, contact angle and induction time measurements) and surface charge characteristics (as given by zeta potential measurements). Microscopic characterization of the coal included surface elemental composition analysis by ESCA (Electron Spectroscopy for Chemical Analysis) and determination of organic functional groups by DRIFT.

Results of the wettability analyses using film flotation, contact angle measurements and induction time measurements indicate that the hydrophobicity of the coal follows the order:

Upper Freeport PA > Pittsburgh No. 8 > Illinois No. 6.

Zeta potential measurements show that the isoelectric point (IEP), that is the condition (pH) at which the zeta potential is reversed, of the three base coals decreases in the following order:

Upper Freeport PA (pH 5.5) > Pittsburgh No. 8 (pH 3.5) > Illinois No. 6 (pH 2.5).

The order of both hydrophobicity and the IEP of the three base coals is the same as that of the bulk carbon-to-oxygen ratio and the dmmf carbon content of the three coals.

The surface area measurements using carbon dioxide as an adsorbate gas show that Illinois No. 6 coal has the highest specific surface area ($172 \text{ m}^2/\text{g}$), followed by Pittsburgh No. 8 ($150 \text{ m}^2/\text{g}$) and Upper Freeport PA ($147 \text{ m}^2/\text{g}$). The surface elemental composition determined by ESCA and the oxygen functional groups determined by DRIFT of the three base coals were not very conclusive.

15.6 Standard Grinding Tests

For preparing the feed for the flotation experiments, the coal was first crushed to minus 1/4-inch under inert conditions, and then ground in a stainless steel laboratory rod mill to produce material that was either 95% minus 28-mesh or 95% minus 200-mesh. For each grind, 500 grams of coal was charged to the mill so that four 125-gram samples could be produced to be used in each series of flotation tests. Stainless steel rods rather than balls were used as the grinding media because the product size distribution produced by rod action more closely simulates the product produced by an industrial ball mill/classifier circuit. For each coal and each grind, whether wet or dry, a standard grinding condition was developed, including rod charge and grind time, to prepare flotation feed with a size distribution of 95% passing either 28 mesh or 200 mesh. Identical mills and rods were located at each research site so that identical test feeds could be prepared at each site. To increase the time (for better control) required for grinding the coal to 28 mesh, the mill was charged with only 24 rods, whereas for grinding to 200 mesh, the number of rods was increased to 42. From the standard grinding tests, the relative grindability of the three base coals is in the following order:

Dry: Upper Freeport PA > Pittsburgh No. 8 > Illinois No. 6

Wet: Upper Freeport PA > Pittsburgh No. 8 $\approx$ Illinois No. 6

The results also show clearly that wet grinding is more energy efficient (by nearly a factor two) than dry grinding for all three coals.

15.7 Standard Flotation Tests

A standard flotation test was developed in order to evaluate the effects of the grinding environment and the addition of surface modifying reagents on the flotation response of the coals. Pure dodecane was selected as the collector and MIBC (methylisobutylcarbinol) as the frother. For these standard tests, a 2-liter Denver laboratory flotation machine was used, with the cell configured according to the DOE (PETC) design. The standard test procedures, that is, the physical and operating variables developed for each coal are identical to each other except for the dosage of collector and frother added to the flotation cell. Extensive preliminary studies showed that the amount of collector and frother required are coal-specific and depend on the inherent hydrophobicity of the coal. The standard dosages were selected so that the flotation recovery was between 60 and 80 percent, but no attempt was made initially to optimize these tests. Further, separate standard tests were developed for each coal sample with respect to whether it had been ground dry or wet or to a nominal topsize of 28 mesh or 200 mesh. This resulted in four standard flotation reagent additions for each coal. The collector and frother requirements in the standard flotation of the three base coals is in the following order, in accordance with the relative hydrophobicity of each coal:

Upper Freeport PA < Pittsburgh No. 8 < Illinois No. 6

An extensive QA/QC program shows that the results of both the standard flotation tests and coal analysis obtained in the three different research sites (universities) agree quite well.

Although increased efficiency of separation is always achieved by refloating the concentrate, the standard test was designed to be single-stage only for simplicity.

The so-called Hancock efficiency index was used for assessing the separation efficiency of the various flotation experiments. This index simplifies down to the sum of the combustible matter recovery plus the pyritic sulfur rejection minus 100. Thus, if the initial project objective of 90% Btu recovery at 90% pyritic sulfur rejection were achieved, the efficiency index (El_H)

would be 80. For 200-mesh wet-ground coal samples, the separation efficiency for the flotation separation of Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA coals under the standard test conditions is 36, 50 and 52, respectively.

Optimization of the reagent addition with Illinois No. 6 indicated that the reagent dosages used in the Standard Flotation Test were almost identical to the optimized dosage, while the results of optimization tests of flotation machine parameters with Pittsburgh No. 8 showed that the flotation machine parameters had little effect on the efficiency of separation and that the standard tests also produced results close to the optimum.

In order to determine the best results achievable with dodecane and MIBC using flotation, release analysis curves were determined for 200-mesh grinds of each base coal. The maximum separation efficiency obtained from the release analysis for Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA coals is 65, 56 and 64, respectively. These numbers are to be compared with those obtained from the standard flotation tests, namely 36, 50 and 52, respectively. As expected, the results of the release analysis (flotation separation) for all three base coals are not as good as those of the gravity-based washability tests, which can achieve a pyritic sulfur rejection of 90% at a Btu recovery of 90% with a separation efficiency of 80 at a 200-mesh grind. This is because washability tests (if done correctly) make a separation based on particle composition (specific gravity) whereas flotation makes a separation based on surface composition and relative surface hydrophobicity. Release analysis curves provide a good base for assessing the flotation performance of various reagents used in comparing flotation separations.

A procedure was developed for estimating the pyritic sulfur content of coal separation products from their total sulfur and ash content. By assuming that the organic sulfur content in the combustible material in a coal is constant, a linear relationship is found among the pyritic sulfur, total sulfur and the ash content of a coal sample. This linear relationship has been confirmed from analysis of the products obtained from the various flotation and washability tests. The values of the proportionality constants in the linear equation for a specific coal are first determined from the experimental pyritic sulfur, total sulfur and ash contents of several coal separation products of the same coal using linear regression analysis. After the parameters in the equation have been established for a particular coal, the pyritic sulfur content of other separation

products of the same coal can be calculated based on their total sulfur and ash content using the equation. The results have shown that the calculated pyritic sulfur contents of various coal samples agree very well with their experimental values. The main advantage of this procedure is that it is reliable and rapid because total sulfur and ash content of coal can be determined easily using microprocessor-controlled machines.

15.8 Effect of Grinding Environment

A study of the grinding environment indicated that wet grinding resulted in a higher flotation yield and combustible material recovery than dry grinding for 200-mesh Illinois No. 6 and Upper Freeport PA coals at the same standard reagent dosages, respectively. A further study on behavior of 200-mesh Pittsburgh No. 8 coal showed that the separation efficiency of wet-ground samples is significantly higher than that of dry-ground samples. On the other hand, grinding in an inert atmosphere and floating with either air or argon as the carrier gas had little effect on the flotation performance for all three coals, as compared to grinding and floating the coal in an uncontrolled atmosphere. Therefore, it was concluded that grinding and floating the coal in air (uncontrolled atmosphere) should be satisfactory for industrial purposes. Grinding the coal wet should also be used industrially because of the reduced energy consumption for grinding. With respect to the points of reagent addition, both the flotation yield and the flotation rate constant of hydrophilic Illinois No. 6 coal were higher when the collector was added to the mill before grinding than when the collector was added to the cell after grinding. However, for Pittsburgh No. 8 and Upper Freeport PA coals (more hydrophobic coals), the flotation yield is lower when the standard dosage of the collector is added to the mill before grinding. Similarly, the flotation yield is significantly reduced when the standard dosage of the frother was added to the mill for all three coals. Finally, grinding coal in a ceramic mill was not found to improve the flotation performance of the coal studied.

15.9 Surface Modification

Surface modifiers, which are reagents that interact with or adsorb on the surface of coal or on pyrite and thereby should make the coal surface more hydrophobic or pyrite more

hydrophilic, were tested to enhance the separation of pyrite from coal by advanced froth flotation. The effect of pH, controlled by the addition of lime, was studied as a first step and the results showed that the flotation yield of 28-mesh dry-ground Pittsburgh No. 8 coal decreased when the pH was raised above 10. At the same time, there was a steady increase in the pyritic sulfur rejection from minus 28-mesh coal, prepared either by dry grinding or wet grinding. The drop in the yield and the efficiency index above pH 6 was more dramatic for the minus 200-mesh dry-ground material and above pH 8 for the minus 200-mesh wet-ground coal. Similar results were obtained for Illinois No. 6 coal. The Upper Freeport PA sample did not exhibit any drop in yield up to pH 10. Because the flotability of both coal and pyrite decreases at pH's above 8, rejection of pyrite at high Btu recovery can not be accomplished solely by controlling the pH.

Although the effect of pH on the flotation behavior was easily observed, the physical changes occurring on the surfaces of the coal and pyrite upon change in pH, were not obvious and an extensive study was undertaken to try to elucidate the underlying phenomena. The major finding of this investigation was that the pH at which the grinding step is carried out is extremely important. When coal is ground at low pH, various soluble metal cations are introduced into the coal slurry and if the pH is subsequently increased, these cations begin to precipitate as hydroxides, often onto the surface of coal and pyrite particles, causing the yield to decrease. On the other hand, if the coal is ground at a higher pH, metal cations are not solubilized and therefore do not subsequently precipitate during flotation. It was found that when Pittsburgh No. 8 coal was ground and floated at relatively high pH (6-10), the flotation yield is higher than when the coal was ground at natural pH and subsequently floated at high pH. The overall effect of grinding and floating at the same pH results in an increase in the separation efficiency.

One series of modifiers tested involved various anionic surfactants. Coal surface modification was attempted by adding these surfactants to the flotation cell and evaluating their effect on the flotation product. Of the anionic reagents tested, only Aerosol OT (2-ethylhexylsulfosuccinate) resulted in a small improvement in the flotation response of Pittsburgh No. 8 coal, using the standard collector and frother dosages. Other anionic surfactants

tested, 4-t-butylpyridine and 2-(1-butylpentyl)pyridine, do not appear to have promise as flotation reagents for aiding coal desulfurization.

A number of nonreactive nonpolymerizable reagents, such as methanol, ethanol, alkylbenzaldehyde and glyoxal, were also tested to determine their effect on the flotation performance of the three base coals. The addition of methanol during the wet-grinding of Upper Freeport PA coal to minus 200 mesh resulted in a combustible material recovery of 86% and a pyritic sulfur rejection of 81% ($EI_H = 67$), representing a substantial improvement over the standard flotation test. However, no such improvement was observed for Pittsburgh No. 8 or for Illinois No. 6 coal. The improvement obtained with methanol suggested that other alcohols may also have beneficial effects on flotation. While the improvements in performance for Upper Freeport PA coal using ethanol were comparable to methanol, some improvements were achieved with Pittsburgh No. 8 coal but none with Illinois No. 6 coal. Tests with butylbenzaldehyde, another non-polymerizable reagent, did not result in any improvement in performance over the standard test. In addition to these non-polymerizable reagents, a number of other organic reagents that have functional groups which might interact with coal surface sites and possibly alter the flotation behavior were also examined. These included ethylene glycol, polyethylene glycol, succinic acid, pentyl ether, butylamine and dipentylamine. However, none of these reagents was found to improve the pyrite separation efficiency over that obtained in the standard tests.

Organic monomers, which can polymerize to form large hydrocarbon macromolecules, were added during grinding to determine if mill conditions were conducive for the polymerization reaction to occur between the monomer and newly generated coal surface sites. For a number of practical considerations vinyl acetate, styrene, diisobutylene and methyl methacrylate were selected for testing. The coals were dry ground with the monomers in an argon-purged rod mill. Some increase in yield was observed. After extensive testing, observed increases in flotation yield were attributed to an increase in the amount of hydrocarbons present during flotation and not due to graft polymer formation because saturated analogs of the monomers such as ethyl benzene, ethyl acetate and 1,1,4-trimethylpentane affected flotation to the same extent. Commercially available monomers are generally available only with an inhibitor added to prevent violent polymerization from occurring. Since the inhibitor may prevent polymerization with coal surface

sites, vinyl acetate was distilled to remove the inhibitor, thereby obtaining a pure monomer for testing. The tests using vinyl acetate without inhibitor did not result in any improved flotation performance over that in which the inhibitor was present. This research indicates that without the addition of external free-radical initiators, the formation of in-situ graft polymers on the coal surface does not appear to be a technically feasible approach in advanced coal flotation.

The function of dodecane in the flotation of coal is to enhance the hydrophobicity of the coal particles. However, a detailed study of dodecane addition on coal flotation showed that dodecane adsorption is non-selective and at high dosages, pyrite and ash are also rendered hydrophobic. In order to obtain better separation efficiency, lower dodecane dosages and higher MIBC dosages should be used in the flotation. For a relatively hydrophilic coal such as Illinois No. 6, larger dodecane additions are required to recover the combustible material, which contributes to increasing the flotation of pyrite and ash in the clean coal. For this reason, a series of nonionic surfactants were tested to determine if they provided better flotation selectivity than dodecane for hydrophilic coal. These reagents, oxygenated alkyl aromatic compounds, were found to be powerful collecting reagents for Illinois No. 6 coal at reagent additions as low as 1/10th of that of dodecane. The oxygen groups tended to decrease the separation efficiency of the reagents, with nonylbenzene giving the best separation with the purely alkyl aromatic series. These reagents have a similar effect on Pittsburgh No. 8 coal. Because the interaction of oxygenated compounds seems to be dependent on oxygen sites on the coal surface, this investigation was extended to the flotation of oxidized coals. Laboratory-oxidized Pittsburgh No. 8 coal was floated with dodecane and with two different compounds of this series. Dodecane alone was not able to render the surface of the coal sufficiently hydrophobic, resulting in low combustible matter recovery (CMR values). However, the addition of oxygenated compound resulted in large CMR's at relatively low dosages as compared to that of dodecane.

The effect of commonly used dispersants, namely sodium silicate and sodium hexametaphosphate, on the separation efficiency of Pittsburgh No. 8 coal was also studied. The results show that neither of them has much effect on pyritic sulfur rejection or on ash rejection.

15.10 Pyrite Depressants

A number of organic reagents having different functional groups were tested to determine their pyrite-depressing effect. The results show that glycerol monothioglycolate, mercaptoacetic acid, 2-mercaptoethanesulfonic acid, aminoacetic acid, aminoethanesulfonic acid and sulfosalicylic acid improve the separation efficiency when added to the mill in the grinding stage. For instance, grinding Pittsburgh No. 8 coal with 2-mercaptoethanesulfonic acid (1.0 lb/T) results in an improvement of pyrite rejection of about 10 percent at similar CMR with single-stage flotation, an improvement of about 20% with two-stage flotation.

However, a number of effective ore pyrite depressants, such as calcium cyanide, xanthated polyacrylic acid, xanthated sucrose, and xanthated citric acid, were found to be ineffective depressants for pyrite in coal flotation. In order to reduce the hydrophobicity of pyrite, hydrogen peroxide (H_2O_2) was also tested as a selective oxidant for pyrite in coal flotation. The results show that the addition of hydrogen peroxide reduces the flotability of pyrite, but it also reduces the flotability of coal to the same extent. Thus the addition of hydrogen peroxide to coal flotation has no effect on pyritic sulfur rejection and ash rejection.

Because Illinois No. 6 coal is relatively hydrophilic and almost no material floats without the addition of an oily collector, flotation of pyrite from this coal, known as reverse flotation, using xanthate and dithiophosphates as collector was tested in order to increase the separation efficiency. However, the results were not promising because the collector dosages required were high and substantial amounts of coal were also recovered in the froth.

15.11 Coal Flotation Kinetics

A flotation kinetic study was undertaken to systematically delineate the effect of particle size on the recovery and rate constants for the flotation of coal and the associated mineral matter. For this purpose, the three bituminous coals were ground wet to produce flotation feed at three different top sizes, namely 28, 100 and 200 mesh. This study showed that in the batch flotation, the kinetic behavior of coal flotation can most simply be described by a first-order kinetic model with a sine function (in the range of 0 to $\pi/2$) of flotabilities for the floatable particles. The ultimate recovery and flotation rate constant obtained by fitting experimental results with this

kinetic model represent the ultimate recovery of coal and the rate at which this final state can be reached, respectively.

The ultimate recovery of the combustible material is significantly higher than that of ash and pyrite, whereas the flotation rate constant of the combustible material is only slightly higher than that of ash and pyrite. This may indicate that the floated portion of ash and pyrite is either locked with coal or carried over by the froth with the coal.

At constant reagent dosages, the flotation rate constant decreases significantly as the grind size of the flotation feed is decreased, due not only to the low collector coverage on the surface of the coal particles but also to froth overloading during the first half minute of flotation of the fine feed.

The effect of particle size on the flotation recovery and rate constant of coal is not as pronounced as in mineral flotation due to the low specific gravity and the hydrophobic nature of coals. The effect of particle size is small for the more hydrophobic coals, higher reagent dosages and smaller top feed size of coal. For minus 28-mesh coal, the intermediate size fractions (48 x 100 and 100 x 200 mesh) exhibit higher ultimate combustible material recovery than the larger (plus 48 mesh) and the smaller (minus 200 mesh) size fractions for all three coals. The flotation rate constant of Illinois No. 6 coal also exhibits a maximum at intermediate sizes, whereas the rate constant of Pittsburgh No. 8 and Upper Freeport PA coals increases monotonically with the increasing particle size. For 100-mesh grinds, the ultimate CMR increases and the flotation rate constant decreases with decreasing particle size. For 200-mesh grinds, both the ultimate CMR and the rate constant increase as the particle size is decreased.

An increase in reagent dosage for minus 200-mesh coal increases the flotation recovery and the rate constant of the plus 400-mesh size fraction more than that of the minus 400-mesh size fraction. At the same time, the increase in pyrite recovery is greater than that of coal, resulting in an overall decrease in the separation efficiency. In general, low recovery of the relatively coarse coal particles and high recovery of fine pyrite particles are the major problems in separating pyrite from fine coal using froth flotation.

15.12 Flotation Optimization and Circuits

The effect of frother conditioning time on separation efficiency and flotation kinetics of the base coals was studied. The results show that the separation efficiency does not vary with the frother conditioning time whereas the flotation rate constant decreases with the increase in the frother conditioning time (in the range 0.5 to 3 minutes). At optimized reagent dosages of dodecane and MIBC, the maximum separation efficiency of 100-, 200- and 325-mesh Upper Freeport PA coal is about 65 with a single-stage flotation. This indicates that grinding this coal finer than 100 mesh does not result in significant improvement of the separation efficiency, which is in accordance with the results obtained in the washability tests.

The effect of various flotation circuit configurations on the separation efficiency of the base coals was investigated. These circuits consisted of closed-circuit flotation, cleaner-scavenger circuits, and circuits with staged-reagent additions. In general, two-stage flotation (recleaning the concentrates) can improve the separation efficiency significantly over single-stage flotation. The best results were obtained by refloating the middlings (the tailings from the cleaning stage) with new flotation feed (closed-circuit flotation).

The flotation column is generally considered to be more efficient than conventional flotation machines for fine coal cleaning. The separation efficiency in a single-stage column flotation was found to be the same as that obtained in two-stage flotation with the conventional mechanical flotation cell. By pre-cleaning 28-mesh coal with a shaking table and floating the table concentrates after they had been reground to minus 200 mesh, the separation efficiency can be improved significantly over that obtained by the standard flotation of 200-mesh coal. This separation efficiency was found to be equivalent or better than that obtained by two-stage grinding along with two-stage flotation.

Under the best flotation conditions, the maximum separation efficiency obtained for 200-mesh Upper Freeport PA coal is 75 (combustible material recovery of 89% and pyritic sulfur rejection of 86%). This value, reported by the University of Utah, was achieved by floating wet-ground Upper Freeport PA coal at natural pH in a closed circuit for five cycles with 4.0 lb/T butanol added to the grinding stage. This efficiency index is significantly higher than that obtained in the standard flotation test ($EI_H = 52$) and in the release analysis using dodecane and

MIBC ($EI_H = 64$). For 200-mesh Pittsburgh No. 8 coal, the maximum efficiency index is 71 (CMR = 91% and PSR = 80%), reported by Columbia University. This result was obtained by two-stage flotation of 200-mesh Pittsburgh No. 8 coal, which had been ground wet at pH 8 (with sodium hydroxide added to the grinding stage). The flotation test was conducted using 1.95 lb/T of MIBC at the same pH as in the grinding stage. The maximum efficiency index for Pittsburgh No. 8 is also significantly higher than that obtained with the standard flotation procedure ($EI_H = 50$) and in the release analysis using dodecane and MIBC ($EI_H = 56$). For 200-mesh Illinois No. 6 coal, the maximum efficiency index that was obtained is 56 (CMR = 78% and PSR = 78%), which was achieved by two-stage flotation of a coal sample that had been wet ground with 1.0 lb/T of mercaptoacetic acid at natural pH (about 4). The reagents used in these tests were the standard dosages of dodecane and MIBC. Although this value is low, it is still significantly higher than that obtained in the standard flotation test ($EI_H = 36$) and in the release analysis for the sample that had undergone the same storage time ($EI_H = 42$). However, this value is lower than the value of EI_H obtained in release analysis for the coal samples with a shorter storage time ($EI_H = 65$) where less degradation of the coal samples had taken place during the storage. Illinois No. 6 coal is more susceptible to oxidation than Pittsburgh No. 8 and Upper Freeport PA coals. Because of the lower hydrophobicity, Illinois No. 6 coal is also the most difficult coal to clean among the three base coals.

15.13 Coal Weathering

Coal weathering studies were conducted on three size fractions, plus 1-inch, 1-inch x 1/4-inch, and 1/4-inch x 0. Each size fraction was screened in the field and stored in three different modes: i) inert, in drums under an argon atmosphere; ii) covered, in drums without lids but with plywood boards on top; and iii) open, in a stockpile completely exposed to the atmosphere. A total of thirteen weathering increments was collected, the first six on a biweekly basis starting in November 1988, the next four on a monthly basis, and the final three increments on a bimonthly basis. Each increment was inerted with argon before being shipped to Berkeley and to the University of Pittsburgh for characterization studies.

Physical and chemical changes that might occur during the storage of coal were examined by characterizing the weathered coal samples using various techniques. This characterization included size distribution, proximate analysis, DRIFT spectroscopy, zeta potential, standard flotation, film flotation and induction time determinations. The proximate analysis showed that no appreciable decrease in the volatile matter of the weathered samples occurred over the 13-month weathering period. The particle size distribution of the minus 28-mesh fraction screened from the minus 1/4-inch sample stored in open mode indicated that for Illinois No. 6 coal, particles in the intermediate size fractions (such as 28 x 35 mesh) became more friable, contributing to an increase in the amount of material in the finer size fractions as the weathering time increased. Particle size analysis of the 1/4-inch x 28 mesh material indicated a progressive increase in the fines as weathering progressed. Interestingly, the size degradation that was observed for the Illinois No. 6 coal sample did not occur with either Pittsburgh No. 8 or Upper Freeport PA coal.

A comparison of the DRIFT spectra of the research and weathered samples for each of the three base coals showed some differences between them. Spectra for Illinois No. 6 weathered for short periods of time showed the presence of a peak corresponding to the carbonyl group (1655 cm^{-1}), indicating a certain degree of oxidation. No carbonyl peaks were found in the DRIFT spectra of the weathered samples of Pittsburgh No. 8, indicating that this coal is less susceptible to oxidation by weathering for short periods of time. No peaks associated with oxidation products were obtained for Upper Freeport PA weathered samples.

Film flotation tests conducted on the weathered samples of the three base coals showed a slow drop in hydrophobicity initially, followed by a rapid decrease after five months of weathering. Weathering began in the winter, and only after the weather began to warm up in the spring did the Pittsburgh No. 8 and Upper Freeport coals begin to weather. Furthermore, both film flotation and induction time measurements clearly indicated that the hydrophobicity of the weathered samples is a function of the storage environment. Specifically, the hydrophobicity of coal stored in the inert mode is greater than that for coal stored in the covered and open modes for similar periods of weathering. The bubble/particle contact time for Illinois No. 6 stored in the open mode increased from 60 milliseconds for Increment 1 to 1750 milliseconds for

Increment 13. The same trend was observed with the other two coals, but, the increase in induction time was not as dramatic. Wettability tests done with the Hallimond tube (up to Increment 4) indicated that the hydrophobicity of the weathered samples drops only marginally and that the yields were less than that of the research samples, probably due to the lower ash content in the research samples.

The standard flotation test procedure developed for this program for the 28-mesh ground coal was also used to float the minus 28-mesh weathered material screened from minus 1/4-inch fraction. The results showed that there is a decrease in the flotation yield as compared to the freshly ground research samples. This drop was higher for the open mode as compared to the covered and inert modes for the same weathering time. The rate of decrease in the yield is also coal-dependent, with Illinois No. 6 coal yield decreasing very rapidly initially as compared to the other two coals before levelling off at a yield of 28%. The recovery of Pittsburgh No. 8 and Upper Freeport PA coals decreased little during the first 5 months of weathering and then decreased rapidly for coals weathered through the summer months. This behavior seems to correlate quite well with the temperature in the weathering sites. When the weathering temperature rises, the oxidation of the coals is more significant. After 9 months of weathering, the yield for these two coals increased slightly, the reason for which is not clear at this point. Induction time measurements also show an increase in the apparent hydrophobicity of Upper Freeport PA and Pittsburgh No. 8 coals that had been weathered for longer than 9 months. Flotation pulp pH in the standard flotation of the minus 28-mesh weathered coals was also decreased as the weathering time increased.

Grinding of the 1/4-inch x 28-mesh material to minus 28 mesh before flotation restored the yield equivalent to that of the research samples, indicating that the effect of weathering is mainly limited to the external surfaces only. Therefore, coal should be stored as coarse as possible in order to minimize the detrimental effect of oxidation on the surface hydrophobicity of coal. In addition, the largest decreases in the hydrophobicity of the coals occurred at the onset of spring and summer temperatures, suggesting that if coal must be stored, significant changes can be expected to occur and these will be a function of ambient temperature.

15.14 Degradation of Stored Coal Samples

Although the research samples were inerted carefully under argon atmosphere, degradation of the stored coal samples has been observed during the course of this research. The separation efficiency under the same flotation conditions decreases for coal samples that had been stored under the "inert" environment for a long time. This suggests that in any research effort extending over a period of time, it is necessary to regularly collect newly mined samples that are as close to the original location in the region as possible. Without this regeneration of coal samples, the results obtained from the latter part of any research project must be viewed with caution.

16.0 RECOMMENDATIONS FOR FUTURE WORK

This extensive investigation on surface control for pyritic sulfur removal from coal by employing advanced flotation concepts has shown the necessity for in-depth fundamental research in a number of areas which impinge on this problem. Recommendations are outlined briefly in the paragraphs that follow:

- 1) Because locked coal-pyrite particles account for some of the pyritic sulfur in the cleaned product, research should be directed at finding innovative routes for enhanced liberation at coarser sizes, using new energy-efficient, industrially-proven grinding machines, such as the high-pressure roll mill, possibly in combination with conventional milling, and stirred ball milling.
- 2) More detailed study of the kinetics of coal flotation should be carried out to delineate more fully the distribution in flotation rate constants, for quantifying the effects of
 - particle size (coal, ash, pyrite)
 - reagent type and dosage
 - aeration rate and other operating variables
 - flotation machine design

This should be of value for realistic simulation and design of advanced flotation systems.

- 3) Delineate the mechanism of pyrite flotation in coal, including factors that contribute to the hydrophobicity of pyrite, entrainment of pyrite in the froth and the flotation of locked particles. Such an investigation should cover the effect of froth stability and other characteristics of the froth in order to understand the physical phenomena responsible for entrainment of pyrite in the froth.
- 4) Study of pyrite surface modifiers and their selective adsorption on pyrite. Continued search for new functional groups on reagents to act as surface modifier/pyrite depressants in coal flotation should be carried out and tested. Interesting leads should be followed in order to come up with the most promising compounds with regard to cost and effectiveness. New compounds and functional groups should be explored as depressants

for ash. Full characterization of the systems should be carried out in order to provide fundamental knowledge of surface modification processes.

- 5) Study of the modification of oxidized coal surfaces in order to restore hydrophobicity. Some coals oxidize more readily than others and this results in a lower combustible matter recovery. The highly effective oxygenated alkyl reagents should be studied further to find a more selective surface modifier, which could lead to a more selective, yet efficient, way of enhancing the flotation of oxidized coal.
- 6) More detailed investigation of the modification of coal and pyrite that occurs during grinding should be carried out. The reaction of reagents with the new surface that is formed during the grinding step could result in lower reagent dosages and shorter conditioning times. Savings in reagent cost as well as equipment cost per ton of coal might result.
- 7) Investigate the development of laboratory-scale procedures to simulate flotation circuits with optimum combination of experimental inputs and circuit modeling. Studies should be carried out to clearly show whether release analysis or tree flotation is indeed machine-independent. This should be highly useful for more realistic evaluation of new reagents and also for advanced flotation circuit simulation.
- 8) Consider more complex circuits, for example the use of classified feeds or combined gravity/flotation circuits. Each process has its own advantages as well as limitations, and a combination process might result in the best of each approach. Hybrid processes, such as combination of agglomeration/flotation processes, might prove beneficial.

APPENDIX
DATA FOR FIGURES

ORIGINAL SAMPLE, UNCRUSHED

ELEMENTARY DATA										COMPUTED DATA: CUMULATIVE									
SPECIFIC GRAVITY		(%)	(%)	(%)	(%)	BTU	\$ SO2/ M BTU	(%)	(%)	(%)	(%)	BTU	BTU REC	(%)	# SO2/ M BTU REJECTED				
SINK	FLOAT	WT%	ASH	TOT SUL	PYR SUL			WT%	ASH	TOT SUL	PYR SUL								
1.30		20.7	4.40	2.98	0.68	13801	4.31	20.7	4.40	2.98	0.68	13801	24.10	4.31	95.25				
1.30	1.35	29.8	8.41	3.45	1.09	13166	5.24	50.5	6.77	3.26	0.92	13426	57.23	4.85	94.27				
	1.40	24.8	13.87	3.81	1.71	12289	6.19	75.3	9.10	3.44	1.18	13052	82.90	5.27	69.95				
1.35		11.9	21.13	5.46	3.64	11098	9.84	87.2	10.74	3.71	1.52	12785	94.04	5.81	55.25				
1.40	1.60	4.2	34.42	6.82	5.63	8977	15.20	91.4	11.84	3.86	1.71	12609	97.25	6.12	47.16				
1.60	1.80	8.6	65.01	16.39	16.16	3784	86.65	100.0	16.42	4.94	2.95	11849	100.00	8.33	0.00				
1.80																			

WASHABILITY DATA
SEAM: ILLINOIS NO. 6
RANDOLPH COUNTY, ILLINOIS

[illegible]

WASHABILITY DATA
SEAM: ILLINOIS NO. 6
RANDOLPH COUNTY, ILLINOIS

ORIGINAL SAMPLE, CRUSHED TO 1/8" TOPSIZE

1/8" X 0 COMPOSITE

ELEMENTARY DATA

COMPUTED DATA: CUMULATIVE

SINK	SPECIFIC GRAVITY	VTX	(%)	ASH	TGT SUL	(%)	2.75	0.42	BTU	# SO2/ M BTU	VTX	(%)	ASH	TOT SUL	(%)	2.75	0.42	BTU	BTU REC	(%)	# SO2/ M BTU REJECTED
1.30	1.30	34.5	34.5	3.34	2.75	0.42	13817	3.98	13817	3.98	34.5	34.5	3.34	2.75	0.42	13817	40.54	3.98	40.54	3.98	95.07
1.35	1.35	16.2	50.7	7.27	3.07	0.82	13194	4.65	13194	4.65	50.6	50.6	4.59	2.85	0.55	13618	58.72	4.19	58.72	4.19	90.58
1.40	1.40	12.0	62.7	10.47	3.27	1.07	12723	5.13	12723	5.13	62.6	62.6	5.72	2.93	0.65	13447	71.69	4.36	71.69	4.36	86.25
1.40	1.40	18.9	81.6	18.26	3.81	2.01	11553	6.60	11553	6.60	81.5	81.5	8.63	3.14	0.97	13008	90.29	4.82	90.29	4.82	73.48
1.60	1.60	6.3	87.9	26.72	4.24	2.84	10093	8.39	10093	8.39	87.8	87.8	9.92	3.22	1.10	12798	95.70	5.02	95.70	5.02	67.45
1.80	1.80	12.2	100.0	60.79	16.68	16.41	4140	80.57	4140	80.57	100.0	100.0	16.13	4.86	2.97	11742	100.00	8.27	100.00	8.27	0.00

WASHABILITY DATA
SEAM: ILLINOIS NO. 6
RANDOLPH COUNTY, ILLINOIS

ORIGINAL SAMPLE, CRUSHED TO 28 M TOPSIZE

28 M X 0 COMPOSITE

ELEMENTARY DATA

COMPUTED DATA: CUMULATIVE

SINK	SPECIFIC GRAVITY	VTX	(%)	ASH	TGT SUL	(%)	2.45	0.39	BTU	# SO2/ M BTU	VTX	(%)	ASH	TOT SUL	(%)	2.45	0.39	BTU	BTU REC	(%)	# SO2/ M BTU REJECTED
1.30	1.30	1.8	1.8	1.31	2.45	0.39	14169	3.45	14169	3.45	1.8	1.8	1.31	2.45	0.39	14169	2.11	3.45	2.11	3.45	99.76
1.35	1.35	25.5	27.3	2.45	2.64	0.52	13923	3.79	13923	3.79	27.3	27.3	2.37	2.62	0.51	13939	32.26	3.77	32.26	3.77	95.16
1.40	1.40	32.7	60.0	6.42	2.93	0.85	13271	4.41	13271	4.41	60.0	60.0	4.58	2.79	0.70	13575	69.12	4.11	69.12	4.11	85.53
1.60	1.60	25.0	84.9	17.42	3.57	1.81	11651	6.13	11651	6.13	84.9	84.9	8.35	3.02	1.02	13009	93.81	4.64	93.81	4.64	69.90
1.80	1.80	3.3	88.2	36.54	5.76	4.26	8651	13.31	8651	13.31	88.2	88.2	9.41	3.12	1.14	12846	96.24	4.86	96.24	4.86	65.01
		11.8	100.0	62.27	16.75	15.95	3768	88.90	3768	88.90	100.0	100.0	15.62	4.72	2.88	11779	100.00	8.02	100.00	8.02	0.00

SIZE FRACTION: 200 M X 0 - 100.00 X

A-3

WASHABILITY DATA
SEAM: PITTSBURGH NO. 8
BELMONT COUNTY, OHIO

ORIGINAL SAMPLE, UNCRUSHED

4" X 0 COMPOSITE

ELEMENTARY DATA

COMPUTED DATA: CUMULATIVE

SINK	FLOAT	(%)	WT%	ASH	TOT SUL	PYR SUL	BTU	M BTU	# SO2/	WT%	ASH	TOT SUL	PYR SUL	BTU	BTU REC	(%)	# SO2/	XPYR SUL
1.30	1.30	18.3	18.3	4.34	2.12	0.93	14224	2.99	16.3	16.3	4.34	2.12	0.93	14224	20.04	2.99	94.24	94.24
1.35	1.35	54.6	54.6	6.37	3.04	2.06	13864	4.39	72.9	72.9	5.86	2.81	1.78	13953	78.21	4.03	56.26	56.26
1.40	1.40	10.8	10.8	10.34	4.84	3.96	13206	7.34	83.7	83.7	6.44	3.07	2.06	13858	89.15	4.44	41.83	41.83
1.60	1.60	7.9	7.9	18.48	6.01	5.42	11841	10.15	91.6	91.6	7.48	3.33	2.35	13684	96.35	4.86	27.34	27.34
1.80	1.80	2.8	2.8	34.29	6.05	5.35	9227	13.10	94.3	94.3	8.26	3.41	2.44	13554	98.30	5.03	22.36	22.36
1.80	1.80	5.7	5.7	64.88	12.12	11.68	3893	62.26	100.0	100.0	11.47	3.90	2.96	13007	100.00	6.00	0.00	0.00

WASHABILITY DATA
SEAM: PITTSBURGH NO. 8
BELMONT COUNTY, OHIO

ORIGINAL SAMPLE, CRUSHED TO 1-1/2" TOPSIZE

1-1/2" X 0 COMPOSITE

ELEMENTARY DATA

COMPUTED DATA: CUMULATIVE

SINK	FLOAT	(%)	WT%	ASH	TOT SUL	PYR SUL	BTU	M BTU	# SO2/	WT%	ASH	TOT SUL	PYR SUL	BTU	BTU REC	(%)	# SO2/	XPYR SUL
1.30	1.30	31.8	31.8	4.57	2.28	1.15	14265	3.20	31.8	31.8	4.57	2.28	1.15	14265	35.00	3.20	87.51	87.51
1.35	1.35	39.1	39.1	6.25	3.04	1.99	13926	4.37	70.9	70.9	5.49	2.70	1.61	14078	76.99	3.83	61.04	61.04
1.40	1.40	11.3	11.3	9.85	4.59	3.73	13319	6.89	82.1	82.1	6.09	2.96	1.90	13974	88.55	4.23	46.74	46.74
1.60	1.60	8.2	8.2	18.02	6.01	5.23	11890	10.11	90.4	90.4	7.18	3.24	2.20	13784	96.10	4.70	32.06	32.06
1.80	1.80	2.8	2.8	33.45	5.64	5.00	9340	12.07	93.1	93.1	7.95	3.31	2.29	13653	98.02	4.84	27.37	27.37
1.80	1.80	6.9	6.9	66.27	12.08	11.65	3615	66.85	100.0	100.0	11.97	3.91	2.93	12962	100.00	6.04	0.00	0.00

ORIGINAL SAMPLE, CRUSHED TO 1/8" TOPSIZE

31504103 0 X 18/1

ELEMENTARY DATA										COMPUTED DATA: CUMULATIVE													
SINK	SPECIFIC GRAVITY	FLOAT	(%)		(%)		(%)		# SO2/ M BTU	BTU	WTX	ASH	(%)	SUL	(%)	PYR	SUL	(%)	BTU	REC	(%)	# SO2/ M BTU	REJECTED
			WTX	ASH	TOT	SUL	PYR	SUL															
	1.30		46.4	3.34	1.85	0.60			2.56	14406	46.4	3.34	1.85	0.60				51.63				2.56	90.01
1.30	1.35		25.4	6.66	2.93	1.82		4.24	13803	71.7	4.51	2.23	1.03	1.03				78.71				3.14	73.37
1.35	1.40		8.3	10.29	4.21	3.14		6.38	13184	80.0	5.11	2.43	1.25	1.25				87.18				3.45	63.99
1.40	1.60		8.8	16.96	6.05	4.57		19.05	12037	88.8	6.28	2.79	1.58	1.58				95.34				4.02	49.56
1.60	1.80		2.8	32.26	7.46	6.74		15.76	9462	91.6	7.08	2.93	1.74	1.74				97.39				4.27	42.77
1.80			8.4	65.52	14.74	14.18		73.24	4024	100.0	11.98	3.92	2.78	2.78				100.00				6.06	0.00

WASHABILITY DATA
SEAM: PITTSBURGH NO. 8
BELMONT COUNTY, OHIO

ORIGINAL SAMPLE, CRUSHED TO 28 H TOPSIZE

28 H X 0 COMPOSITE

ELEMENTARY DATA										COMPUTED DATA: CUMULATIVE									
SPECIFIC GRAVITY		(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
SINK	FLOAT	WT%	ASH	TOT SUL	PYR SUL	BTU	# SO2/ M BTU	WT%	ASH	TOT SUL	PYR SUL	BTU	# SO2/ M BTU	WT%	ASH	TOT SUL	PYR SUL	BTU	# SO2/ M BTU
1.30		51.1	2.66	1.59	0.33	14421	2.20	51.1	2.66	1.59	0.33	14421	2.20	51.1	2.66	1.59	0.33	14421	2.20
1.35		18.7	6.15	2.45	1.31	13883	3.53	69.7	3.59	1.82	0.59	14277	77.43	69.7	3.59	1.82	0.59	14277	85.09
1.35		1.40	8.3	8.42	2.94	13412	4.36	78.1	4.11	1.94	0.73	14184	86.12	78.1	4.11	1.94	0.73	14184	93.91
1.40		1.60	9.2	15.34	4.50	12313	7.31	87.3	5.29	2.21	1.02	13987	94.94	87.3	5.29	2.21	1.02	13987	98.07
1.60		1.80	2.7	30.61	6.93	9462	14.31	90.0	6.05	2.35	1.17	13858	96.97	90.0	6.05	2.35	1.17	13858	100.00
1.80		10.0	65.78	17.79	17.22	3896	91.33	100.0	12.03	3.90	2.78	12860	100.00	100.0	12.03	3.90	2.78	12860	100.00

WASHABILITY DATA
 SEAH: PITTSBURGH NO. 8
 BELMONT COUNTY, OHIO

ORIGINAL SAMPLE, CRUSHED TO 200 M TOPSIZE

SIZE FRACTION: 200 M X 0 - 100.0 %

ELEMENTARY DATA

COMPUTED DATA: CUMULATIVE

SPECIFIC GRAVITY		(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
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WASHABILITY DATA
SEAM: UPPER FREEPORT
INDIANA COUNTY, PA

ORIGINAL SAMPLE, UNCRUSHED

4" X 0 COMPOSITE

ELEMENTARY DATA

COMPUTED DATA: CUMULATIVE

SINK	FLOAT	(%)	WT%	ASH	TOT SUL	ASH	TOT SUL	PYR SUL	WT%	# SO2/ M BTU	BTU	PYR SUL	ASH	TOT SUL	(%)	BTU	BTU REC	(%)	# SO2/ M BTU	PYR SUL	REJECTED
1.30	1.35	34.3	35.6	3.62	1.23	3.62	1.23	0.65	34.3	15037	15037	0.65	3.62	1.23	0.65	15037	38.36	1.64	85.44		
1.35	1.40	12.5	13.97	9.65	1.45	13.97	1.45	0.94	69.9	14059	14059	0.80	6.69	1.34	0.80	14539	75.63	1.85	63.78		
1.40	1.60	8.9	21.82	21.82	2.44	21.82	2.44	1.04	82.4	13300	13300	0.83	7.80	1.40	0.83	14351	87.98	1.95	55.37		
1.60	1.80	2.4	34.11	34.11	3.90	34.11	3.90	1.54	91.2	11926	11926	0.90	9.16	1.50	0.90	14115	95.85	2.12	46.49		
1.80		6.4	60.26	60.26	10.82	60.26	10.82	3.07	100.0	9659	9659	0.96	9.80	1.56	0.96	14001	97.58	2.23	41.68		
										5109	5109	1.54	13.02	2.15	1.54	13433	100.01	3.20	-0.01		

WASHABILITY DATA
SEAM: UPPER FREEPORT
INDIANA COUNTY, PA

ORIGINAL SAMPLE, CRUSHED TO 1-1/2" TOPSIZE

1-1/2" X 0 COMPOSITE

ELEMENTARY DATA

COMPUTED DATA: CUMULATIVE

SINK	FLOAT	(%)	WT%	ASH	TOT SUL	ASH	TOT SUL	PYR SUL	WT%	# SO2/ M BTU	BTU	PYR SUL	ASH	TOT SUL	(%)	BTU	BTU REC	(%)	# SO2/ M BTU	PYR SUL	REJECTED
1.30	1.35	35.7	26.9	3.71	1.20	3.71	1.20	0.67	35.7	15057	15057	0.67	3.71	1.20	0.67	15057	39.91	1.60	83.31		
1.35	1.40	18.3	13.21	8.29	1.47	13.21	1.47	0.83	62.6	14301	14301	0.74	5.68	1.32	0.74	14732	68.45	1.79	67.69		
1.40	1.60	10.3	21.53	21.53	2.48	21.53	2.48	1.24	80.9	13434	13434	0.75	7.37	1.36	0.75	14439	86.64	1.88	57.92		
1.60	1.80	2.7	32.94	32.94	3.48	32.94	3.48	2.11	91.2	11974	11974	0.80	8.98	1.49	0.80	14160	95.82	2.10	49.03		
1.80		6.1	60.95	60.95	11.60	60.95	11.60	10.67	100.0	9843	9843	0.84	9.66	1.54	0.84	14037	97.78	2.20	45.09		
										4924	4924	1.44	12.78	2.16	1.44	13483	100.00	3.20	0.00		

WASHABILITY DATA
SEAM: UPPER FREEPORT
INDIANA COUNTY, PA

ORIGINAL SAMPLE, CRUSHED TO 1/8" TOPSIZE

1/8" X 0 COMPOSITE

ELEMENTARY DATA

COMPUTED DATA: CUMULATIVE

SPECIFIC GRAVITY	(%)	WT%	ASH	TOT SUL	PYR SUL	BTU	# SO2/ M BTU	(%)	WT%	ASH	TOT SUL	PYR SUL	BTU	(%)	# SO2/ M BTU	BTU REJECTED	2PYR SUL
SINK	1.30	42.3	3.29	1.12	0.53	15204	1.47	42.3	42.3	3.29	1.12	0.53	15204	47.30	1.47	85.88	
	1.35	25.4	8.87	1.33	0.69	14250	1.87	67.7	67.7	5.38	1.20	0.59	14847	73.89	1.62	74.98	
	1.40	10.0	13.00	1.37	0.74	13419	2.05	77.8	77.8	6.36	1.22	0.61	14663	83.78	1.67	70.18	
	1.40	12.2	18.97	1.91	1.07	12376	3.09	89.9	89.9	8.07	1.31	0.67	14353	94.85	1.83	61.93	
	1.60	3.5	32.74	2.93	1.72	9936	5.90	93.5	93.5	9.00	1.38	0.71	14186	97.43	1.94	58.09	
	1.80	6.5	58.02	14.79	14.06	5374	55.05	100.0	100.0	12.21	2.25	1.58	13610	100.01	3.31	-0.01	

WASHABILITY DATA
SEAM: UPPER FREEPORT
INDIANA COUNTY, PA

ORIGINAL SAMPLE, CRUSHED TO 28 M TOPSIZE

28 M X 0 COMPOSITE

ELEMENTARY DATA

COMPUTED DATA: CUMULATIVE

SPECIFIC GRAVITY	(%)	WT%	ASH	TOT SUL	PYR SUL	BTU	# SO2/ M BTU	(%)	WT%	ASH	TOT SUL	PYR SUL	BTU	(%)	# SO2/ M BTU	BTU REJECTED	2PYR SUL
SINK	1.30	44.9	32	0.89	0.11	15197	1.17	44.9	44.9	2.32	0.89	0.11	15197	50.91	1.17	96.19	
	1.35	17.4	6.85	1.07	0.36	14441	1.49	62.3	62.3	3.58	0.94	0.18	14986	69.61	1.26	91.59	
	1.40	5.3	11.34	1.17	0.52	13708	1.71	67.6	67.6	4.19	0.96	0.21	14887	74.99	1.29	89.56	
	1.40	17.8	15.27	1.25	0.57	12913	1.94	85.4	85.4	6.50	1.02	0.28	14475	92.14	1.41	81.95	
	1.60	4.5	23.54	1.58	1.15	11389	2.78	89.9	89.9	7.36	1.05	0.33	14319	95.99	1.47	78.08	
	1.80	10.1	57.07	12.41	10.41	5335	46.52	100.0	100.0	12.37	2.19	1.34	13413	100.00	3.27	0.00	

WASHABILITY DATA
SEAM: UPPER FREEPORT
INDIANA COUNTY, PA

ORIGINAL SAMPLE, CRUSHED TO 200 M TOPSIZE

SIZE FRACTION: 200 M X 0 - 100.00 %

COMPUTED DATA: CUMULATIVE

ELEMENTARY DATA

SPECIFIC GRAVITY	SINK	FLOAT	ELEMENTARY DATA										COMPUTED DATA: CUMULATIVE												
			(%)	WT%	(%)	ASH	(%)	TOT SUL	(%)	PYR SUL	(%)	BTU	# SO2/ M BTU	(%)	WT%	(%)	ASH	(%)	TOT SUL	(%)	PYR SUL	(%)	BTU	BTU REC	(%)
1.30			54.9	1.87	0.79	0.05	15240	1.04	54.9	1.87	0.79	0.05	15240	62.27	62.27	1.04	98.06								
1.35			15.6	4.73	0.89	0.10	14708	1.21	70.5	2.50	0.81	0.06	15122	79.31	79.31	1.07	96.97								
1.35			0.8	5.89	0.90	0.14	14605	1.23	71.2	2.54	0.81	0.06	15117	80.16	80.16	1.08	96.89								
1.40			9.8	12.60	0.91	0.34	13260	1.37	81.0	5.75	0.82	0.10	14893	89.81	89.81	1.11	94.55								
1.60			7.0	23.52	1.16	0.54	11287	2.06	88.0	5.33	0.85	0.13	14606	95.70	95.70	1.17	91.88								
1.60			12.0	60.41	13.36	10.86	4830	55.32	100.0	11.94	2.35	1.42	13433	100.01	100.01	3.50	-0.01								
1.80																									

WASHABILITY DATA
SEAM: ILLINOIS No. 6
RANDOLPH COUNTY, ILLINOIS

FINE COAL WASHABILITY
SAMPLE COMMUNITED TO 100 M TOPSIZE

ELEMENTARY DATA										COMPUTED DATA: CUMULATIVE													
SPECIFIC GRAVITY		(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	# SO2/ M BTU	WTX	(%)	(%)	(%)	(%)	(%)	(%)	(%)	BTU	BTU REC	(%)	# SO2/ M BTU	%PYR SUL REJECTED
SINK	FLOAT																						
1.30		22.6	1.97	2.69	0.13	14032	3.83	22.6	1.97	2.69	0.13	14032	3.83	22.6	1.97	2.69	0.13	14032	27.29		3.83	98.90	
1.35		2.8	2.73	2.65	0.12	13787	3.84	25.4	2.05	2.69	0.13	14005	3.84	25.4	2.05	2.69	0.13	14005	30.61		3.84	98.76	
1.35		20.9	3.00	2.56	0.13	13733	3.73	46.3	2.48	2.63	0.13	13882	3.79	46.3	2.48	2.63	0.13	13882	55.32		3.79	97.77	
1.40		31.8	10.26	2.77	0.54	12571	4.41	78.1	5.65	2.69	0.30	13348	4.03	78.1	5.65	2.69	0.30	13348	89.72		4.03	91.36	
1.60		6.3	28.46	3.40	1.62	9702	7.01	84.4	7.35	2.74	0.40	13076	4.19	84.4	7.35	2.74	0.40	13076	94.98		4.19	87.56	
1.80		15.6	62.97	15.68	15.05	3740	83.85	100.0	16.03	4.76	2.68	11620	8.19	100.0	16.03	4.76	2.68	11620	100.00		8.19	0.00	

WASHABILITY DATA
SEAM: ILLINOIS No. 6
RANDOLPH COUNTY, ILLINOIS

FINE COAL WASHABILITY
SAMPLE COMMUNITED TO 200 M TOPSIZE

ELEMENTARY DATA												COMPUTED DATA: CUMULATIVE													
SPECIFIC GRAVITY		(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	
SINK	FLOAT	WTX	ASH	TOT SUL	PYR SUL	BTU	# SO2/ M BTU	WTX	ASH	TOT SUL	PYR SUL	BTU	# SO2/ M BTU	WTX	ASH	TOT SUL	PYR SUL	BTU	# SO2/ M BTU	WTX	ASH	TOT SUL	PYR SUL	BTU	# SO2/ M BTU
1.30		23.6	2.18	2.71	0.11	13986	3.88	23.6	2.18	2.71	0.11	13986	3.88	23.6	2.18	2.71	0.11	13986	3.88	23.6	2.18	2.71	0.11	13986	3.88
1.35		1.0	3.33	2.34	0.12	13647	3.43	24.6	2.23	2.69	0.11	13972	3.86	24.6	2.23	2.69	0.11	13972	3.86	24.6	2.23	2.69	0.11	13972	3.86
1.35		1.40	7.1	4.80	0.15	13680	3.74	31.7	2.80	2.66	0.12	13907	3.83	31.7	2.80	2.66	0.12	13907	3.83	31.7	2.80	2.66	0.12	13907	3.83
1.40		1.60	44.7	7.33	0.38	13200	4.06	76.4	5.45	2.67	0.27	13493	3.96	76.4	5.45	2.67	0.27	13493	3.96	76.4	5.45	2.67	0.27	13493	3.96
1.60		1.80	7.5	26.09	1.38	10057	6.36	83.9	7.30	2.72	0.37	13186	4.13	83.9	7.30	2.72	0.37	13186	4.13	83.9	7.30	2.72	0.37	13186	4.13
1.80		16.1	62.03	16.70	15.42	3386	98.64	100.0	16.11	4.97	2.79	11608	8.57	100.0	16.11	4.97	2.79	11608	8.57	100.0	16.11	4.97	2.79	11608	8.57

WASHABILITY DATA
SEAM: ILLINOIS NO. 6
RANDOLPH COUNTY, ILLINOIS

FINE COAL WASHABILITY

SAMPLE COMMUNITED TO 325 M TOPSIZE

ELEMENTARY DATA										COMPUTED DATA: CUMULATIVE									
SINK	GRAVITY	W%	ASH	TOT SUL	PYR SUL	BTU	# SO ₂ /M BTU	W%	ASH	TOT SUL	PYR SUL	BTU	BTU REC	(%)	# SO ₂ /M BTU	REJECTED			
1.30	1.35	8.9	1.86	2.62	0.07	13948	3.76	8.9	1.86	2.62	0.07	13948	10.88	3.76	99.77				
1.35	1.40	12.9	3.80	2.61	0.08	13648	3.82	21.8	3.01	2.61	0.08	13770	26.32	3.80	99.39				
1.40	1.60	45.9	7.52	2.51	0.24	12817	3.92	76.9	5.80	2.53	0.17	13169	88.79	3.85	95.04				
1.60	1.80	7.9	25.43	2.83	0.96	10000	5.66	84.8	7.63	2.56	0.25	12874	95.72	3.98	92.24				
1.80		85.2	64.00	16.79	16.41	3212	104.55	100.0	16.20	4.72	2.70	11405	100.00	8.28	0.00				

WASHABILITY DATA
SEAM: ILLINOIS NO. 6
RANDOLPH COUNTY, ILLINOIS

FINE COAL WASHABILITY

SAMPLE COMMUNITED TO 400 M TOPSIZE

ELEMENTARY DATA										COMPUTED DATA: CUMULATIVE									
SINK	GRAVITY	W%	ASH	TOT SUL	PYR SUL	BTU	# SO ₂ /M BTU	W%	ASH	TOT SUL	PYR SUL	BTU	BTU REC	(%)	# SO ₂ /M BTU	REJECTED			
1.30	1.35	7.0	2.05	2.81	0.05	13768	4.08	7.0	2.05	2.81	0.05	13768	8.78	4.08	99.85				
1.35	1.40	6.9	5.43	2.70	0.08	12947	4.17	13.9	3.73	2.76	0.06	13360	16.91	4.12	99.61				
1.40	1.60	15.9	4.57	2.63	0.08	12862	4.09	29.8	4.18	2.69	0.07	13095	35.54	4.11	99.07				
1.60	1.80	46.2	6.42	2.23	0.14	12312	3.62	76.0	5.54	2.41	0.11	12619	87.35	3.82	96.30				
1.80		9.3	18.60	2.43	0.58	10058	4.83	85.3	6.96	2.41	0.16	12340	95.87	3.91	93.99				
		14.7	65.73	16.24	14.94	3084	105.32	100.0	15.60	4.44	2.34	10979	100.00	8.10	0.00				

WASHABILITY DATA
SEAM: PITTSBURGH No. 8
BELMONT COUNTY, OHIO

ADDITIONAL WASHABILITY
ORIGINAL SAMPLE, CRUSHED TO 100 M TOPSIZE

ELEMENTARY DATA										COMPUTED DATA: CUMULATIVE									
SINK	FLOAT	(%)	WT%	ASH	TOT SUL	(%)	PYR SUL	(%)	# SO2/ M BTU	WT%	ASH	TOT SUL	(%)	PYR SUL	(%)	BTU	BTU REC	(%)	# SO2/ M BTU REJECTED
1.30		60.4	2.73	1.57	0.16	14417	2.18	60.4	2.73	1.57	0.16	14417	68.28	2.18	96.36				
1.35		12.1	4.84	1.83	0.44	17976	2.62	72.5	3.08	1.61	0.21	14343	81.54	2.25	94.39				
1.35		5.2	8.20	2.27	0.98	13383	3.39	77.7	3.42	1.66	0.26	14279	87.00	2.32	92.48				
1.40		8.5	13.69	2.95	1.66	12359	4.77	86.2	4.44	1.78	0.40	14090	95.24	2.53	87.20				
1.60		2.7	27.31	4.39	3.15	9928	8.84	88.9	5.13	1.86	0.48	13963	97.34	2.67	84.02				
1.80		11.1	66.16	21.92	20.22	3055	143.50	100.0	11.91	4.09	2.67	12753	100.00	6.41	0.00				

WASHABILITY DATA
SEAM: PITTSBURGH No. 8
BELMONT COUNTY, OHIO

ADDITIONAL WASHABILITY
ORIGINAL SAMPLE, CRUSHED TO 200 M TOPSIZE

ELEMENTARY DATA										COMPUTED DATA: CUMULATIVE									
SINK	FLOAT	(%)	WT%	ASH	TOT SUL	(%)	PYR SUL	(%)	# SO2/ M BTU	WT%	ASH	TOT SUL	(%)	PYR SUL	(%)	BTU	BTU REC	(%)	# SO2/ M BTU REJECTED
1.30		61.7	2.45	1.45	0.17	14372	2.02	61.7	2.45	1.45	0.17	14372	70.47	2.02	96.19				
1.35		10.0	5.53	1.83	0.44	13799	2.65	71.7	2.88	1.50	0.21	14292	81.44	2.10	94.59				
1.35		4.5	7.32	2.03	0.68	13517	3.00	76.2	3.14	1.53	0.24	14246	86.27	2.15	93.48				
1.40		8.5	13.78	2.61	1.39	12454	4.19	84.7	4.21	1.64	0.35	14066	94.68	2.33	89.19				
1.60		3.5	22.61	3.55	2.13	10704	6.63	88.2	4.94	1.72	0.42	13933	97.66	2.47	86.48				
1.80		11.8	66.61	21.76	20.18	2494	174.50	100.0	12.15	4.08	2.75	12563	100.00	6.49	0.00				

WASHABILITY DATA
SEAM: PITTSBURGH No. 8
BELMONT COUNTY, OHIO

ADDITIONAL WASHABILITY

ORIGINAL SAMPLE, CRUSHED TO 325 M TOPSIZE

ELEMENTARY DATA										COMPUTED DATA: CUMULATIVE									
SINK	FLOAT	(%)	WTX	(%)	ASH	TOT SUL	(%)	PYR SUL	(%)	ASH	TOT SUL	(%)	PYR SUL	(%)	BTU	BTU REC	(%)	# SO2/ M BTU	# SO2/ XPYR SUL
1.30	1.30	50.6	1.86	1.43	0.06	1.43	0.06	1.43	0.06	1.86	1.43	0.06	1.43	0.06	14541	57.79	1.97	1.97	98.75
1.35	1.35	16.6	4.32	1.59	0.19	1.404	2.26	67.2	1.47	2.47	1.47	0.09	1.47	0.09	14418	76.10	2.04	2.04	97.35
1.40	1.40	7.2	6.53	1.65	0.36	13448	2.45	74.4	2.86	2.86	1.49	0.12	1.49	0.12	14324	83.71	2.08	2.08	96.38
1.40	1.60	11.1	11.19	1.92	0.70	12698	3.02	85.5	3.94	3.94	1.54	0.19	1.54	0.19	14113	94.78	2.19	2.19	93.17
1.60	1.80	3.4	25.76	2.84	1.65	989	5.74	88.9	4.78	4.78	1.59	0.25	1.59	0.25	13952	97.42	2.28	2.28	90.86
1.80		11.1	65.99	21.15	19.85	2957	143.05	100.0	11.57	11.57	3.76	2.42	3.76	2.42	12731	100.00	5.91	5.91	0.00

WASHABILITY DATA
SEAM: PITTSBURGH No. 8
BELMONT COUNTY, OHIO

ADDITIONAL WASHABILITY

ORIGINAL SAMPLE, CRUSHED TO 400 M TOPSIZE

ELEMENTARY DATA										COMPUTED DATA: CUMULATIVE									
SINK	FLOAT	(%)	WTX	(%)	ASH	TOT SUL	(%)	PYR SUL	(%)	ASH	TOT SUL	(%)	PYR SUL	(%)	BTU	BTU REC	(%)	# SO2/ M BTU	# SO2/ XPYR SUL
1.30	1.30	39.4	1.73	1.43	0.05	1.43	0.05	1.43	0.05	1.73	1.43	0.05	1.43	0.05	14400	46.31	1.99	1.99	99.18
1.35	1.35	15.8	3.57	1.48	0.12	13977	2.12	55.2	2.26	2.26	1.44	0.07	1.44	0.07	14279	64.34	2.02	2.02	98.39
1.40	1.40	8.9	5.46	1.55	0.23	13504	2.30	64.1	2.70	2.70	1.46	0.09	1.46	0.09	14171	74.15	2.06	2.06	97.54
1.40	1.60	13.2	9.23	1.68	0.50	12570	2.67	77.3	3.82	3.82	1.50	0.16	1.50	0.16	13698	87.69	2.15	2.15	94.80
1.60	1.80	12.2	23.02	2.54	1.42	9967	5.10	89.5	6.43	6.43	1.64	0.33	1.64	0.33	13362	97.61	2.45	2.45	87.59
1.80		10.5	66.11	25.69	20.06	2784	184.55	100.0	12.70	12.70	4.16	2.40	4.16	2.40	12251	100.00	6.80	6.80	-0.00

WASHABILITY DATA
SEAM: UPPER FREEPORT PA
INDIANA COUNTY, PENNSYLVANIA

ADDITIONAL WASHABILITY

ORIGINAL SAMPLE, CRUSHED TO 100 M TOPSIZE

ELEMENTARY DATA										COMPUTED DATA: CUMULATIVE									
SINK	FLOAT	(%)	WT%	(%)	ASH	TOT SUL	PYR SUL	(%)	# SO2/ M BTU	BTU	WT%	(%)	ASH	TOT SUL	PYR SUL	(%)	BTU	BTU REC	# SO2/ M BTU REJECTED
1.30	1.30	51.0	51.0	1.88	0.86	0.05	0.05	0.86	1.12	15325	51.0	1.88	0.86	0.05	0.05	58.36	15325	58.36	1.12
1.35	1.35	12.7	12.7	5.85	1.00	0.17	0.17	0.89	1.37	14613	63.7	2.67	0.89	0.07	0.07	72.21	15183	72.21	1.17
1.35	1.40	10.2	10.2	10.39	1.10	0.25	0.25	0.92	1.60	13784	73.9	3.74	0.92	0.10	0.10	82.71	14990	82.71	1.22
1.40	1.60	13.0	13.0	19.15	1.22	0.52	0.52	0.96	2.00	12219	86.9	6.04	0.96	0.16	0.16	94.57	14575	94.57	1.32
1.60	1.80	4.0	4.0	33.32	1.89	1.05	1.05	1.00	3.98	9494	90.9	7.24	1.00	0.20	0.20	97.41	14352	97.41	1.40
1.80		9.1	9.1	65.42	14.19	13.65		2.20	74.33	3818	100.0	12.54	2.20	1.42	1.42	100.00	13393	100.00	3.29
																			0.00

WASHABILITY DATA
SEAM: UPPER FREEPORT PA
INDIANA COUNTY, PENNSYLVANIA

ADDITIONAL WASHABILITY

ORIGINAL SAMPLE, CRUSHED TO 200 M TOPSIZE

ELEMENTARY DATA										COMPUTED DATA: CUMULATIVE									
SINK	FLOAT	(%)	WT%	(%)	ASH	TOT SUL	PYR SUL	(%)	# SO2/ M BTU	BTU	WT%	(%)	ASH	TOT SUL	PYR SUL	(%)	BTU	BTU REC	# SO2/ M BTU REJECTED
1.30	1.30	54.7	54.7	1.81	0.83	0.03	0.03	0.83	1.09	15280	54.7	1.81	0.83	0.03	0.03	62.94	15280	62.94	1.09
1.35	1.35	10.4	10.4	5.32	0.99	0.11	0.11	0.86	1.36	14599	65.1	2.37	0.86	0.04	0.04	74.37	15171	74.37	1.13
1.35	1.40	7.4	7.4	8.81	1.11	0.29	0.29	0.88	1.59	13938	72.5	3.03	0.88	0.07	0.07	82.14	15045	82.14	1.17
1.40	1.60	13.2	13.2	16.03	1.22	0.30	0.30	0.93	1.92	12681	85.7	5.03	0.93	0.10	0.10	94.74	14681	94.74	1.27
1.60	1.80	3.9	3.9	31.67	1.65	0.76	0.76	0.96	3.37	9787	89.6	6.19	0.96	0.13	0.13	97.62	14468	97.62	1.33
1.80		10.4	10.4	66.48	13.95	13.62		2.32	91.69	3043	100.0	12.46	2.32	1.54	1.54	100.00	13280	100.00	3.49
																			-0.00

WASHABILITY DATA
SEAM: UPPER FREEPORT PA
INDIANA COUNTY, PENNSYLVANIA

ADDITIONAL WASHABILITY

ORIGINAL SAMPLE, CRUSHED TO 325 M TOPSIZE

ELEMENTARY DATA										COMPUTED DATA: CUMULATIVE																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																							
SPECIFIC GRAVITY		(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)

WASHABILITY DATA
SEAM: UPPER FREEPORT PA
INDIANA COUNTY, PENNSYLVANIA

ADDITIONAL WASHABILITY

ORIGINAL SAMPLE, CRUSHED TO 400 M TOPSIZE

ELEMENTARY DATA										COMPUTED DATA: CUMULATIVE																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																	
SPECIFIC GRAVITY		(%)	(%)	(%)	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)	(%)	(%)	# SO2/ M BTU	BTU	(%)	(%)

WASHABILITY DATA
SEAM: UPPER FREEPORT
GRANT COUNTY, WEST VIRGINIA

ORIGINAL SAMPLE UNCRUSHED

4" X 0 COMPOSITE

ELEMENTARY DATA

COMPUTED DATA: CUMULATIVE

SPECIFIC GRAVITY		(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
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WASHABILITY DATA
SEAM: UPPER FREEPORT
GRANT COUNTY, WEST VIRGINIA

SAMPLE CRUSHED TO 1-1/2" TOPSIZE

1-1/2" X 0 COMPOSITE

ELEMENTARY DATA

COMPUTED DATA: CUMULATIVE

SINK		SPECIFIC GRAVITY		(%)	WT%	(%)	ASH	(%)	TOT SUL	(%)	PYR SUL	BTU	# SO2/ M BTU	(%)	WT%	(%)	ASH	(%)	TOT SUL	(%)	PYR SUL	BTU	BTU REC	(%)	# SO2/ M BTU	XPYR SUL REJECTED
1.30		1.30		33.8	23.8	2.36	7.10	0.73	0.13	0.73	0.13	15400	0.95	33.8	2.36	0.73	0.13	0.73	0.13	0.73	0.13	15400	39.59	0.95	98.05	
1.35		1.35		23.8	11.8	7.10	11.73	1.11	0.48	1.11	0.48	14570	1.52	57.6	4.32	0.89	0.27	0.89	0.27	0.89	0.27	15057	65.96	1.18	92.75	
1.35		1.40		11.8	16.2	11.73	18.79	1.89	1.25	1.89	1.25	13691	2.77	69.4	5.58	1.06	0.44	1.06	0.44	1.06	0.44	14824	78.28	1.43	85.93	
1.40		1.60		4.0	4.0	18.79	32.96	3.26	2.86	3.26	2.86	12420	5.24	85.6	9.19	1.66	1.07	1.66	1.07	1.66	1.07	14369	93.62	2.05	64.47	
1.60		1.80		10.4	10.4	64.03	13.43	11.53	4.87	5.58	4.87	9863	11.32	89.6	100.0	14.90	2.88	2.16	2.88	2.16	14166	96.61	2.34	55.51		
1.80												4273	62.88	100.0								13138	99.99	4.39	0.01	

1/8" x 0 COMPOSITE

WASHABILITY DATA
SEAM: UPPER FREEPORT
GRANT COUNTY, WEST VIRGINIA

SAMPLE COMMINUTED TO 28 M TOPSIZE

28 M X 0 COMPOSITE

[illegible]

WASHABILITY DATA
SEAM: UPPER FREEPORT
GRANT COUNTY, WEST VIRGINIA

SAMPLE CONTINUED TO 200 M TOPSIZE

SIZE FRACTION: 200M X 0 - 100.00 %

ELEMENTARY DATA

COMPUTED DATA: CUMULATIVE

SINK	FLOAT	(%)	WT%	ASH	TOT SUL	PYR SUL	BTU	# SO2/ M BTU	WT%	ASH	TOT SUL	PYR SUL	BTU	BTU REC	# SO2/ M BTU REJECTED
1.30	1.30	24.8	15.1	1.39	0.64	0.02	15411	0.83	24.8	1.39	0.64	0.02	15411	29.57	0.83
1.35	1.35	17.8	17.8	2.31	0.75	0.06	15192	0.99	39.9	1.74	0.68	0.04	15328	47.25	0.89
1.40	1.40	20.0	20.0	4.26	0.79	0.11	14821	1.07	57.7	2.51	0.71	0.06	15172	67.61	0.94
1.60	1.60	7.7	20.78	9.99	1.01	0.17	13645	1.48	77.6	4.44	0.79	0.09	14780	88.64	1.07
1.80	1.80	14.7	62.77	20.78	1.46	0.47	11589	2.52	85.3	5.90	0.85	0.12	14493	95.50	1.17
							3961	76.34	100.0	14.27	2.95	1.77	12944	100.00	4.56
															0.00

WASHABILITY: RAW COAL
SEAM: KENTUCKY NO. 9
HOPKINS COUNTY, KENTUCKY

ORIGINAL SAMPLE

4" x 0 COMPOSITE

ELEMENTARY DATA

COMPUTED DATA: CUMULATIVE

SINK	FLOAT	(%)	WT%	ASH	TOT SUL	PYR SUL	BTU	M BTU	WT%	ASH	TOT SUL	PYR SUL	BTU	BTU REC	(%)	# SO2/ M BTU	# SO2/ 2PYR SUL
1.30		30.9	30.9	4.36	2.38	0.63	14075	3.38	30.9	4.36	2.38	0.63	14075	35.02	35.02	3.38	93.22
1.35		35.9	35.9	7.86	2.76	1.11	13508	4.09	66.8	6.24	2.58	0.89	13771	74.06	74.06	3.75	79.39
1.35		13.2	13.2	11.81	4.05	2.57	12842	6.30	80.0	7.16	2.83	1.16	13618	87.68	87.68	4.15	67.63
1.40		7.7	7.7	16.70	7.25	5.92	11964	12.12	87.7	8.00	3.21	1.56	13472	95.11	95.11	4.77	51.76
1.60		2.5	2.5	25.11	9.98	9.57	10533	18.94	90.2	8.47	3.40	1.80	13391	97.22	97.22	5.08	43.46
1.80		9.8	9.8	66.55	13.25	12.82	3539	74.88	100.0	14.14	4.36	2.88	12429	100.00	100.00	7.02	0.00

WASHABILITY: RAW COAL
SEAM: KENTUCKY NO. 9
HOPKINS COUNTY, KENTUCKY

SAMPLE CRUSHED TO 1-1/2" TOPSIZE

1-1/2" x 0 COMPOSITE

ELEMENTARY DATA

COMPUTED DATA: CUMULATIVE

SINK	FLOAT	(%)	WT%	ASH	TOT SUL	PYR SUL	BTU	M BTU	WT%	ASH	TOT SUL	PYR SUL	BTU	BTU REC	(%)	# SO2/ M BTU	# SO2/ 2PYR SUL
1.30		29.9	29.9	3.98	2.36	0.56	14125	3.34	29.9	3.98	2.36	0.56	14125	33.87	33.87	3.34	94.20
1.35		38.4	38.4	7.41	2.75	1.08	13571	4.06	68.3	5.91	2.58	0.85	13814	75.59	75.59	3.74	79.99
1.35		12.4	12.4	11.79	4.09	2.52	12864	6.36	80.7	6.81	2.81	1.11	13668	88.32	88.32	4.11	69.30
1.40		7.3	7.3	16.37	6.67	5.45	12040	11.09	86.0	7.61	3.13	1.47	13533	95.40	95.40	4.63	55.54
1.60		2.1	2.1	24.62	10.41	9.67	10538	19.76	90.1	8.01	3.30	1.66	13462	97.19	97.19	4.91	48.52
1.80		9.9	9.9	65.73	14.97	14.32	3563	84.04	100.0	13.70	4.45	2.91	12486	100.00	100.00	7.13	0.00

1/8" X 0 COMPOSITE

ELEMENTARY DATA						COMPUTED DATA: CUMULATIVE							
SINK GRAVITY SINK FLOAT	(%) WT%	(%) ASH	(%) TOT SUL	(%) PYR SUL	# SO2/ M BTU	(%) WT%	(%) ASH	(%) TOT SUL	(%) PYR SUL	# SO2/ M BTU	(%) BTU REC	(%) SO2/ M BTU REJECTED	%PYR SUL REJECTED
1.30	60.8	4.98	2.40	0.70	13994	60.8	4.98	2.40	0.70	13994	69.32	3.43	86.51
1.35	11.9	9.79	3.28	1.72	13217	72.7	5.77	2.54	0.87	13866	82.16	3.67	80.01
1.35	6.7	12.66	4.17	2.71	12705	79.4	6.35	2.68	1.02	13768	89.11	3.89	74.23
1.40	6.4	18.23	6.07	4.95	11735	85.8	7.24	2.93	1.32	13616	95.24	4.31	64.15
1.60	2.3	23.83	7.39	6.36	10731	88.1	7.67	3.05	1.44	13542	97.22	4.50	59.60
1.80	11.9	67.18	16.23	15.73	2662	100.0	14.76	4.62	3.15	12268	100.00	7.53	0.00

WASHABILITY: RAW COAL
SEAM: KENTUCKY NO. 9
HOPKINS COUNTY, KENTUCKY

SAMPLE COMMUNITED TO 28 MESH TOPSIZE

28 M X O COMPOSITE

[illegible]

WASHABILITY: RAW COAL
SEAM: KENTUCKY NO. 9
HOPKINS COUNTY, KENTUCKY

SAMPLE COMMUNITED TO 200 MESH TOPSIZE

SIZE FRACTION: 200M X 0 100.00 % OF ORIGINAL SAMPLE

ELEMENTARY DATA										COMPUTED DATA: CUMULATIVE									
SINK	FLOAT	(%)	WT%	ASH	TOT SUL	PYR SUL	BTU	# SO2/ M BTU	WT%	ASH	TOT SUL	PYR SUL	BTU	(%)	BTU REC	(%)	# SO2/ M BTU	BTU REJECTED	# SO2/ M BTU
1.30	1.35	45.1	9.9	2.13	2.01	0.15	14223	2.83	45.1	2.13	2.01	0.15	14223	53.03	53.03	2.83	2.83	97.74	97.74
1.35	1.40	5.5	5.5	5.16	2.05	0.43	13732	2.99	55.0	2.68	2.02	0.20	14135	64.27	64.27	2.85	2.85	96.32	96.32
1.40	1.60	21.4	21.4	6.30	2.09	0.53	13471	3.10	60.5	3.00	2.02	0.23	14074	70.39	70.39	2.88	2.88	95.35	95.35
1.60	1.80	3.6	3.6	9.97	2.36	0.85	13104	3.60	81.9	4.82	2.11	0.39	13821	93.57	93.57	3.06	3.06	89.28	89.28
		14.5	14.5	17.93	3.66	2.86	10697	6.84	85.5	5.38	2.18	0.50	13689	96.76	96.76	3.18	3.18	85.84	85.84
				67.81	18.38	17.74	2705	135.90	100.0	14.43	4.53	3.00	12097	100.00	100.00	7.46	7.46	0.00	0.00

WASHABILITY: RAW COAL
SEAM: WYODAK
CAMPBELL COUNTY, WYOMING

ORIGINAL SAMPLE

4" X 0 COMPOSITE

ELEMENTARY DATA										COMPUTED DATA: CUMULATIVE									
SINK	FLOAT	(%)	WT%	ASH	TOT SUL	PYR SUL	BTU	M BTU	# S02/	WT%	ASH	TOT SUL	PYR SUL	BTU	BTU REC	(%)	# S02/	M BTU	XPYR SUL
1.30		94.4	94.4	7.02	0.51	0.05	12021	0.84		94.4	7.02	0.51	0.05	12021	95.15		0.84		40.42
1.35		2.2	2.2	11.44	0.66	0.13	11356	1.16		96.5	7.12	0.51	0.05	12006	97.22		0.85		36.75
1.35	1.40	2.1	2.1	18.18	0.87	0.29	10481	1.65		98.7	7.35	0.52	0.05	11973	99.09		0.87		28.44
1.40	1.60	1.1	1.1	24.17	0.96	0.45	9665	1.99		99.8	7.54	0.52	0.06	11947	99.99		0.88		21.70
1.60	1.80	0.1	0.1	40.16	1.84	1.32	6459	5.06		99.9	7.58	0.52	0.06	11942	100.04		0.88		19.88
1.80		0.2	0.2	70.77	8.51	7.15	2278	74.73		100.1	7.71	0.54	0.07	11922	100.08		0.91		0.00

WASHABILITY: RAW COAL
SEAM: WYODAK
CAMPBELL COUNTY, WYOMING

SAMPLE CRUSHED TO 1-1/2" TOPSIZE

1-1/2" X 0 COMPOSITE

ELEMENTARY DATA										COMPUTED DATA: CUMULATIVE									
SINK	FLOAT	(%)	WT%	ASH	TOT SUL	PYR SUL	BTU	M BTU	# S02/	WT%	ASH	TOT SUL	PYR SUL	BTU	BTU REC	(%)	# S02/	M BTU	XPYR SUL
1.30		94.6	94.6	6.98	0.49	0.04	12143	0.80		94.6	6.98	0.49	0.04	12143	95.48		0.80		48.54
1.35		2.4	2.4	8.12	0.70	0.25	11222	1.25		97.0	7.00	0.49	0.04	12120	97.70		0.81		40.01
1.35	1.40	1.6	1.6	17.00	0.78	0.35	10551	1.49		98.6	7.16	0.50	0.05	12095	99.06		0.82		32.22
1.40	1.60	1.0	1.0	24.61	0.80	0.34	9583	1.67		99.6	7.34	0.50	0.05	12069	99.88		0.83		27.25
1.60	1.80	0.2	0.2	42.47	3.14	2.52	6285	10.00		99.8	7.40	0.50	0.06	12059	99.97		0.83		20.92
1.80		0.2	0.2	80.68	7.30	6.53	1562	93.53		100.0	7.57	0.52	0.07	12035	100.00		0.86		0.00

WASHABILITY: RAW COAL
SEAM: WYODAK
CAMPBELL COUNTY, WYOMING

SAMPLE COMMUNITED TO 28 MESH TOPSIZE

28 M X 0 COMPOSITE

ELEMENTARY DATA

COMPUTED DATA: CUMULATIVE

SPECIFIC GRAVITY		(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
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WASHABILITY: RAW COAL
SEAM: WYODAK
CAMPBELL COUNTY, WYOMING

SAMPLE COMMUNITED TO 200 MESH TOPSIZE

SIZE FRACTION: 200M X 0 100.00 % OF ORIGINAL SAMPLE

ELEMENTARY DATA

COMPUTED DATA: CUMULATIVE

SPECIFIC GRAVITY		(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
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WASHABILITY: RAW COAL
SEAM: WYODAK
CAMPBELL COUNTY, WYOMING

SAMPLE CRUSHED TO 1/8" TOPSIZE

1/8" X 0 COMPOSITE

ELEMENTARY DATA

COMPUTED DATA: CUMULATIVE

SPECIFIC GRAVITY	SINK	FLOAT	ELEMENTARY DATA										COMPUTED DATA: CUMULATIVE									
			(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
			WT%	ASH	TOT SUL	PYR SUL	BTU	M BTU	WT%	ASH	TOT SUL	PYR SUL	BTU	M BTU	WT%	ASH	TOT SUL	PYR SUL	BTU	M BTU	WT%	ASH
1.30			85.9	6.13	0.42	0.01	12142	0.70	85.9	6.13	0.42	0.01	12142	0.70	85.9	6.13	0.42	0.01	12142	0.70	85.9	6.13
1.35			5.9	9.64	0.50	0.07	11136	0.89	91.8	6.36	0.43	0.01	12077	0.71	91.8	6.36	0.43	0.01	12077	0.71	91.8	6.36
1.40			4.0	11.64	0.47	0.09	10802	0.87	95.8	6.58	0.43	0.02	12024	0.72	95.8	6.58	0.43	0.02	12024	0.72	95.8	6.58
1.60			3.3	18.69	0.56	0.21	9807	1.15	99.1	6.98	0.44	0.02	11950	0.73	99.1	6.98	0.44	0.02	11950	0.73	99.1	6.98
1.80			0.5	38.22	1.43	1.11	6860	4.18	99.6	7.13	0.44	0.03	11926	0.74	99.6	7.13	0.44	0.03	11926	0.74	99.6	7.13
1.80			0.5	75.50	9.11	7.90	2166	84.08	100.0	7.44	0.48	0.06	11882	0.81	100.0	7.44	0.48	0.06	11882	0.81	100.0	7.44

Figure 4.19 Particle size distribution for Illinois No. 6 coal washability
test samples ground to 100, 200 and 400 mesh top size fraction.

100 MESH X 0		200 MESH X 0		400 MESH X 0	
Size	Cum. % pass.	Size	Cum. % pass.	Size	Cum. % pass.
2.8	4.1	2.8	6.1	2.8	17.3
3.9	6.6	3.9	10.2	3.9	23.4
5.5	10.4	5.5	12.8	5.5	33.5
7.8	15.4	7.8	17.4	7.8	44.1
11.0	21.8	11.0	25.6	11.0	55.9
16.0	29.9	16.0	35.0	16.0	69.1
22.0	37.6	22.0	44.7	22.0	80.8
31.0	47.3	31.0	58.1	31.0	92.2
44.0	56.9	44.0	75.4	44.0	100.0
62.0	65.0	62.0	90.0		
88.0	78.6	88.0	100.0		

Figure 4.23 Particle size distribution for Pittsburgh No. 8 coal washability
test samples ground to 100, 200 and 400 mesh top size fraction.

100 MESH X 0		200 MESH X 0		400 MESH X 0	
Size	Cum. % pass.	Size	Cum. % pass.	Size	Cum. % pass.
2.8	2.1	2.8	4.5	2.8	9.9
3.9	4.3	3.9	7.1	3.9	14.6
5.5	8.3	5.5	11.7	5.5	21.8
7.8	13.1	7.8	18.3	7.8	31.0
11.0	18.8	11.0	26.8	11.0	43.5
16.0	25.5	16.0	35.5	16.0	56.8
22.0	37.6	22.0	47.9	22.0	71.6
31.0	41.3	31.0	63.0	31.0	89.0
44.0	51.3	44.0	78.6	44.0	100.0
62.0	60.9	62.0	92.2		
88.0	72.2	88.0	99.8		
105.0	84.4	125.0	100.0		
150.0	97.4				

Figure 4.28 Particle size distribution for Upper Freeport PA coal washability
test samples ground to 100, 200 and 400 mesh top size fraction.

100 MESH X 0		200 MESH X 0		400 MESH X 0	
Size	Cum. % pass.	Size	Cum. % pass.	Size	Cum. % pass.
2.8	4.7	2.8	10.7	2.8	13.8
3.9	7.3	3.9	14.5	3.9	19.1
5.5	11.8	5.5	21.9	5.5	28.0
7.8	17.2	7.8	30.5	7.8	39.2
11.0	24.1	11.0	40.8	11.0	51.2
16.0	31.7	16.0	53.2	16.0	64.4
22.0	40.0	22.0	66.1	22.0	77.7
31.0	49.6	31.0	80.5	31.0	90.7
44.0	58.6	44.0	93.7	44.0	100.0
62.0	66.2	62.0	100.0		
150.0	95.9				

Figure 5.1 Film flotation partition curves for the three base coals.

<u>mN/m</u>	<u>YIELD, PERCENT</u>		
	<u>Ill. 6</u>	<u>Pitt. 8</u>	<u>UFGA</u>
33.0	0.0	0.0	0.0
36.5	0.0	2.4	1.0
40.5	2.8	14.3	4.6
45.0	14.4	44.4	41.0
48.0	48.7	62.9	64.8
51.0	54.4	81.7	86.0
54.5	67.7	92.6	97.1
58.8	87.0	96.9	99.0
64.5	84.3	97.9	100.0
72.7	96.6	99.1	100.0

Figure 5.2 Induction time curves indicating the percentage of fruitful contacts as a function of contact time in microseconds for a 100 x 150 mesh sample of Pittsburgh No. 8 coal.

<u>Contact time</u> <u>microseconds</u>	<u>Fruitful contacts,</u> <u>percent</u>
1100	90
1000	100
900	80
800	80
700	70
600	50
550	45
500	30
450	30
300	15
250	20
200	10

Figure 5.3 Induction time curves indicating the percentage of fruitful contacts as a function of contact time in microseconds for 100 x 150 mesh samples of three base coals.

<u>ILLINOIS NO. 6</u>		<u>PITTSBURGH NO. 8</u>	
<u>Contact time,</u> <u>microsecond</u>	<u>Fruitful contacts,</u> <u>percent</u>	<u>Contact time,</u> <u>microseconds</u>	<u>Fruitful contacts,</u> <u>percent</u>
1400	90	1100	90
1400	75	1000	100
1200	70	900	80
1200	60	800	80
1000	55	700	70
800	50	600	50
500	40	550	45
400	15	500	30
400	20	400	30
		300	15
		250	20
		200	10

<u>UPPER FREEPORT PA</u>	
<u>Contact time</u> <u>microseconds</u>	<u>Fruitful contacts</u> <u>percent</u>
350	100
300	90
250	80
200	80
170	70
150	65
120	40
100	55
80	45
70	40
50	30
40	20

Figure 5.4 Induction time curves indicating the percentage of fruitful contacts as a function of contact time for Illinois No. 6 for both undeslimed and deslimed fresh ground samples.

UNDESIMED		DESIMED	
Contact time, ms	Fruitful contact, %	Contact time, ms	Fruitful contact, %
200	14	100	6
250	22	150	14
300	18	200	14
350	28	250	20
400	24	300	22
420	32	350	34
450	46	400	41
470	46	450	44
500	50	470	51
520	56	500	52
550	51	550	60
570	54	600	66
600	62	650	60
620	60	700	64
670	65	750	82
700	78	800	74
800	74	850	70
900	84	900	80

Figure 5.5 Induction time curves indicating the percentage of fruitful contacts as a function of contact time for Pittsburgh No. 8 coal for undeslimed and deslimed fresh ground samples.

UNDESIMED		DESIMED	
Contact time, ms	Fruitful contact, %	Contact time, ms	Fruitful contact, %
100	22	50	18
150	36	70	18
200	42	100	32
250	46	120	38
260	50	150	46
270	50	170	46
290	56	180	50
300	53	200	52
320	56	210	58
350	59	220	62
400	62	250	72
500	74	280	84
600	76	300	90
		330	90
		350	94

Figure 5.6 Induction time curves indicating the percentage of fruitful contacts as a function of contact time for Upper Freeport PA undeslimed and deslimed fresh ground samples.

UNDESIMED		DESIMED	
Contact time, ms	Fruitful contact, %	Contact time, ms	Fruitful contact, %
50	26	50	25
70	34	60	35
80	38	70	44
90	42	80	52
100	46	90	52
110	55	100	61
130	64	150	78
150	73	200	90
200	86	250	94
250	90	300	98
300	96		

Figure 5.7 Zeta Potential versus pH Curve for Illinois No. 6 Coal.

2.00E ⁻³ M KNO ₃		TRIPLY DISTILLED WATER	
pH	Zeta pot., mV	pH	Zeta pot., mV
2.75	-1.60	2.83	5.10
4.03	-16.00	3.42	-4.40
5.89	-22.30	6.78	-31.10
5.95	-24.20	7.65	-38.70
7.28	-39.60	9.27	-39.50
7.94	-41.40	10.89	-58.90
8.63	-44.60		
9.02	-47.20		
10.31	-52.50		
10.98	-55.80		

Figure 5.8 Zeta Potential versus pH Curve for Pittsburgh No. 8 Coal.

2.00E ⁻³ M KNO ₃		TRIPLY DISTILLED WATER	
pH	Zeta pot., mV	pH	Zeta pot., mV
2.70	10.00	2.83	9.30
4.44	-5.57	4.50	-1.50
4.84	-2.50	7.15	-31.60
5.53	-21.80	7.55	-46.70
5.61	-21.90	9.99	-56.00
6.56	-23.10	10.88	-61.20
8.26	-50.15		
10.18	-61.10		
10.44	-69.00		
11.48	-58.20		

Figure 5.9 Zeta Potential versus pH Curve for Upper Freeport PA Coal.

2.00E ⁻³ M KNO ₃		TRIPLY DISTILLED WATER	
pH	Zeta pot., mV	pH	Zeta pot., mV
2.82	24.07	2.80	18.70
4.57	3.90	5.40	-5.90
6.02	-17.20	7.20	-31.90
6.45	-15.40	7.40	-49.50
7.51	-34.80	8.10	-47.60
10.37	-75.40	11.30	-65.80
11.50	-71.60		

Figure 6.1 Comparison of the size distribution of the products obtained by grinding Illinois No. 6 coal in ball and rod mills for four minutes.

Size, mesh	CUMULATIVE WEIGHT PERCENT PASSING		
	Mill feed	Rod mill	Ball Mill
200	0.5	17.0	34.7
150	0.6	20.5	40.1
100	1.1	25.9	48.5
65	2.2	32.5	57.4
48	4.0	40.3	63.2
35	6.0	51.5	66.4
28	8.7	64.2	68.0
20	12.5	80.5	69.2
14	16.8	91.6	70.1
10	24.0	96.6	71.5
8	33.0	98.0	73.1
7	--	--	--
6	49.4	--	--
5	--	--	--
4	--	--	--
3.5	87.5	--	--

Figure 6.2 Particle size distribution of Illinois No. 6 coal ground in the rod mill for different grind times.

Size, Mesh	CUMULATIVE WEIGHT PERCENT PASSING FOR GRIND TIMES (IN MINUTES)								
	1	2	4	8	16	32	64	128	256
400	5.27	8.56	12.63	17.95	29.81	57.3	76.8	90.49	96.9
270	6.1	9.1	14.26	19.53	33.81	65.96	86.93	97.95	98.78
200	7.14	10.13	17.03	22.26	40.45	80.46	97.72	99.69	99.21
150	8.78	12.17	20.53	27.85	50.13	95.05	99.49	99.82	99.4
100	0.95	15.61	25.95	36.12	61.35	99.16	99.82	99.89	
65	13.75	20.95	32.54	47.19	75.84	99.89	99.92	99.93	
48	17.43	27.61	40.3	61.3	92.29	99.97	99.97	100.0	
35	24.5	36.26	51.57	81.38	98.95	100.0	100.0		
28	30.18	45.61	64.16	94.91	99.44				
20	38.14	58.57	80.55	98.05	99.54				
14	46.38	71.53	91.6	98.52	99.61				
10	58.85	85.46	96.59	98.92	99.65				
8	69.89	91.98	97.96	99.09	99.67				

Figure 6.3 Particle size distribution of Pittsburgh No. 8 coal ground in the rod mill for different grind times.

Size Mesh	CUMULATIVE WEIGHT PERCENT PASSING FOR GRIND TIMES (IN MINUTES)						
	1	2	4	8	16	32	128
400	5.99	9.26	--	--	--	--	--
270	7.08	10.72	--	--	--	--	--
200	8.65	13.11	18.57	31.73	44.08	60.67	98.82
150	10.81	16.55	22.89	37.67	51.89	68.32	99.78
100	13.51	21.17	28.86	46.00	63.42	81.66	99.87
65	16.95	26.89	37.19	56.56	77.47	96.3	--
48	21.25	34.08	46.58	69.85	92.09	99.7	--
35	26.94	42.71	58.87	85.43	99.23	99.94	--
28	33.68	53.18	72.9	96.51	99.93	99.97	--
20	42.00	66.75	88.58	99.74	99.98	99.98	--
14	50.81	79.55	95.67	99.91	--	--	--
10	62.98	91.94	97.42	--	--	--	--
8	74.9	95.95	97.76	--	--	--	--

Figure 6.4 Particle size distribution of Upper Freeport PA coal ground in the rod mill for different grind times.

Size Mesh	CUMULATIVE WEIGHT PERCENT PASSING FOR GRIND TIMES (IN MINUTES)		
	2	8	32
400	8.65	33.48	95.49
270	10.26	43.13	99.39
200	13.25	54.03	99.67
150	17.74	68.55	99.8
100	23.12	85.68	99.84
65	30.49	96.45	99.9
48	39.55	99.87	99.95
35	51.54	99.94	--
28	65.46	99.97	--
20	82.06	--	--
14	89.81	--	--
10	95.54	--	--
8	96.89	--	--

Figure 6.5 Effect of number of rods on the particle size distribution of ground product of Illinois No. 6 coal conforming to 95 percent passing through 28 mesh.

Size Mesh	CUMULATIVE WEIGHT PERCENT PASSING (NO. OF RODS & GRIND TIMES)			
	24/8	36/3.5	42/3	48/2.75
200	36.4	--	--	--
150	42.66	27.84	--	28.19
100	52.76	36.86	37.87	38.47
65	66.89	49.8	50.2	51.66
48	80.77	65.88	65.3	67.22
35	89.41	84.47	82.61	83.91
28	92.83	95.22	94.47	93.78
20	94.47	98.43	98.1	97.18
14	95.31	99.22	98.58	98.26
10	95.93	99.69	98.81	98.92
8	96.13	99.84	98.87	98.92

Figure 6.6 Effect of number of rods on the production of minus 200 mesh for Illinois No. 6 coal for a grind time of 8 minutes.

No. of Rods	CUMULATIVE PERCENT PASSING 200 MESH	
	Dry Grinding	Wet Grinding
24	36.4	29.07
36	47.1	51.56
42	51.4	60.56
48	53.6	64.92

Figure 6.7 Effect of the number of rods on 28-mesh material produced during dry grinding of Illinois No. 6 coal as a function of grinding time.

Time, min.	CUMULATIVE WEIGHT PERCENT PASSING 28 MESH	
	24 rods	42 rods
2	62.58	81.57
2	--	80.09
3	--	94.47
4	--	98.56
8	92.83	99.64
32	99.08	--

Figure 6.8 Effect of the number of rods on 200-mesh material produced during dry grinding of Illinois No. 6 coal as a function of grinding time.

Grind time, min.	CUMULATIVE WEIGHT PERCENT PASSING 200 MESH	
	24 rods	42 rods
2	16.84	16.3
2	--	18.43
4	--	30.87
8	36.4	51.37
16	--	73.87
32	63.79	92.64
48	--	95.92
64	73.00	--
128	86.00	--

Figure 6.9 Effect of the number of rods on 200-mesh material produced during the dry grinding of Upper Freeport PA coal as a function of grinding time.

24 RODS		42 RODS	
Grind time	Cum. %	Grind time	Cum. %
min.		min.	
30.00	84	2	31
48.84	95	15	90
		20	94
		21.85	95
		23	96

Figure 6.10 Effect of the number of rods on 200-mesh material produced during the wet grinding of Upper Freeport PA coal as a function of grinding time.

24 RODS		42 RODS	
Grind time	Cum. %	Grind time	Cum. %
min.		min.	
4	29	8	87
10	49	10	90
30	88	11	94
40	95	12	96

Figure 6.11 Size distribution of Illinois No. 6 coal prepared for washability testing and 200-mesh wet and dry-ground products prepared by standard grinding procedure.

Particle size, micron	Wash. Grind	Dry Grind	Wet Grind
3.9	9.0	15.64	6.25
5.5	13.2	23.74	9.82
7.8	17.2	30.78	13.77
11.0	24.2	39.49	19.21
16.0	36.0	47.86	25.54
22.0	49.8	57.33	34.43
31.0	66.3	67.57	45.18
40.0	--	76.09	58.11
44.0	88.0	--	--
48.0	--	84.3	69.99
62.0	99.7	89.17	78.75
88.0	96.75	93.15	--

Figure 6.12 - Measured and SEM-AIA predicted washability results for 200-mesh Illinois No. 6 coal.

SEM-AIA PREDICTED				WASHABILITY		
CMR %	PSR %	CMR %	AR %	CMR %	PSR %	AR %
99.6	31.0	99.6	25.0	27.5	99.07	96.8
98.8	44.5	99.2	36.5	28.7	99.03	96.6
98.5	51.0	98.5	43.5	36.7	98.65	94.5
97.3	62.0	97.3	53.5	86.1	92.57	74.2
95.3	71.0	96.1	63.0	92.7	88.86	62.0
93.5	78.5	90.0	79.0			
89.6	87.0	82.3	88.0			
82.3	93.0	52.7	100.0			
52.7	100.0					

Figure 6.13 - Measured and SEM-AIA predicted washability results for 200-mesh Pittsburgh No. 8 coal.

SEM-AIA PREDICTED				WASHABILITY		
CMR	PSR	CMR	AR	CMR	PSR	AR
<u>1</u>	<u>1</u>	<u>1</u>	<u>1</u>	<u>1</u>	<u>1</u>	<u>1</u>
99.6	31.0	99.6	33.0	88.51	96.19	87.56
99.2	39.0	98.8	40.0	79.27	94.59	83.0
98.8	49.0	98.1	48.0	82.02	93.48	80.31
98.1	55.0	96.9	53.5	92.36	89.19	70.65
96.9	63.0	93.8	64.0	95.44	86.48	64.14
93.8	72.0	89.2	75.0			
89.2	86.0	80.8	86.0			
80.8	93.0	53.1	100.0			
53.1	100.0					

Figure 6.14 - Measured and SEM-AIA predicted washability results for 200-mesh Upper Freeport PA coal

SEM-AIA PREDICTED				WASHABILITY		
CMR	PSR	CMR	AR	CMR	PSR	AR
<u>1</u>	<u>1</u>	<u>1</u>	<u>1</u>	<u>1</u>	<u>1</u>	<u>1</u>
99.6	39.5	99.2	33.0	61.35	98.93	92.05
99.2	48.5	98.1	45.0	72.6	98.19	87.62
98.1	57.0	96.9	53.5	80.31	96.79	82.37
96.9	66.0	95.4	63.5	92.97	94.21	65.4
95.4	76.0	92.3	73.0	96.02	92.28	55.48
92.3	85.0	89.2	81.5			
89.2	91.0	80.8	91.5			
80.8	97.0	63.8	100.0			
63.8	100.0					

Figure 7.3 Effect of collector and frother dosages on the flotation yield of 200-mesh wet-ground Pittsburgh No. 8 coal.

Collector, <u>lb/T</u>	Frother, <u>lb/T</u>	Flotation yield, <u>percent</u>
1.68	0.29	66.2
1.92	0.29	75.7
2.16	0.29	73.2
2.4	0.29	74.8
1.92	0.2	59.2
1.92	0.26	68.9
1.92	0.29	75.7
1.92	0.32	73.6
1.92	0.39	83.7

Figure 7.4 Flotation yield of the three base coals as a function of flotation time using standard collector and frother dosages as tabulated in Table 7.1.

Flotation time <u>min.</u>	FLOTATION YIELD, PERCENT		
	<u>Illinois No. 6</u>	<u>Pittsburgh No. 8</u>	<u>Upper Freeport PA</u>
0.5	32.34	22.6	29.6
1	51.75	41.5	49.1
2	66.00	65.8	67.2
4	75.8	76.1	77.2
8	81.21	81.3	84.3

Figure 7.9 Release analysis, washability data, and SEM-AIA data showing CMR (or Btu) recovery as a function of pyritic sulfur rejection for Illinois No. 6 coal.

FLOTABILITY (CMR)		WASHABILITY (BTU)		SEM-AIA	
PSR	CMR	PSR	CMR	PSR	CMR
<u>z</u>	<u>z</u>	<u>z</u>	<u>z</u>	<u>z</u>	<u>z</u>
0.0	100.0	99.1	27.5	8.0	100.0
58.3	94.7	99.0	28.7	18.5	99.2
67.6	93.5	98.7	36.7	28.0	98.5
73.9	90.3	92.6	86.1	45.0	95.4
78.2	87.1	88.9	92.7	72.0	86.5
81.4	79.1			95.0	60.0
89.2	56.3				
93.5	36.8				
98.7	11.2				
100.0	0.0				

Figure 7.10 Ash rejection as a function of (100-Yield) for Illinois No. 6 coal for the kinetic flotation tests, release analysis and washability studies.

FLOTABILITY		WASHABILITY	
100-Yield	Ash Rej. <u>z</u>	100-Yield	Ash Rej. <u>z</u>
0.0	0.0	0.0	0.0
12.3	49.0	15.0	61.45
14.8	58.1	18.8	67.62
18.5	64.8	34.9	80.47
22.0	69.3	42.0	85.7
29.4	73.6	65.5	93.7
50.2	84.1	100.0	100.0
67.6	90.3		
90.2	97.4		
100.0	100.0		

1-STEP FLOTATION	
100-Yield	Ash Rej. <u>z</u>
0.0	0.0
25.0	57.7
30.7	61.8
52.1	74.2
68.0	82.6
100.0	100.0

Figure 7.11 Pyritic sulfur rejection as a function of (100-Yield) for Illinois No. 6 for the kinetic flotation tests, release analysis and washability studies.

2-FLOTABILITY		1-WASHABILITY	
100-Yield	Pyr. S Rej. <u>z</u>	100-Yield	Pyr. S Rej. <u>z</u>
0.0	0.0	0.0	0.0
12.33	58.3	15.0	91.0
14.86	67.6	18.8	92.49
18.55	73.9	34.9	95.9
22.01	78.2	42.0	96.73
50.22	89.2	65.5	98.28
67.57	93.5	100.0	100.0
90.21	98.7		
100.0	100.0		

1-STEP FLOTATION	
100-Yield	Pyr. S Rej. <u>z</u>
0.0	0.0
25.0	57.7
30.7	61.8
52.1	74.4
68.0	82.8
100.0	100.0

Figure 7.13 Release analysis, washability data, and SEM-AIA data showing CMR (or Btu recovery) as a function of pyritic sulfur rejection for Pittsburgh No. 8 coal.

FLOTABILITY		WASHABILITY		SEM-AIA	
PSR	CMR	PSR	CMR	PSR	CMR
%	%	%	%	%	%
41.4	97.5	96.2	68.5	25.0	99.6
57.7	93.1	94.6	79.3	34.0	99.2
68.1	88.0	93.5	82.0	42.0	96.2
83.4	65.6	89.2	92.4	76.5	82.3
88.8	49.6	86.5	95.4	96.5	57.7
93.3	32.6			96.5	57.7
97.0	17.3				
99.6	3.3				

Figure 7.17 Release analysis, washability data and SEM-AIA data showing CMR as (or Btu recovery) as a function of pyritic sulfur rejection for Upper Freeport PA coal.

FLOTABILITY		WASHABILITY		SEM-AIA	
PSR	CMR	PSR	CMR	PSR	CMR
%	%	%	%	%	%
41.8	97.5	98.9	61.4	15.0	100.0
59.6	95.3	98.2	72.6	32.0	99.0
70.1	93.4	96.8	80.3	35.0	98.5
82.2	81.7	94.2	93.0	55.0	95.4
89.6	63.5	92.3	96.0	78.5	88.5
94.7	41.0			96.5	68.5

Figure 7.18 Relationship between combustible material recovery and Btu recovery, for Illinois No. 6 coal.

Sample No.	Yield %	Ash %	Btu/lb	CMR %	Btu rec. %
1	25.0	7.34	12888	27.6	28.2
2	48.0	7.84	12888	52.4	53.5
3	60.7	6.85	12951	66.6	68.0
4	70.2	4.85	12712	76.4	77.2
5	78.7	9.18	12665	84.7	86.3
Feed			11554		

Figure 7.19 Relationship between combustible material recovery and Btu recovery for Pittsburgh No. 8 coal.

Sample No.	Yield %	Ash %	Btu/lb	CMR %	Btu Rec. %
1	25.6	4.31	13960	27.5	28.1
2	54.0	4.53	13965	58.1	59.3
3	73.8	4.74	13894	78.7	80.6
4	82.2	5.08	13809	88.4	89.2
5	91.7	7.42	13509	96.1	97.7
Feed			12726		

Figure 7.20 Relationship between combustible material recovery and Btu recovery for Upper Freeport PA coal.

Sample No.	Yield %	Ash %	Btu/lb	CMR %	Btu Rec. %
1	24.2	3.54	15087	26.6	29.7
2	51.7	5.25	14943	54.7	57.2
3	64.8	50.30	14898	70.3	71.5
4	79.9	6.34	14706	85.7	87.0
5	81.3	4.45	14053	87.7	89.4
6	81.6	4.77	14060	87.9	89.6
7	92.6	8.16	14327	96.8	98.2
Feed			13507		

Figure 7.21 Correlation between pyritic sulfur content of Illinois No. 6 coal determined using the ASTM procedure with the value calculated using Equation 3 for flotation samples of Illinois No. 6 coal.

Calculated Pyr. S	Determined Pyr. S
1.13	1.15
1.1	1.04
1.08	1.1
2.39	2.37
1.08	1.11
0.95	0.92
1.02	1.05
1.5	1.46
1.8	1.87
1.05	1.05
1.18	1.16
1.6	1.56
2.07	2.09
0.93	0.88
1.16	1.12
1.87	1.82
1.11	1.17
1.34	1.34
2.11	2.12
2.05	2.09

Figure 7.22 Correlation between pyritic sulfur content of Pittsburgh No. 8 coal determined using the ASTM procedure with the value calculated from Equation 4 for flotation samples of Pittsburgh No. 8 coal.

Calc. Pyr. S	Det. Pyr. S	Calc. Pyr. S	Det. Pyr. S
<u>X</u>	<u>X</u>	<u>X</u>	<u>X</u>
2.76	2.73	1.31	1.26
2.5	2.51	1.23	1.19
1.35	1.38	1.27	1.22
2.55	2.59	1.17	1.15
1.38	1.43	1.19	1.19
1.32	1.31	2.42	2.42
1.22	1.24	1.18	1.24
1.42	1.46	1.24	1.27
1.31	1.3	1.2	1.22
1.35	1.38	1.13	1.12
1.21	1.22	2.4	2.38
2.78	2.76	1.46	1.46
1.38	1.38	1.02	1.03
1.48	1.47	1.02	1.02
1.34	1.29	1.02	1.02
2.95	2.93	2.26	2.21
1.24	1.28	1.19	1.16
1.3	1.35	1.13	1.13
1.31	1.36	1.16	1.14
1.32	1.4	1.38	1.39
1.56	1.54	1.4	1.36
1.59	1.63	0.39	0.32
1.56	1.62	0.57	0.51
2.35	2.38	0.62	0.6
1.3	1.31	0.8	0.8
1.51	1.52	0.92	0.94
1.37	1.42	1.73	1.82
		2.6	2.56

Figure 7.23 Correlation between pyritic sulfur content of Upper Freeport PA coal determined using the ASTM procedure with the value calculated from Equation 5 for flotation samples of Upper Freeport PA coal.

Calculated Pyr. S	Determined Pyr. S
<u>X</u>	<u>X</u>
0.39	0.37
0.4	0.4
0.53	0.53
1.14	1.14
0.44	0.45
0.48	0.48
0.52	0.53
0.91	0.91
0.44	0.43
0.46	0.46
0.56	0.53
0.13	0.14
0.25	0.25
0.35	0.35
0.59	0.61
1.41	1.41

Figure 7.24 Correlation between the pyritic sulfur content determined experimentally using the ASTM procedure and the value calculated using Equation 6 for washability samples of Pittsburgh No. 8 coal.

Exp. Pyr S. %	Calc. Pyr S. %
0.93	0.97
2.06	1.93
3.96	3.82
5.42	5.12
5.35	5.35
1.15	1.14
1.99	1.93
3.73	3.56
5.23	5.11
5	4.92
0.6	0.68
1.82	1.83
3.14	3.18
4.57	5.14
6.74	6.77
0.33	0.41
1.31	1.33
1.84	1.86
3.47	3.54
6.15	6.21

Figure 8.1 Effect of the grinding mode on the separation of non-pyritic minerals from coal and comparison of washability and flotation selectivity curves for 28 mesh grind.

DRY GRIND		WET GRIND		WASHABILITY	
CMR %	NPAR %	CMR %	NPAR %	CMR %	NPAR %
88.4	52.6	91.7	51.1	56.9	87.4
86.1	53.7	91.5	49.6	76.9	78.3
84.0	57.4	89.4	53.7	85.7	72.7
72.7	63.9	86.4	55.9	84.6	61.8
71.5	66.5	75.0	64.5	86.7	55.0
55.3	75.3	59.5	73.3		

Figure 8.2 Effect of the grinding mode on the separation of non-pyritic minerals from coal and comparison of washability and flotation selectivity curves for 200 mesh grind.

DRY GRIND		WET GRIND		WASHABILITY	
CMR %	NPAR %	CMR %	NPAR %	CMR %	NPAR %
87.9	53.9	90.6	58.4	60.4	88.3
80.9	60.5	85.6	64.0	67.7	85.4
80.5	61.9	78.7	69.5	77.1	81.2
75.7	64.2	77.7	68.9	95.1	63.0
70.9	67.0	74.9	71.3	96.4	59.4
50.2	77.1	53.7	80.9		

Figure 8.3 Comparison of flotation selectivity and washability curves for the 28 mesh and 200 mesh grinds of Pittsburgh No. 8 coal.

200 MESH FLOTATION		28 MESH FLOTATION		200 MESH WASHABILITY		28 MESH WASHABILITY	
CMR	NPAR	CMR	NPAR	CMR	NPAR	CMR	NPAR
$\bar{x}$	$\bar{x}$	$\bar{x}$	$\bar{x}$	$\bar{x}$	$\bar{x}$	$\bar{x}$	$\bar{x}$
87.9	53.9	88.4	32.6	60.4	88.3	56.9	87.4
80.9	60.5	86.1	53.7	67.7	85.4	76.9	78.3
80.5	61.9	84.0	57.4	77.1	81.2	85.7	72.7
75.7	64.2	72.7	63.9	95.1	63.0	94.6	61.8
70.9	67.0	71.5	66.5	96.4	59.4	96.7	55.0
50.2	77.1	55.3	75.3				

Figure 8.4 Effect of grinding mode on separation of pyrite for Pittsburgh No. 8 coal and a comparison of the washability and flotation selectivity curves for the 28 mesh grind.

DRY GRIND		WET GRIND		WASHABILITY	
CMR	PSR	CMR	PSR	CMR	PSR
$\bar{x}$	$\bar{x}$	$\bar{x}$	$\bar{x}$	$\bar{x}$	$\bar{x}$
88.4	44.4	91.7	41.7	56.9	93.8
86.1	48.6	91.5	40.8	76.9	84.9
84.0	50.6	89.4	44.8	85.7	79.0
72.7	61.4	86.4	49.8	94.6	67.3
71.5	61.1	75.0	60.0	96.7	61.3
55.3	72.9	59.5	71.3		

Figure 8.5 Effect of grinding mode on the separation of pyrite from Pittsburgh No. 8 coal and a comparison of washability and flotation selectivity curves for the 200 mesh grind.

DRY GRIND		WET GRIND		WASHABILITY	
CMR	PSR	CMR	PSR	CMR	PSR
$\bar{x}$	$\bar{x}$	$\bar{x}$	$\bar{x}$	$\bar{x}$	$\bar{x}$
87.9	57.2	90.6	63.8	60.4	98.2
80.9	62.2	85.6	69.1	67.7	97.3
80.5	63.8	78.7	73.6	77.1	96.2
75.7	68.1	77.7	74.5	95.1	90.1
70.9	70.6	74.9	76.2	96.4	87.7
50.2	80.8	53.7	84.8		

Figure 8.6 Particle size distribution for Illinois No. 6 research sample grind under standard dry and wet conditions and in a ceramic and steel ball mill.

Size, microns	CUMULATIVE WEIGHT PERCENT PASSING			
	Ceramic	Steel	Std. dry	Std. wet
75	92.1	92.3	96.8	93.2
53	81.1	79.7	89.2	78.7
45	74.2	73.7	84.3	70.0
38	67.0	62.9	76.1	58.1
26	57.5	52.6	67.6	45.2
19	47.4	41.7	57.3	34.4
13	38.0	32.4	47.9	25.5
9	29.9	24.9	39.5	19.2
6.8	22.8	18.2	30.8	13.8
4.4	17.4	13.5	23.7	9.8
3.4	11.1	8.6	15.6	6.3

Figure 8.7 Effect of adding collector to the grinding mill on the yield, ash and total sulfur content of the concentrate for minus 28-mesh wet-ground Illinois No. 6 coal.

<u>Dosage, lb/T</u>	<u>FLOTATION PRODUCT ANALYSIS</u>		
	<u>Yield, %</u>	<u>Ash, %</u>	<u>Tot. S., %</u>
0.48	45.4	9.4	3.81
0.96	72.5	10.3	4.28
1.92	82.0	11.2	4.19
4.8	90.2	11.2	4.51

Figure 8.8 Effect of adding part of the collector to the grinding mill and the balance to the flotation cell, on the yield, ash and total sulfur content of the concentrate of minus 28-mesh wet-ground Illinois No. 6 coal.

<u>Dosage, lb/T</u>	<u>FLOTATION PRODUCT ANALYSIS</u>		
	<u>Yield, %</u>	<u>Ash, %</u>	<u>Tot. S., %</u>
0.48	82.9	10.6	4.21
0.96	82.4	11.4	4.58
1.92	87.9	10.9	4.39
4.82	90.7	11.2	4.51

Figure 8.9 Effect of adding part of the collector to the grinding mill on the yield, ash and total sulfur content of the concentrate of minus 200-mesh wet-ground Illinois No. 6 coal.

<u>Collector Dosage, lb/T</u>	<u>FLOTATION PRODUCT ANALYSIS</u>		
	<u>Conc. Yield, %</u>	<u>Ash, %</u>	<u>Tot. S, %</u>
1.93	66.1	7.8	3.62
4.36	83.3	9.6	4.4
4.82	83.6	9.3	4.18
5.78	87.2	9.3	4.25

Figure 8.10 Effect of adding part of the collector to the grinding mill and the balance to the flotation cell on the yield, ash and total sulfur content of the concentrate of minus 200-mesh wet-ground Illinois No. 6 coal.

<u>Collector Dosage, lb/T</u>	<u>FLOTATION PRODUCT ANALYSIS</u>		
	<u>Conc. Yield, %</u>	<u>Ash, %</u>	<u>Tot. S, %</u>
1.93	80.8	8.3	4.00
4.36	81.5	9.2	4.32
4.82	85.8	9.3	4.33
5.78	87.2	9.3	4.25

Figure 8.11 Effect of adding frother (MIBC) to the grinding mill and in the flotation cell on the yield of the concentrate obtained using Illinois No. 6 coal.

<u>Frother Dosage lb/T</u>	<u>FLOTATION CONC. YIELD, %</u>			
	<u>28 M Wet Grind</u>		<u>200 M wet grind</u>	
	<u>Cell</u>	<u>Mill</u>	<u>Cell</u>	<u>Mill</u>
0.35	89.9	39.7	36.1	0.0
1.04	91.0	82.8	78.3	45.8
1.75	92.4	--	83.2	75.8

Figure 8.12 Flotation yield of 200-mesh Illinois No. 6 coal obtained by adding 5.8 lb/T of dodecane to the grinding mill or flotation cell.

Time, Minutes	YIELD, %	
	Cell	Mill
0.5	28.9	48.5
1.0	44.9	70.2
2.0	66.5	81.2
4.0	79.3	85.5
5.0	81.6	86.4

Figure 8.13 Flotation yield of 200-mesh Pittsburgh No. 8 coal obtained by adding 1.9 lb/T of dodecane to the grinding mill or flotation cell.

Time, Minutes	YIELD, %	
	Cell	Mill
0.0	0.0	0.0
0.5	21.6	17.6
1.0	39.7	30.7
2.0	64.4	44.1
4.0	75.6	55.2
5.0	78.8	57.8

Figure 8.14 Flotation yield of 200-mesh Upper Freeport PA coal obtained by adding 0.5 lb/T of dodecane to the grinding mill or flotation cell.

Flot. Time Minutes	YIELD, %	
	Cell	Mill
0.0	0.0	0.0
0.5	29.0	22.7
1.0	49.4	39.9
2.0	67.8	59.3
4.0	77.9	72.6
5.0	81.4	76.7

Figure 8.15 Flotation yield of 200-mesh Illinois No. 6 coal obtained by adding 0.7 lb/T of dodecane to the grinding mill or flotation cell.

Flot. Time Minutes	YIELD, %	
	Cell	Mill
0.0	0.0	0.0
0.5	14.25	9.63
1.0	25.02	18.81
2.0	37.44	29.24
4.0	50.32	40.83
8.0	61.37	51.17

Figure 8.16 Flotation yield of 200-mesh Illinois No. 6 coal obtained by adding 2.9 lb/T of dodecane to the grinding mill or flotation cell.

Flot. Time Minutes	YIELD, %	
	Cell	Mill
0.0	0.0	0.0
0.5	25.34	44.67
1.0	43.49	66.57
2.0	58.68	73.45
4.0	68.91	78.72
8.0	76.55	83.24

Figure 8.17 Flotation yield of 200-mesh Pittsburgh No. 8 coal obtained by adding 0.7 lb/T of dodecane to the grinding mill or flotation cell.

Flot. Time Minutes	YIELD, %	
	Cell	Mill
0.0	0.0	0.0
0.5	8.11	6.73
1.0	19.42	14.74
2.0	37.28	24.29
4.0	55.69	32.22
8.0	67.29	39.13

Figure 8.18 Flotation yield of 200-mesh Pittsburgh No. 8 coal obtained by adding 2.9 lb/T of dodecane to the grinding mill or flotation cell.

Flot. Time Minutes	YIELD, %	
	Cell	Mill
0.0	0.0	0.0
0.5	24.58	26.3
1.0	44.4	44.97
2.0	66.98	57.79
4.0	76.51	63.96
8.0	81.19	67.29

Figure 8.19 Flotation yield of 200-mesh Upper Freeport PA coal obtained by adding 0.24 lb/T of dodecane to the grinding mill or flotation cell.

Flot. Time Minutes	YIELD, %	
	Cell	Mill
0.0	0.0	0.0
0.5	18.17	13.35
1.0	34.72	27.08
2.0	55.35	43.13
4.0	68.68	57.98
8.0	75.81	68.56

Figure 8.20 Flotation yield of 200-mesh Upper Freeport PA coal obtained by adding 2.9 lb/T of dodecane to the grinding mill or flotation cell.

Flot. Time Minutes	YIELD, %	
	Cell	Mill
0.0	0.0	0.0
0.5	47.35	30.55
1.0	69.33	55.29
2.0	78.50	72.9
4.0	82.77	80.82
8.0	86.33	85.66

Figure 8.21 The effect of aging after grinding on the flotation yield of 200-mesh Illinois No. 6 coal obtained by adding 2.9 lb/T of dodecane to the grinding mill or flotation cell.

Flot. Time Minutes	YIELD, %, NO AGING		YIELD, %, AGING	
	Cell	Mill	Cell	Mill
0.0	0.0	0.0	0.0	0.0
0.5	25.34	44.67	26.15	24.95
1.0	43.49	66.57	40.4	38.63
2.0	58.68	73.45	52.76	49.35
4.0	68.91	78.72	62.67	59.52
8.0	76.55	83.24	71.96	68.58

Figure 8.22 The effect of aging after grinding on the flotation yield of 200-mesh Pittsburgh No. 8 coal obtained by adding 2.9 lb/T of dodecane to flotation cell.

Flot. Time Minutes	YIELD, %	
	No Aging	Aging
0.0	0.0	0.0
0.5	24.58	24.09
1.0	44.4	42.44
2.0	66.98	62.46
4.0	76.51	73.1
6.0	81.19	80.71

Figure 9.1 Effect of Aerosol OT addition on rejection of pyrite from wet-ground minus 200 mesh Pittsburgh No. 8 coal.

DOD. + MIBC IN CELL		AOT. IN MILL		AOT. IN CELL		AOT. + MIBC IN CELL	
CMR	PSR	CMR	PSR	CMR	PSR	CMR	PSR
<u>%</u>	<u>%</u>	<u>%</u>	<u>%</u>	<u>%</u>	<u>%</u>	<u>%</u>	<u>%</u>
90.6	63.8	5.3	97.8	28.7	91.9	75.4	72.4
85.6	69.1	51.5	84.2	52.0	84.3	93.7	54.2
78.7	73.6	72.5	72.2	84.6	64.4	95.6	50.8
77.7	74.5	88.2	61.7			89.8	64.2
74.9	76.2	96.0	49.1				
53.7	84.8	99.8	28.3				
		99.1	43.3				

Figure 9.2 Effect of Aerosol OT addition on rejection of non-pyritic minerals from wet-ground minus 200 mesh Pittsburgh No. 8 coal.

DOD. + MIBC IN CELL		AOT. IN MILL		AOT. IN CELL		AOT. + MIBC IN CELL	
CMR	NPAR	CMR	NPAR	CMR	NPAR	CMR	NPAR
<u>%</u>	<u>%</u>	<u>%</u>	<u>%</u>	<u>%</u>	<u>%</u>	<u>%</u>	<u>%</u>
90.6	58.4	5.3	97.1	28.7	88.9	75.4	70.1
85.6	64.0	51.5	80.6	52.0	79.1	93.7	54.0
78.7	69.5	72.5	70.9	84.6	60.9	95.6	57.2
77.7	68.9	88.2	59.7	99.8	45.5	89.8	61.6
74.9	71.3	96.0	53.4	99.1	46.9		
		53.7	80.9				

Figure 9.3 Effect of Aerosol OT addition on rejection of pyrite from wet-ground minus 200 mesh Illinois No. 6 coal.

DOD. + MIBC IN CELL		AOT. IN MILL + MIBC IN CELL	
CMR %	PSR %	CMR %	PSR %
87.1	64.7	93.6	48.1
74.3	69.3	93.6	48.0
84.8	64.9	85.7	58.1
82.1	66.9	88.2	54.3

Figure 9.4 Effect of Aerosol OT addition on rejection of non-pyritic minerals from wet ground minus 200 mesh Illinois No. 6 coal.

DOD. + MIBC IN CELL		AOT. IN MILL + MIBC IN CELL	
CMR %	NPAR %	CMR %	NPAR %
87.1	59.7	93.6	41.0
74.3	60.6	93.6	38.7
84.8	59.6	85.7	46.7
		88.2	54.3

Figure 9.5 Effect of Aerosol OT addition on rejection of pyrite from wet-ground minus 200 mesh Upper Freeport PA coal.

DOD. + MIBC IN CELL		AOT. IN MILL	
CMR %	PSR %	CMR %	PSR %
31.5	90.7	83.8	71.9
66.2	80.0	87.6	69.0
89.7	64.2	88.4	66.0
97.3	56.8	89.0	65.2
		74.0	80.1
		82.4	73.9
		84.1	70.8
		85.4	71.8

Figure 9.6 Effect of Aerosol OT addition on rejection of non-pyritic minerals from wet ground minus 200 mesh Upper Freeport PA coal.

DOD. + MIBC IN CELL		AOT. IN MILL	
CMR %	NPAR %	CMR %	NPAR %
31.5	88.5	83.8	57.9
66.2	75.3	87.6	53.0
89.7	56.0	88.4	50.7
97.3	45.0	89.0	48.8
		74.0	67.3
		82.4	61.3
		84.1	57.9
		85.4	56.2

Figure 9.7 Effect of 4-tert. butylpyridine addition on rejection of pyrite from wet ground minus 200 Mesh Pittsburgh No. 8 coal.

DOD. + MIBC IN CELL		4-TERT. IN MILL AND DOD. + MIBC IN CELL		4-TERT. IN MILL AND MIBC IN CELL	
CMR	PSR	CMR	PSR	CMR	PSR
90.6	63.8	97.5	44.6	94.9	40.1
85.6	69.1	96.5	44.2	85.0	51.7
78.7	73.6	96.0	51.1	61.4	69.5
77.7	74.5	92.1	52.9		
74.9	76.2	89.4	56.2		
53.7	84.8	89.2	54.4		
		87.9	59.8		
		69.6	68.8		

Figure 9.8 Effect of 4-tert. butylpyridine addition on rejection of non-pyritic minerals from wet ground minus 200 Mesh Pittsburgh No. 8 coal.

DOD. + MIBC IN CELL		4-TERT. IN MILL AND DOD. + MIBC IN CELL		4-TERT. IN MILL AND MIBC IN CELL	
CMR %	NPAR %	CMR %	NPAR %	CMR %	NPAR %
90.6	58.4	97.5	54.2	94.9	55.6
85.6	64.0	96.5	56.1	85.0	64.7
78.7	69.5	96.0	53.7	61.4	76.2
77.7	68.9	92.1	58.7		
74.9	71.3	89.4	59.1		
53.7	80.9	89.2	60.0		
		87.9	63.5		
		69.6	71.6		

Figure 9.9 Effect of 4-(1-butylpentyl)pyridine addition on rejection of pyrite from wet ground minus 200 mesh Pittsburgh No. 8 coal.

DOD. + MIBC IN CELL		2.1 lb/T IN MILL AND DOD. + MIBC IN CELL		4.2 lb/T IN MILL AND DOD. + MIBC IN CELL	
CMR %	PSR %	CMR	PSR %	CMR %	PSR %
90.6	63.8	87.0	57.7	99.6	28.7
85.6	69.1	88.9	55.0	97.9	30.0
78.7	73.6	82.5	63.3	98.0	38.9
77.7	74.5	47.7	82.2		
74.9	76.2				
53.7	84.8				

Figure 9.10 Effect of 4-(1-butylpentyl)pyridine addition on rejection of non-pyritic minerals from wet ground minus 200 Mesh Pittsburgh No. 8 coal.

DOD. + MIBC IN CELL		2.1 lb/T IN MILL AND DOD. + MIBC IN CELL		4.2 LB/T IN MILL AND DOD. + MIBC IN CELL	
CMR %	NP AR%	CMR %	NP AR%	CMR %	NP AR%
90.6	58.4	87.0	62.0	99.6	38.7
85.6	64.0	88.9	61.9	97.9	41.9
78.7	69.5	82.5	62.6	98.0	43.9
77.7	68.9	47.7	80.2		
74.9	71.3				
53.7	80.9				

Figure 9.11 Comparison of the collecting ability of the GH series of reagent with that of dodecane in the flotation of 200-mesh wet-ground Illinois No. 6 coal.

POUNDS PER TON			MOLES PER TON			Yield, %
<u>Coll.</u>	<u>Froth.</u>	<u>Mod.</u>	<u>Coll.</u>	<u>Froth.</u>	<u>Mod.</u>	
standard						
5.75	1.16	0.0	15.44	5.16	0.0	78.8
8.63	1.16	0.0	23.14	5.16	0.0	80.6
11.5	1.16	0.0	30.83	5.16	0.0	84.3
14.38	1.16	0.0	38.55	5.16	0.0	83.0
GHO						
0.0	1.16	0.52	0.0	5.16	1.07	75.3
0.0	1.16	0.78	0.0	5.16	1.61	82.8
0.0	1.16	1.17	0.0	5.16	2.41	91.8
GH1.5						
0.0	1.16	0.58	0.0	5.16	0.92	62.9
0.0	1.16	0.86	0.0	5.16	1.36	77.6
0.0	1.16	1.3	0.0	5.16	2.06	87.5
GH4						
0.0	1.16	0.58	0.0	5.16	0.66	66.2
0.0	1.16	0.88	0.0	5.16	1.01	76.8
0.0	1.16	1.17	0.0	5.16	1.34	82.3
0.0	1.16	1.31	0.0	5.16	1.5	86.5
GH6						
0.0	1.16	0.59	0.0	5.16	0.55	26.1
0.0	1.16	0.88	0.0	5.16	0.82	37.0
0.0	1.16	1.18	0.0	5.16	1.11	47.3
0.0	1.16	1.32	0.0	5.16	1.24	66.6

Figure 9.12 Comparison of the collecting ability of GH series with that of dodecane in the flotation of 200-mesh wet-ground Pittsburgh No. 8 coal.

POUNDS PER TON			MOLES PER TON			Yield, %
Coll.	Froth.	Mod.	Coll.	Froth.	Mod.	
standard						
1.92	0.3	0.0	5.15	1.33	0.0	78.8
2.88	0.3	0.0	7.72	1.33	0.0	80.6
3.84	0.3	0.0	10.29	1.33	0.0	84.3
5.76	0.3	0.0	15.44	1.33	0.0	83.0
GHO						
0.0	0.3	0.78	0.0	1.33	1.61	75.3
0.0	0.3	0.91	0.0	1.33	1.88	82.8
0.0	0.3	1.04	0.0	1.33	2.14	91.8
GH4						
0.0	0.3	0.58	0.0	1.33	0.66	66.2
0.0	0.3	0.88	0.0	1.33	1.01	76.8
0.0	0.3	1.17	0.0	1.33	1.34	82.3
GH6						
0.0	0.3	0.88	0.0	1.33	0.82	26.1
0.0	0.3	1.03	0.0	1.33	0.96	37.0
0.0	0.3	1.18	0.0	1.33	1.11	47.3

Figure 9.13 Effect of 2,n-butylthiophene addition on rejection of pyrite from wet-ground minus 200 mesh Pittsburgh No. 8 coal.

DOD. + MIBC IN CELL		2,N-BUTYL. IN MILL AND DOD. + MIBC IN CELL		2,N-BUTYL. + DOD. + MIBC IN CELL	
CMR %	PSR %	CMR %	PSR %	CMR %	PSR %
44.3	88.8	77.9	76.7	63.2	84.3
78.0	75.6	80.0	72.7	78.0	76.3
89.0	69.3	79.6	72.6	80.0	75.7
89.9	68.0			65.7	81.4
69.1	79.7			68.8	81.2
				74.2	77.9
				78.4	75.3

Figure 9.14 Effect of 2,n-butylthiophene addition on rejection of non-pyritic minerals from wet ground minus 200 mesh Pittsburgh No. 8 coal.

DOD. + MIBC IN CELL		2,N-BUTYL. IN MILL AND DOD. + MIBC IN CELL		2,N-BUTYL. + DOD. + MIBC IN CELL	
CMR %	NPAR %	CMR %	NPAR %	CMR %	NPAR %
44.3	84.2	77.9	74.5	63.2	82.8
78.0	67.4	80.0	72.6	78.0	75.3
89.0	56.2	79.6	72.8	80.0	73.9
89.9	57.1			65.7	80.1
69.1	74.3			68.8	80.2
				74.2	75.9
				78.4	73.6

Figure 9.15 Effect of dodecane addition on rejection of pyrite from wet-ground minus 200 Mesh Pittsburgh No. 8 coal - single stage flotation results.

NO DODECANE		0.2 LB/T DODECANE		2.0 LB/T DODECANE		20.0 LB/T DODECANE	
CMR %	PSR %	CMR %	PSR %	CMR %	PSR %	CMR %	PSR %
54.1	86.5	62.3	84.1	85.3	67.9	92.7	56.3
92.0	65.5	89.0	67.4	95.4	57.9	87.9	59.5
82.4	73.2	92.9	65.2	90.6	63.8	96.7	50.3
72.0	79.1	95.2	81.0	85.6	69.1	74.5	72.3
88.9	68.3	47.1	87.4	78.7	73.6	64.4	76.9
26.7	93.3	78.9	75.7	77.7	74.5		
43.5	89.2			74.9	75.2		
89.5	68.9			53.7	84.8		
76.8	78.2						

Figure 9.16 Effect of dodecane addition on rejection of pyrite from wet-ground minus 200 Mesh Pittsburgh No. 8 coal - two-stage flotation results.

NO DODECANE		0.2 LB/T DODECANE		2.0 LB/T DODECANE		20.0 LB/T DODECANE	
CMR %	PSR %	CMR %	PSR %	CMR %	PSR %	CMR %	PSR %
85.9	75.8	73.5	81.4	92.2	64.2	77.5	72.7
71.4	83.1	90.6	70.8	94.7	65.2	85.5	86.0
91.6	72.0	88.3	75.2	74.7	79.0	62.4	80.6
51.1	89.8	55.8	87.7	45.6	88.0	95.7	52.0
79.7	80.0			80.3	75.2		
46.7	90.4						

Figure 9.17 Effect of dodecane addition on rejection of non-pyritic minerals from wet-ground minus 200 Mesh Pittsburgh No. 8 coal - single stage flotation results.

NO DODECANE		0.2 LB/T DODECANE		2.0 LB/T DODECANE		20.0 LB/T DODECANE	
CMR %	NPAR %	CMR %	NPAR %	CMR %	NPAR %	CMR %	NPAR %
54.1	80.1	62.3	76.6	85.3	67.4	92.7	60.0
92.0	58.6	89.0	62.8	95.4	56.7	87.9	64.5
82.4	66.8	92.9	59.5	90.6	58.4	96.7	56.7
72.0	71.6	95.2	57.7	85.6	64.0	74.5	72.9
88.9	62.1	47.1	83.6	78.7	69.5	64.4	76.6
26.7	90.8	78.9	68.6	77.7	68.9		
43.5	84.4			74.9	71.7		
89.5	60.9			53.7	80.9		
76.8	68.7						

Figure 9.18 Effect of dodecane addition on rejection of non-pyritic minerals from wet-ground minus 200 mesh Pittsburgh No. 8 coal - two-stage flotation results.

NO DODECANE		0.2 LB/T DODECANE		2.0 LB/T DODECANE		20.0 LB/T DODECANE	
CMR %	NPAR %	CMR %	NPAR %	CMR %	NPAR %	CMR %	NPAR %
85.9	74.0	73.5	80.0	92.2	71.0	77.5	76.3
71.4	80.1	90.6	72.2	94.7	66.6	85.5	73.5
91.6	69.3	88.3	71.5	74.7	77.4	62.4	83.2
51.1	87.7	55.8	85.3	45.6	88.3	95.7	66.2
79.7	75.0			80.3	74.9		
46.7	88.4						

Figure 9.19 Comparison of performances of butanol, hexanol and MIBC in terms of reagent consumption.

MIBC		BUTANOL		HEXANOL	
CMR %	Dosage lb/T	CMR %	Dosage lb/T	CMR %	Dosage lb/T
46.9	1.2	89.1	17.6	90.0	2.7
72.5	1.4	83.7	18.9	59.8	1.8
74.2	1.8	75.1	15.6	81.5	2.3
74.2	1.6	75.5	15.6	80.0	2.2
86.9	1.8	74.3	15.9	92.8	2.9
90.9	2.0	68.2	15.2	83.5	2.5
		54.5	14.3		

Figure 9.20 Effect of frother type on pyrite rejection from wet-ground minus 200 mesh Pittsburgh No. 8 coal - single stage flotation.

MIBC		BUTANOL		HEXANOL	
CMR %	PSR %	CMR %	PSR %	CMR %	PSR %
91.1	74.0	63.6	85.9	63.5	86.2
87.5	76.3	72.1	82.5	84.2	79.3
75.5	81.9	78.6	80.4	94.4	71.2
25.5	96.2	93.8	69.0	43.1	92.5
54.2	89.3	88.8	75.3	56.1	89.3
		92.6	71.6		
		92.8	70.9		
		91.4	73.9		
		90.1	75.2		

Figure 9.21 Effect of frother type on pyrite rejection from wet-ground minus 200 mesh Pittsburgh No. 8 coal - two-stage flotation.

MIBC		BUTANOL		HEXANOL	
CMR %	PSR %	CMR %	PSR %	CMR %	PSR %
46.9	93.6	89.2	0.7	80.8	90.0
72.5	87.2	83.7	83.2	59.8	91.5
74.2	86.6	75.1	86.5	81.5	83.1
74.2	86.1	75.5	86.2	80.0	85.1
86.9	82.0	74.3	86.6	92.8	78.8
90.9	79.9	68.2	88.2	83.5	83.0
		54.5	91.7		

Figure 9.22 Effect of frother type on non-pyritic minerals rejection from wet-ground minus 200 mesh Pittsburgh No. 8 coal - single stage flotation.

MIBC		BUTANOL		HEXANOL	
CMR %	NPAR %	CMR %	NPAR %	CMR %	NPAR %
91.1	58.9	63.6	77.9	63.5	78.5
87.5	62.9	72.1	74.0	84.2	67.1
75.5	71.1	78.6	70.2	94.4	58.1
25.5	92.0	93.8	56.4	43.1	86.0
54.2	80.7	88.8	62.6	56.1	80.8
		92.6	58.1		
		92.8	57.2		
		91.4	58.8		
		90.1	61.0		

Figure 9.23 Effect of frother type on non-pyritic minerals rejection from wet-ground minus 200 mesh Pittsburgh No. 8 coal - two-stage flotation.

MIBC		BUTANOL		HEXANOL	
CMR %	NPAR %	CMR %	NPAR %	CMR %	NPAR %
46.9	88.5	89.1	68.9	90.0	69.5
72.5	78.9	83.7	72.9	59.8	84.2
74.2	77.7	75.1	78.2	81.5	75.5
74.2	78.6	75.5	79.2	80.0	76.2
86.9	72.1	74.3	79.2	92.8	69.0
90.9	69.4	68.2	82.1	83.5	73.9
		54.5	86.4		

Figure 9.25 Comparison of effects of sodium hydroxide addition in the mill and in the cell on coal flotation recovery.

NaOH ADDED IN MILL		NaOH ADDED IN CELL	
pH	CMR %	pH	CMR %
4	77.7	4	78.7
8	79.9	6	69.1
4.8	79.5	8	44.7
5.2	80.3	10	23.7
7.5	79.1		
9.2	66.2		
10.0	56.5		

Figure 9.26 Effect of mode of sodium hydroxide addition (mill or cell) on pH - time relationship.

NaOH ADDED IN CELL		NaOH ADDED IN MILL	
Time, min	pH	Time, min	pH
0.0	6.4	0.0	12.6
2.0	5.1	2.0	10.3
5.0	4.2	5.0	8.7
10.0	4.1	10.0	8.3
17.0	4.0	17.0	8.1
81.0	3.9	81.0	8.1
81.5	6.0	81.5	8.0
82.0	8.0	82.0	8.1
92.0	8.0	92.0	8.0
97.0	7.9	97.0	8.0

Figure 9.27 Effect of pH (adjusted by adding sodium hydroxide in mill) on dissolved species concentration in the supernatant of 28 mesh wet-ground Pittsburgh No. 8 coal.

pH	DISSOLVED SPECIES CONCENTRATION			
	Fe	Al	Mg	Ca
3.9	5.0	0.41	0.8	2.5
4.1	4.8	0.41	0.8	2.4
4.8	4.3	0.07	0.8	2.3
5.2	2.7	0.0	0.8	2.3
6.1	0.8	0.0	0.7	2.2
6.7	0.3	0.0	0.7	2.1
7.5	0.0	0.0	0.7	1.9
9.2	0.0	0.0	0.4	1.5
10.0	0.0	0.0	0.1	1.4

Figure 9.28 Effect of pH (adjusted by adding sodium hydroxide in cell) on dissolved species concentration in the supernatant of 200 mesh wet-ground Pittsburgh No. 8 coal.

pH	DISSOLVED SPECIES CONCENTRATION			
	Fe	Al	Mn	Ca
3.9	4.98	0.4	0.75	2.4
4.0	5.0	0.4	0.79	2.5
4.8	4.77	0.0	0.75	2.4
5.0	3.76	0.0	0.79	2.4
5.9	0.84	0.0	0.71	2.3
6.0	0.9	0.0	0.71	2.3
6.7	0.25	0.0	0.67	2.1
7.0	0.16	0.0	0.67	2.0
8.0	0.02	0.0	0.63	1.8
9.0	0.0	0.0	0.6	1.5
10.0	0.0	0.0	0.0	1.33

Figure 9.29 Effect of ferrous chloride addition on washed coal flotation recovery.

Conc., ppm	CMR %
0	84.4
0	80.3
140	71.1
280	41.9
420	33.2
560	29.0
280	43.6

Figure 9.30 Zeta potential behaviors of coal samples prepared by grinding the coal in mill and in cell, and metal-hydroxides precipitated from coal supernatant.

NaOH ADDED IN MILL		NaOH ADDED		PRECIPITATES FROM COAL SLURRY	
pH	Zeta pot. mV	pH	Zeta pot. mV	pH	Zeta pot. mV
3.08	11.54	3.08	11.54	5.10	5.82
3.86	5.01	3.86	5.19	5.10	3.88
4.52	-2.62	4.7	-0.37	5.80	4.15
4.52	-1.72	5.1	-2.59	6.10	4.9
5.92	-11.01	5.9	-2.19	8.10	3.15
7.38	-19.89	5.9	-0.17	10.70	1.1
7.44	-18.34	6.96	-5.33	9.2	3.45
9.24	-34.18	8.10	-11.82		
11.20	-52.90	9.30	-20.38		
		9.31	-18.41		
		11.02	-31.13		

Figure 9.31 Effect of sodium hydroxide addition in cell on pyrite rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal.

pH 4		pH 6		pH 8		pH 10	
CMR %	PSR %	CMR %	PSR %	CMR %	PSR %	CMR %	PSR %
90.6	63.8	44.3	88.8	66.4	82.3	23.7	95.0
85.6	69.1	78.0	75.6	44.7	89.3	74.8	80.5
78.7	73.6	89.0	69.3	83.5	74.5	75.0	78.7
77.7	74.5	89.9	68.0	87.6	73.6	85.3	73.8
74.9	76.2	69.1	79.7	90.9	69.5	51.5	87.3
53.7	84.8					91.2	69.0

Figure 9.32 Effect of sodium hydroxide addition in mill on pyrite rejection from wet-ground minus 200 mesh Pittsburgh No. 8 coal.

pH 4.0		pH 4.8		pH 5.2		pH 7.5	
CMR %	PSR %	CMR %	PSR %	CMR %	PSR %	CMR %	PSR %
90.6	63.8	55.7	84.5	63.8	81.5	61.2	84.8
85.6	69.1	79.5	73.6	80.3	73.9	79.1	77.8
78.7	73.6	81.1	86.2	84.3	83.5	80.9	70.3
77.7	74.5	83.5	83.5	84.5	83.1	92.4	70.7
74.9	76.2						
53.7	84.8						

pH 8.0		pH 8.2	
CMR %	PSR %	CMR %	PSR %
94.0	67.8	30.4	93.9
89.8	74.4	46.9	89.8
79.9	78.1	91.7	72.5
68.4	83.5	66.2	84.2
43.5	90.7	84.3	76.9
		88.3	74.1

Figure 9.33 Comparison of effects of sodium hydroxide additions in mill and cell on pyrite rejection from wet-ground minus 200 Mesh Pittsburgh No. 8 coal.

NaOH ADDED IN CELL		NaOH ADDED IN MILL	
CMR %	PSR %	CMR %	PSR %
66.4	82.3	94.0	67.8
44.7	89.3	89.8	74.4
83.5	74.5	79.9	78.1
87.6	73.6	68.4	83.5
90.9	69.5	43.5	90.7

Figure 9.34 Zeta potential behaviors of coal and pyrite samples prepared by grinding the feeds in mill, and metal-hydroxides precipitated from coal supernatant.

NON-PRECIPITATION COAL		NON-PRECIPITATION PYRITE		PRECIPITATES FROM COAL SUPERNATANT	
pH	Zeta pot., mV	pH	Zeta pot., mV	pH	Zeta pot., mV
3.08	11.54	2.93	4.44	5.10	5.82
3.86	5.01	3.01	1.28	5.10	3.88
4.52	-2.62	4.20	-2.95	5.80	4.15
4.52	-1.72	5.40	-6.46	6.10	4.90
5.92	-11.01	6.20	-9.13	8.10	3.15
7.38	-19.89	8.20	-9.95	10.70	1.10
7.44	-18.34	10.30	-11.08	9.20	3.45
9.24	-34.18	11.20	-12.82		
11.20	-52.90				

Figure 9.36 Effect of sodium hydroxide addition in cell on non-pyritic minerals rejection from wet-ground minus 200 mesh Pittsburgh No. 8 coal.

pH 4		pH 6		pH 8		pH 10	
CMR %	NPAR %	CMR %	NPAR %	CMR %	NPAR %	CMR %	NPAR %
90.6	58.4	44.3	84.2	66.4	74.8	23.7	91.7
85.6	64.0	78.0	67.4	44.7	83.8	74.8	69.6
78.7	69.5	89.0	56.2	83.5	63.1	75.0	68.6
77.7	68.8	89.9	57.1	87.6	55.4	85.3	60.4
74.9	71.7	69.1	74.3	90.9	54.9	51.5	81.0
53.7	80.9					91.2	52.9

Figure 9.37 Effect of Sodium Hydroxide Addition in Mill on Non-pyritic Minerals
Rejection from Wet Ground Minus 200 Mesh Pittsburgh No. 8 Coal.

pH 4.0		pH 4.8		pH 5.2		pH 7.5	
CMR %	NPAR %	CMR %	NPAR %	CMR %	NPAR %	CMR %	NPAR %
90.6	58.4	55.7	82.5	63.8	78.2	61.2	79.4
85.6	64.0	79.5	69.1	80.3	67.7	79.1	69.3
78.7	69.5	91.1	60.1	94.3	54.1	90.9	58.9
77.7	68.9	93.5	55.6	94.5	53.1	92.4	57.5
74.9	71.3						
53.7	80.9						

pH 8.0		pH 9.2	
CMR %	NPAR %	CMR %	NPAR %
94.0	52.6	30.4	90.8
89.8	61.5	46.9	85.1
79.9	68.5	91.7	58.2
68.4	77.5	66.2	78.1
43.5	86.6	84.3	66.2
		88.3	62.9

Figure 9.38 Effect of frother dosage on coal flotation recovery under precipitation
(NaOH added in cell) and non-precipitation (NaOH added in mill) conditions.

NaOH ADDED IN MILL		NaOH ADDED IN CELL	
MIBC, lb/T	CMR %	MIBC, lb/T	CMR
0.88	95.3	0.44	66.4
0.59	94.0	0.29	44.7
0.44	89.8	0.59	83.5
0.29	79.9	0.88	87.6
0.22	68.3	0.88	90.9
0.15	43.5		

Figure 9.39 Effect of frother dosage on total water flow out of the cell.

MIBC, lb/T	Water Flow
1.56	1108.5
0.39	318.5
0.935	912.4

Figure 9.40 Comparison of effects of sodium and calcium hydroxides in mill on
pyrite rejection from wet-ground minus 200 Mesh Pittsburgh No. 8 coal.

NaOH		Ca(OH) ₂	
CMR %	PSR %	CMR %	PSR %
95.3	66.0	76.0	81.8
94.0	67.8	92.7	71.6
89.8	74.4	53.2	89.3
79.9	78.1	65.0	85.2
68.3	83.5	87.5	75.6
43.5	90.7		

Figure 9.41 Comparison of effects of sodium and calcium hydroxides in mill on non-pyritic minerals rejection from wet-ground minus 200 mesh Pittsburgh No. 8 coal.

NaOH		Ca(OH) ₂	
CMR %	NPAR %	CMR %	NPAR %
95.3	51.4	76.0	70.9
94.0	52.6	92.7	58.0
89.8	61.5	53.2	81.7
79.9	68.8	65.0	77.6
68.3	76.8	87.5	62.2
43.5	86.1		

Figure 9.42 Effect of frother dosage on coal flotation recovery: comparison of frother consumptions in the presence of sodium hydroxide and calcium hydroxide.

NaOH		Ca(OH) ₂	
MIBC, lb/T	CMR %	MIBC, lb/T	CMR %
0.88	95.3	0.29	76.0
0.59	94.0	0.59	92.7
0.44	89.8	0.15	53.2
0.29	79.9	0.22	65.0
0.22	68.3	0.44	87.5
0.15	43.5		

Figure 9.43 Effect of calcium hydroxide addition in cell on pyrite rejection from dry-ground minus 200 Mesh Pittsburgh No. 8 coal.

pH 4.3		pH 8		pH 8		pH 10	
CMR %	PSR %	CMR %	PSR %	CMR %	PSR %	CMR %	PSR %
87.9	57.2	69.6	72.0	66.4	78.5	43.2	82.3
80.9	62.2						
80.5	63.8						
75.7	68.1						
70.9	70.6						
50.2	80.8						

Figure 9.44 Effect of calcium hydroxide addition in cell on combustible material recovery and efficiency index from wet-ground minus 28 mesh Pittsburgh No. 8 coal.

pH	CMR %	pH	EI _H
3.7	92.4	3.7	25.4
6.0	89.9	6.0	27.1
8.0	94.0	8.0	30.4
10.0	88.1	10.0	30.1

Figure 9.45 Effect of calcium hydroxide addition in cell on combustible material recovery and efficiency index from dry-ground minus 28 mesh Pittsburgh No. 8 coal.

pH	CMR %	pH	EI _H
3.4	86.9	3.7	26.9
6.0	88.6	6.0	32.3
8.2	87.0	8.0	37.0
10.0	78.0	10.0	40.5

Figure 9.46 Effect of calcium hydroxide addition in cell on combustible material recovery and efficiency index from wet-ground minus 200 mesh Illinois No. 6 coal.

pH	CMR %	pH	ELI
6.0	85.2	6.0	51.6
7.1	86.3	7.1	53.9
7.8	83.2	7.8	53.2
10.0	40.4	10.0	30.1

Figure 9.47 Effect of calcium hydroxide addition in cell on combustible material recovery and efficiency index from wet-ground minus 200 mesh Upper Freeport PA coal.

pH	CMR %	pH	ELI
3.5	69.5	3.5	42.9
6.0	77.7	6.0	53.1
8.0	76.5	8.0	56.8
10.0	76.5	10.0	55.5

Figure 9.48 Effect of sodium silicate addition on pyrite rejection from wet-ground minus 200 mesh Pittsburgh No. 8 coal.

NO DISPERSANT		2.2 LB/T SOD. SIL. IN MILL		1.1 LB/T SOD. SIL. IN MILL + 1.1 LB/T SOD. SIL. IN CELL	
CMR %	PSR %	CMR %	PSR %	CMR %	PSR %
30.2	90.6	35.6	90.1	40.6	87.8
56.1	82.5	67.0	79.3	67.7	79.3
74.5	76.6	84.5	72.7	81.8	74.3
84.8	73.2	88.3	71.1	84.8	73.4
90.9	71.0	90.3	70.2	87.6	72.3
		1.1 LB/T SOD. SIL. IN MILL + 1.1 LB/T IN CELL + 15 MIN. U.S.		1.1 LB/T SOD. SIL. IN MILL + 1.1 LB/T IN CELL + 30 MIN. U.S.	
		CMR %	PSR %	CMR %	PSR %
		38.7	88.5	41.8	86.2
		60.6	82.2	67.1	78.7
		82.2	74.2	83.5	72.9
		87.4	71.9	89.0	70.4
		91.4	69.7	92.0	68.6

Figure 9.49 Effect of sodium silicate addition on non-pyritic minerals rejection from wet-ground minus 200 mesh Pittsburgh No. 8 coal.

NO DISPERSANT		2.2 LB/T SOD. SIL. IN MILL		1.1 LB/T SOD. SIL. IN MILL + 1.1 LB/T SOD. SIL. IN CELL	
CMR %	PSR %	CMR %	PSR %	CMR %	PSR %
30.2	87.7	35.6	83.1	40.6	83.3
56.1	76.9	67.0	69.1	67.7	71.3
74.5	67.8	84.5	59.7	81.8	64.0
84.8	61.1	88.3	57.0	84.8	62.4
90.9	56.6	90.3	55.4	87.6	60.7
		1.1 LB/T SOD. SIL. IN MILL + 1.1 LB/T IN CELL + 15 MIN. U.S.		1.1 LB/T SOD. SIL. IN MILL + 1.1 LB/T IN CELL + 30 MIN. U.S.	
		CMR %	PSR %	CMR %	PSR %
		38.7	84.0	41.8	82.8
		60.6	74.8	67.1	71.2
		82.2	64.5	83.5	62.1
		87.4	61.5	89.0	59.0
		91.4	58.5	92.0	56.5

Figure 9.50 Effect of sodium hexametaphosphate addition on pyrite rejection from wet-ground minus 200 mesh Pittsburgh No. 8 coal.

DOD. + MIBC IN CELL		SOD. HEXAMETAPHOSPHATE + DOD. + MIBC	
CMR %	PSR %	CMR %	PSR %
66.4	82.3	91.9	68.0
83.5	74.5	91.0	68.7
87.6	73.6	88.5	68.6
90.9	69.5	88.6	69.0
		81.0	75.3
		84.0	74.6
		88.0	69.4
		86.4	74.8
		89.0	67.0
		90.0	62.0

Figure 9.51 Effect of sodium hexametaphosphate addition on non-pyritic minerals rejection from wet-ground minus 200 mesh Pittsburgh No. 8 coal.

DOD. + MIBC IN CELL		SOD. HEXAMETAPHOSPHATE + DOD. + MIBC	
CMR %	NPAR %	CMR %	NPAR %
66.4	74.6	91.9	57.9
83.5	63.1	91.0	59.2
87.6	55.4	88.5	60.6
90.9	54.9	88.6	62.4
		81.0	65.9
		84.0	64.5
		88.0	61.5
		86.4	62.3
		89.0	62.9
		90.0	63.4

Figure 10.2 Effect of grinding with GMTG on combustible matter recovery and ash and pyritic sulfur rejection in the flotation of Pittsburgh No. 8 coal.

0.0 LB/T GMTG			1.0 LB/T GMTG		
CMR %	AR %	PSR %	CMR %	AR %	PSR %
0.0	100.0	100.0	0.0	100.0	100.0
23.3	89.2	91.7	18.7	93.5	94.2
43.7	80.4	84.2	40.0	85.2	87.9
66.3	70.6	75.4	64.4	75.3	79.6
81.7	63.7	68.7	78.1	68.5	74.7
91.8	57.0	62.3	88.2	61.9	69.4

Figure 10.3 Effect of grinding with GMTG on combustible matter recovery and ash and pyritic sulfur rejection in the flotation of Illinois No. 6 coal.

1-STAGE GRIND W/O GMTG			1-STAGE GRIND W/ GMTG			2-STAGE GRIND W/O GMTG			2-STAGE GRIND W/ GMTG		
CMR %	AR %	PSR %	CMR %	AR %	PSR %	CMR %	AR %	PSR %	CMR %	AR %	PSR %
0.0	100.0	100.0	0.0	100.0	100.0	0.0	100.0	100.0	0.0	100.0	100.0
34.7	81.6	82.9	21.5	89.0	90.2	18.9	93.4	94.7	34.2	88.6	91.1
54.1	71.5	74.0	49.3	74.4	78.5	42.5	85.0	87.5	55.8	80.7	85.1
78.2	56.7	59.3	71.0	62.6	68.3	63.3	76.8	80.7	71.2	74.4	80.7
83.8	51.6	54.3	80.8	56.7	63.6	72.8	72.1	76.9	75.1	72.4	78.5
84.9	53.4	60.8	88.2	50.0	57.8	76.7	69.0	74.3	77.9	70.2	77.8

Figure 10.4 Effect of grinding with GMT on combustible matter recovery and ash and pyritic sulfur rejection in the flotation of Upper Freeport PA coal.

0.0 LB/T		1.0 LB/T GMTG	
CMR	PSR	CMR	PSR
$\bar{x}$	$\bar{x}$	$\bar{x}$	$\bar{x}$
0.0	100.0	0.0	100.0
36.6	90.7	32.5	93.0
59.8	80.6	53.0	84.9
72.7	73.1	64.7	74.9
80.3	65.9	72.7	68.8

Figure 10.5 Effect of grinding with depressants on combustible material recovery and pyritic sulfur rejection in the flotation of Pittsburgh No. 8 coal.

Two-stage flotation					
STANDARD		MCAA		MESA	
CMR	PSR	CMR	PSR	CMR	PSR
$\bar{x}$	$\bar{x}$	$\bar{x}$	$\bar{x}$	$\bar{x}$	$\bar{x}$
22.7	91.1	26.1	91.8	28.8	91.2
42.5	83.2	48.1	84.6	49.3	84.9
64.4	74.2	73.5	75.4	65.6	79.3
79.4	67.4	90.9	65.2	74.2	74.9
89.2	61.0				
AMAA		AESA		SSAA	
CMR	PSR	CMR	PSR	CMR	PSR
$\bar{x}$	$\bar{x}$	$\bar{x}$	$\bar{x}$	$\bar{x}$	$\bar{x}$
34.5	89.1	31.6	89.7	25.3	92.0
58.6	81.0	58.4	79.9	44.1	85.9
77.3	73.7	77.8	71.4	63.6	79.0
89.6	66.4	86.5	66	81.5	70.3
One-stage flotation					
STANDARD		MCAA		MESA	
CMR	PSR	CMR	PSR	CMR	PSR
$\bar{x}$	$\bar{x}$	$\bar{x}$	$\bar{x}$	$\bar{x}$	$\bar{x}$
26.8	87.2	32.1	86.1	27.3	88.5
49.7	76.5	56.4	75.7	49.8	79.1
74.5	64.2	81.1	63.3	75.6	67.1
89.1	55.1	91.6	55.5	91.1	57.4
AMAA		AESA		SSAA	
CMR	PSR	CMR	PSR	CMR	PSR
$\bar{x}$	$\bar{x}$	$\bar{x}$	$\bar{x}$	$\bar{x}$	$\bar{x}$
22.5	89.7	23.3	89.7	20.7	90.9
44.8	80.3	45.6	79.7	39.2	82.6
74.6	65.9	73.1	66.4	62.9	72.3
89.6	56.2	86.8	58.2	85.1	60.1

Figure 10.6 Effect of grinding with pyrite depressants on the kinetics of combustible material and pyritic sulfur recovery in the flotation of Pittsburgh No. 8 coal.

Flotation time, mins.	STANDARD		MESA		SSAA	
	CMR $\bar{x}$	PSR $\bar{x}$	CMR $\bar{x}$	PSR $\bar{x}$	CMR $\bar{x}$	PSR $\bar{x}$
0.5	26.8	12.8	27.3	11.5	20.7	9.1
1	49.7	23.5	49.8	20.9	39.2	17.2
2	74.5	35.8	75.6	32.9	62.9	27.7
5	89.1	44.9	91.1	42.6	85.1	29.9

Figure 10.7 Effect of xanthated polyacrylic acid and xanthated sucrose addition on pyrite rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal.

DOD. + MIBC IN CELL		POLY. XANTHATE + DOD + MIBC		SUCROSE XANTHATE + DOD. + MIBC	
CMR %	PSR %	CMR %	PSR %	CMR %	PSR %
66.4	82.3	79.2	78.3	88.0	75.1
83.5	74.5	85.1	74.9	86.7	77.3
87.6	73.6	89.7	71.0		
90.9	69.5	92.0	65.1		
		91.0	67.7		
		88.8	69.7		
		88.5	66.8		
		87.3	70.3		
		89.8	71.6		
		88.1	70.0		
		88.2	67.9		

Figure 10.8 Effect of xanthated polyacrylic acid and xanthated sucrose additions on non-pyritic minerals rejection from wet ground minus 200 mesh Pittsburgh No. 8 Coal.

DOD. + MIBC IN CELL		POLY. XANTHATE + DOD + MIBC		SUCROSE XANTHATE + DOD + MIBC	
CMR %	NPAR %	CMR %	NPAR %	CMR %	NPAR %
66.4	74.6	79.2	68.2	88.0	59.3
83.5	63.1	85.1	64.7	86.7	60.1
87.6	55.4	89.7	60.9		
90.9	54.9	92.0	57.0		
		91.0	57.6		
		88.8	59.1		
		88.5	61.3		
		87.3	60.7		
		89.8	59.7		
		88.1	62.2		
		88.2	61.1		

Figure 10.9 Effect of xanthated sucrose and xanthated citric acid additions on pyrite rejection from wet ground minus 325 mesh Pittsburgh No. 8 coal.

DOD. + MIBC IN CELL		SUCROSE XANTHATE + DOD. + MIBC		CITRIC XANTHATE + DOD. + MIBC	
CMR %	PSR %	CMR %	PSR %	CMR %	PSR %
79.7	85.3	84.6	78.1	88.2	79.5
86.8	79.7	82.8	80.0	70.4	81.0
87.3	76.8	79.4	81.7	83.9	79.1
89.0	76.9	76.7	81.2	88.8	81.1
89.2	77.2	77.6	81.6		
51.4	86.3	75.4	82.1		

Figure 10.10 Effect of xanthated sucrose and xanthated citric acid additions on non-pyritic minerals rejection from wet ground minus 325 mesh Pittsburgh No. 8 coal.

DOD + MIBC IN CELL		SUCROSE XANTHATE		CITRIC XANTHATE + DOD + MIBC	
CMR %	NPAR %	CMR %	NPAR %	CMR %	NPAR %
79.7	70.3	84.6	60.1	88.2	66.0
86.8	62.0	82.8	59.5	70.4	72.4
87.3	57.2	79.4	65.1	83.9	68.2
89.0	57.0	76.7	64.2	88.8	64.1
89.2	57.8	77.6	65.3		
51.4	74.2	75.4	66.2		

Figure 10.11 Effect of hydrogen peroxide addition on pyrite rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal.

MIBC		0.2% H ₂ O ₂ + MIBC		0.4% H ₂ O ₂ + MIBC		3.0 % H ₂ O ₂ + MIBC	
CMR %	PSR %	CMR %	PSR %	CMR %	PSR %	CMR %	PSR %
95.3	66.0	54.3	80.5	34.5	93.4	86.9	74.9
94.0	67.8	77.3	81.1	62.7	85.8	40.5	91.7
89.8	74.4	89.3	75.4	81.6	79.7	87.0	71.2
79.9	78.1	90.2	73.2	87.1	75.5	67.2	84.4
68.3	83.5						
43.5	90.7						

Figure 10.12 Effect of hydrogen peroxide addition on non-pyritic minerals rejection from wet ground minus 200 mesh Pittsburgh No. 8 coal.

MIBC		0.2% H ₂ O ₂ + MIBC		0.4% H ₂ O ₂ + MIBC		3.0 % H ₂ O ₂ + MIBC	
CMR %	PSR %	CMR %	PSR %	CMR %	PSR %	CMR %	PSR %
95.3	51.4	54.3	82.6	34.5	88.3	86.9	62.1
94.0	52.6	77.3	70.5	62.7	77.6	40.5	86.1
89.8	61.5	89.3	65.1	81.6	66.3	87.0	60.3
79.9	68.8	90.2	62.6	87.1	60.4	67.2	74.6
88.3	76.8						
43.5	86.1						

Note: The data for the figures in Chapter 11 was compiled into several tables, rather than having one table for each individual figure.

Table 11.1 Flotation kinetics of 28-mesh Illinois No. 6 coal.

	Sample ID	441-B1-04-W28										
	Dodecane	4.92 lb/T		MIBC		0.975 lb/T						
	pH	Initial 5.4		Final 5.5								
Size	Product	Weight	Yield	Ash	Tot.S	Pyr.S	CMR	Ash	Pyr. S	EI	Ash	Pyr. S
Mesh	Name	g	%	%	%	%	%	Rel. %	Rel. %	%	Rel. %	Rel. %
+48	Conc 0-0.25 min	51.64	54.31	8.26	3.55	0.96	56.96	64.23	65.21	22.17	35.77	34.79
	Conc 0.25-0.5 min	22.21	23.36	9.32	3.85	1.27	81.18	46.87	45.48	26.66	53.13	54.52
	Conc 0.5-1 min	6.40	6.73	8.82	3.46	0.88	88.20	42.14	41.53	29.72	57.86	58.47
	Tails	14.84	15.61	33.84	6.31	3.98						
	Feed +48	95.09	40.51	12.54	4.04	1.50						
48x100	Conc 0-0.25 min	35.50	66.06	9.28	3.80	1.22	68.39	50.44	53.23	21.61	49.56	46.77
	Conc 0.25-0.5 min	11.70	21.77	10.80	4.04	1.47	90.55	31.43	34.60	25.15	68.57	65.40
	Conc 0.5-1 min	3.54	6.59	9.91	3.61	1.04	97.32	26.16	30.62	27.94	73.84	69.38
	Tails	3.00	5.58	57.93	11.63	0.48						
	Feed 48x100	53.74	22.89	12.37	4.28	1.72						
100x200	Conc 0-0.25 min	20.91	64.76	9.28	3.64	1.06	66.83	50.34	59.79	26.62	49.66	40.21
	Conc 0.25-0.5 min	7.48	23.17	10.70	3.70	1.14	90.37	29.85	44.37	34.73	70.15	55.63
	Conc 0.5-1 min	2.31	7.15	10.68	3.79	1.23	97.64	23.54	39.24	36.87	76.46	60.76
	Tails	1.59	4.92	57.85	15.90	13.64						
	Feed 100x200	32.29	13.76	12.10	4.27	1.71						
-200	Conc 0-0.25 min	23.64	44.09	10.94	3.53	0.98	52.72	81.10	77.71	30.43	18.90	22.29
	Conc 0.25-0.5 min	11.48	21.41	11.11	3.68	1.12	78.27	71.78	65.24	43.51	28.22	34.76
	Conc 0.5-1 min	5.65	10.54	15.62	3.95	1.45	90.21	65.33	57.35	47.55	34.67	42.65
	Tails	12.85	23.96	69.55	6.50	4.62						
	Feed -200	53.62	22.84	25.52	4.32	1.93						
Overall	Conc 0-0.25 min	131.69	56.10	9.18	3.63	1.05	60.24	66.61	64.97	25.21	33.39	35.03
	Conc 0.25-0.5 min	52.87	22.50	10.22	3.83	1.26	84.12	51.70	48.10	32.22	48.30	51.90
	Conc 0.5-1 min	17.90	7.60	11.42	3.69	1.14	92.08	46.07	42.97	35.05	53.93	57.03
	Tails	32.28	13.80	51.47	7.35	5.22						
	Total Feed	234.74	100.00	15.42	4.19	1.68						

Table 11.2 Flotation kinetics of 28-mesh Pittsburgh No. 8 coal.

		Sample ID 442-R2-04-W28											
		Dodecane 1.92 lb/T		MIBC		0.39 lb/T							
pH		Initial 3.2		Final 3.25									
Size	Product	Weight	Yield	Ash	Tot.S	Pyr.S	CGR	Ash	Pyr.S	EI	Ash	Pyr.S	
Mesh	Name	g	%	%	%	%	%	Rel. %	Rel. %		Rel. %	Rel. %	
+48	Conc 0-0.25 min	110.98	70.46	7.16	3.03	1.63	72.93	51.02	43.36	16.29	48.98	56.64	
	Conc 0.25-0.5 min	19.08	12.11	8.08	3.01	1.71	85.34	41.51	33.19	18.53	58.49	66.81	
	Conc 0.5-1 min	12.06	7.66	6.61	2.94	1.52	93.32	36.60	27.44	20.75	63.40	72.58	
	Tails	15.38	9.77	38.63	4.83	5.69							
	Feed +48	157.50	43.00	10.30	3.20	2.03							
48x100	Conc 0-0.25 min	56.98	73.63	7.86	3.34	1.89	75.18	40.71	35.88	11.06	59.29	64.12	
	Conc 0.25-0.5 min	9.90	12.79	8.97	3.41	2.04	88.08	28.95	23.87	11.95	71.05	76.13	
	Conc 0.5-1 min	6.14	7.93	7.06	3.06	1.64	96.25	23.21	17.87	14.13	76.79	82.13	
	Tails	4.37	5.65	40.17	6.41	6.82							
	Feed 48x100	77.39	21.13	9.76	3.50	2.17							
100x200	Conc 0-0.25 min	34.51	72.67	8.00	3.68	2.11	74.27	41.75	38.30	12.57	58.25	61.70	
	Conc 0.25-0.5 min	6.30	13.27	9.90	3.98	2.48	87.54	28.59	25.10	12.64	71.41	74.90	
	Conc 0.5-1 min	4.11	8.65	7.27	3.44	1.90	96.46	22.29	18.51	14.97	77.71	81.49	
	Tails	2.57	5.41	41.16	9.06	8.56							
	Feed 100x200	47.49	12.96	9.98	3.99	2.49							
-200	Conc 0-0.25 min	48.98	58.35	8.40	3.92	2.30	65.71	73.73	62.81	28.53	26.27	37.19	
	Conc 0.25-0.5 min	11.86	14.13	9.00	3.97	2.39	81.52	66.92	53.47	34.99	33.08	46.53	
	Conc 0.5-1 min	8.31	9.90	10.58	4.22	2.69	92.40	61.30	46.09	38.49	38.70	53.91	
	Tails	14.79	17.62	64.95	6.86	9.41							
	Feed -200	83.94	22.91	18.66	4.47	3.61							
Overall	Conc 0-0.25 min	251.45	68.64	7.68	3.36	1.89	72.06	56.31	47.78	19.84	43.69	52.22	
	Conc 0.25-0.5 min	47.14	12.87	8.74	3.47	2.05	85.42	46.99	37.14	22.56	53.01	62.86	
	Conc 0.5-1 min	30.62	8.36	7.87	3.38	1.91	94.18	41.53	30.69	24.87	58.47	69.31	
	Tails	37.11	10.13	49.48	6.12	7.51							
	Total Feed	366.32	100.00	12.07	3.66	2.48							

Table 11.3 Flotation kinetics of 28-mesh Upper Freeport PA coal.

Sample ID		445-R3-03-W28											
Dodecane		0.24 lb/T		MIBC		0.26 lb/T							
pH		Initial 3.25		Final 3.3									
Size	Product	Weight	Yield	Ash	Tot. S	Pyr. S	CGR	Ash	Pyr. S	EI	Ash	Pyr. S	
Mesh	Name	%	%	%	%	%	%	Rel. %	Rel. %	%	Rec. %	Rec. %	
+48	Conc 0-0.25 min	84.62	56.11	7.69	1.28	0.51	60.49	69.97	69.80	30.29	30.03	30.20	
	Conc 0.25-0.5 min	13.12	8.70	8.58	1.27	0.51	69.78	64.78	65.12	34.89	35.22	34.88	
	Conc 0.5-1 min	12.48	8.28	7.75	1.24	0.47	78.69	60.32	60.99	39.68	39.68	39.01	
	Tails	40.59	26.91	32.20	2.71								
	Feed +48	150.81	40.46	14.37	1.66	0.94							
48x100	Conc 0-0.25 min	54.87	63.83	7.53	1.45	0.66	66.53	57.39	61.81	28.33	42.61	38.19	
	Conc 0.25-0.5 min	9.35	10.86	8.90	1.50	0.73	77.68	48.82	54.70	32.38	51.18	45.30	
	Conc 0.5-1 min	10.67	12.39	8.28	1.41	0.63	90.48	39.73	47.62	38.10	60.27	52.38	
	Tails	11.13	12.92	34.70	4.81								
	Feed 48x100	86.12	23.10	11.28	1.88	1.11							
100x200	Conc 0-0.25 min	31.39	60.42	7.00	1.52	0.72	62.98	60.73	66.33	29.31	39.27	33.67	
	Conc 0.25-0.5 min	7.00	13.47	8.39	1.66	0.87	76.81	50.23	57.30	34.11	49.77	42.70	
	Conc 0.5-1 min	7.57	14.57	8.67	1.69	0.90	91.72	38.50	47.19	38.91	61.50	52.81	
	Tails	5.99	11.53	36.00	6.10								
	Feed 100x200	51.95	13.94	10.77	2.09	1.30							
-200	Conc 0-0.25 min	34.16	40.72	6.56	1.59	0.79	43.92	80.02	80.85	24.76	19.98	19.15	
	Conc 0.25-0.5 min	11.73	13.98	6.85	1.57	0.77	58.95	72.86	74.40	33.35	27.14	25.60	
	Conc 0.5-1 min	17.58	20.95	7.58	1.63	0.83	81.30	60.98	63.94	45.24	39.02	36.06	
	Tails	20.43	24.35	33.50	5.11								
	Feed -200	83.90	22.51	13.37	2.45	1.67							
Overall	Conc 0-0.25 min	205.14	55.03	7.35	1.41	0.63	58.55	68.70	70.94	29.49	31.30	29.06	
	Conc 0.25-0.5 min	41.20	11.05	8.13	1.47	0.69	70.22	61.76	64.51	34.73	38.24	35.49	
	Conc 0.5-1 min	48.30	12.96	7.95	1.49	0.71	83.91	53.79	56.82	40.73	46.21	43.18	
	Tails	78.14	20.96	33.19	3.90	3.24							
	Total Feed	372.78	100.00	12.93	1.95	1.19							

Table 11.4 Flotation kinetics of 100-mesh Illinois No. 6 coal.

		Sample ID		467-B1-04-W100																	
		Dodecane		4.82 lb/T		MIBC		0.875 lb/T													
		pH		Initial 4.21		Final 4.49															
Size	Product	Weight	Yield	Ash	Tot.S	Pyr.S	CMR	Ash	Pyr. S	EI	Ash	Pyr. S									
Mesh	Name	g	%	%	%	%	%	Rel. %	Rel. %	%	Rel. %	Rel. %									
+200	Conc 0-0.25 min	74.09	39.17	8.19	3.80	1.20	40.33	70.35	76.42	16.75	29.65	23.58									
	Conc 0.25-0.5 min	50.04	26.46	8.37	3.76	1.17	67.51	49.89	60.98	28.49	50.11	39.02									
	Conc 0.5-1 min	19.33	10.22	8.12	3.64	1.05	78.04	42.22	55.63	33.67	57.78	44.37									
	Conc 1-2 min	4.72	2.50	8.87	3.52	0.94	80.59	40.17	54.46	35.05	59.83	45.54									
	Conc 2-5 min	2.10	1.11	9.82	3.37	0.81	81.71	39.16	54.01	35.72	60.84	45.99									
	Tails	38.87	20.55	20.63	7.78	5.24															
	Feed +200	189.15	42.39	10.82	4.58	2.00															
200x400	Conc 0-0.25 min	28.22	32.28	8.52	4.34	1.73	33.41	76.31	79.11	12.51	23.69	20.89									
	Conc 0.25-0.5 min	21.98	25.14	8.28	4.13	1.53	59.50	58.38	64.78	24.28	41.62	35.22									
	Conc 0.5-1 min	14.91	17.06	8.07	3.73	1.13	77.24	46.52	57.56	34.80	53.48	42.44									
	Conc 1-2 min	5.58	6.38	9.25	3.74	1.16	83.79	41.44	54.80	38.59	58.56	45.20									
	Conc 2-5 min	3.15	3.60	10.56	3.74	1.18	87.44	38.16	53.22	40.66	61.84	46.78									
	Tails	13.58	15.53	28.54	11.71	9.18															
	Feed 200x400	87.42	19.59	11.61	5.27	2.68															
-400	Conc 0-0.25 min	42.27	24.92	12.23	4.41	1.85	29.07	87.69	83.65	12.72	12.31	16.35									
	Conc 0.25-0.5 min	34.51	20.35	11.47	4.39	1.82	53.01	78.27	70.51	23.52	21.73	29.49									
	Conc 0.5-1 min	28.45	16.77	12.41	4.40	1.84	72.54	69.86	59.55	32.08	30.14	40.45									
	Conc 1-2 min	17.76	10.47	19.08	3.72	1.27	83.80	61.79	54.85	38.64	38.21	45.15									
	Conc 2-5 min	11.14	6.57	28.79	5.22	2.85	90.01	54.15	48.20	38.22	45.85	51.80									
	Tails	35.49	20.92	64.07	8.51	6.51															
	Feed -400	169.62	38.02	24.76	5.24	2.82															
Overall	Conc 0-0.25 min	144.58	32.40	9.43	4.08	1.50	35.05	81.22	80.13	15.18	18.78	19.87									
	Conc 0.25-0.5 min	106.53	23.90	9.36	4.04	1.45	60.93	67.49	65.90	26.83	32.51	34.10									
	Conc 0.5-1 min	62.69	14.00	10.06	4.01	1.43	75.97	58.84	57.70	33.67	41.16	42.30									
	Conc 1-2 min	28.06	6.30	15.41	3.69	1.19	82.34	52.88	54.63	36.97	47.12	45.37									
	Conc 2-5 min	16.39	3.70	22.86	4.70	2.27	85.75	47.68	51.19	36.94	52.32	48.81									
	Tails	87.94	19.70	39.40	8.68	6.36															
	Total Feed	446.19	100.00	16.28	4.97	2.44															

Table 11.5 Flotation kinetics of 100-mesh Pittsburgh No. 8 coal.

		468-B2-04-W100										
		Dodecane		1.92 lb/T		MIBC		0.39 lb/T				
		pH		Initial 3.24		Final 3.39						
Size	Product	Weight	Yield	Ash	Tot.S	Pyr.S	CMR	Ash	Pyr. S	EI	Ash	Pyr. S
Mesh	Name	g	%	%	%	%	%	Rel. %	Rel. %	%	Rel. %	Rel. %
+200	Conc 0-0.25 min	63.90	32.52	5.40	2.83	1.34	33.44	77.99	76.52	9.95	22.01	23.48
	Conc 0.25-0.5 min	44.67	22.74	5.35	2.70	1.26	56.82	62.75	61.15	17.97	37.25	38.85
	Conc 0.5-1 min	24.12	12.28	5.01	2.66	1.20	69.49	55.04	53.22	22.72	44.96	46.78
	Conc 1-2 min	7.92	4.03	5.47	2.65	1.24	73.84	52.28	50.54	24.18	47.72	49.46
	Conc 2-5 min	4.78	2.43	8.12	2.76	1.55	76.07	49.80	48.51	24.57	50.20	51.49
	Tails	51.08	26.00	15.29	4.77	3.47						
	Feed +200	196.47	41.31	7.98	3.27	1.86						
200x400	Conc 0-0.25 min	29.81	28.41	5.81	3.26	1.65	29.32	81.07	81.63	10.95	18.93	18.37
	Conc 0.25-0.5 min	24.65	23.49	5.53	3.12	1.54	53.63	66.17	67.49	21.12	33.83	32.51
	Conc 0.5-1 min	18.08	17.23	5.23	2.91	1.38	71.52	55.83	58.19	29.71	44.17	41.81
	Conc 1-2 min	8.65	8.24	5.88	2.86	1.41	80.02	50.28	53.64	33.66	49.72	46.36
	Conc 2-5 min	6.40	6.10	7.80	3.32	1.87	86.19	44.82	49.16	35.35	55.18	50.84
	Tails	17.33	16.52	23.67	10.12	7.58						
	Feed 200x400	104.92	22.06	8.72	4.27	2.55						
-400	Conc 0-0.25 min	41.56	23.86	6.80	3.04	1.60	26.09	89.01	87.57	13.66	10.99	12.43
	Conc 0.25-0.5 min	35.67	20.48	6.22	2.96	1.50	48.62	80.38	77.59	26.22	19.62	22.41
	Conc 0.5-1 min	31.96	18.35	6.10	2.90	1.45	68.84	72.79	68.94	37.78	27.21	31.06
	Conc 1-2 min	19.46	11.17	7.62	3.01	1.66	80.95	67.02	62.91	43.86	32.98	37.09
	Conc 2-5 min	14.85	8.53	12.51	3.90	2.67	89.70	59.80	55.52	45.22	40.20	44.48
	Tails	30.66	17.60	50.13	9.53	9.69						
	Feed -400	174.16	36.62	14.76	4.21	3.08						
Overall	Conc 0-0.25 min	135.27	28.44	5.92	2.99	1.49	29.94	84.14	82.77	12.71	15.86	17.23
	Conc 0.25-0.5 min	104.99	22.08	5.69	2.89	1.41	53.24	72.32	70.15	23.39	27.68	29.85
	Conc 0.5-1 min	74.16	15.59	5.53	2.82	1.35	69.72	64.19	61.58	31.30	35.81	38.42
	Conc 1-2 min	36.03	7.58	6.73	2.89	1.50	77.62	59.39	56.94	34.56	40.61	43.06
	Conc 2-5 min	26.03	5.47	10.54	3.55	2.27	83.10	53.96	51.89	34.99	46.04	48.11
	Tails	99.07	20.83	27.52	7.18	6.11						
	Total Feed	475.55	100.00	10.62	3.84	2.46						

Table 11.6 Flotation kinetics of 100-mesh Upper Freeport PA coal.

		Sample ID 469-B3-04-W100										
		Dodecane		0.24 lb/T		MIBC		0.26 lb/T				
		pH		Initial 3.25		Final 3.39						
Size	Product	Weight	Yield	Ash	Tot.S	Pyr.S	CMR	Ash	Pyr. S	EI	Ash	Pyr. S
Mesh	Name	%	%	%	%	%	%	Rel. %	Rel. %	%	Rel. %	Rel. %
+200	Conc 0-0.25 min	40.02	22.85	5.68	1.22	0.43	24.61	89.53	91.85	16.46	10.47	8.15
	Conc 0.25-0.5 min	36.14	20.64	5.84	1.26	0.47	46.79	79.81	83.82	30.61	20.19	16.18
	Conc 0.5-1 min	31.69	18.10	6.57	1.18	0.40	66.09	70.22	77.79	43.89	29.78	22.21
	Conc 1-2 min	20.83	11.90	8.53	1.35	0.58	78.51	62.04	72.03	50.55	37.96	27.97
	Conc 2-5 min	12.83	7.33	13.26	1.67	0.93	85.77	54.20	66.34	52.11	45.80	33.66
	Tails	33.60	19.19	35.04	4.84	4.15						
	Feed +200	175.11	36.44	12.40	1.96	1.20						
200x400	Conc 0-0.25 min	25.96	23.42	5.85	1.42	0.62	25.03	88.51	92.22	17.25	11.49	7.78
	Conc 0.25-0.5 min	25.27	22.79	6.03	1.38	0.58	49.35	76.98	85.09	34.44	23.02	14.91
	Conc 0.5-1 min	22.49	20.29	6.65	1.38	0.59	70.85	65.66	78.67	49.52	34.34	21.33
	Conc 1-2 min	14.23	12.84	8.92	1.61	0.83	84.12	56.05	72.94	57.06	43.95	27.06
	Conc 2-5 min	9.08	8.19	15.31	2.74	1.96	92.00	45.53	64.30	56.30	54.47	35.70
	Tails	13.83	12.48	43.51	10.50	9.57						
	Feed 200x400	110.86	23.07	11.92	2.67	1.86						
-400	Conc 0-0.25 min	38.41	19.74	6.90	1.58	0.78	21.41	90.38	91.98	13.39	9.62	8.02
	Conc 0.25-0.5 min	37.06	19.04	6.71	1.55	0.75	42.10	81.36	84.55	26.65	18.64	15.45
	Conc 0.5-1 min	39.67	20.38	6.70	1.48	0.68	64.26	71.71	77.29	41.55	28.29	22.71
	Conc 1-2 min	31.34	16.10	7.89	1.67	0.88	81.54	62.74	69.95	51.49	37.26	30.05
	Conc 2-5 min	19.44	9.99	14.05	2.63	1.85	91.54	52.83	60.35	51.89	47.17	39.65
	Tails	28.69	14.74	50.71	8.60	7.86						
	Feed -400	194.61	40.49	14.16	2.71	1.92						
Overall	Conc 0-0.25 min	104.39	21.72	6.17	1.40	0.60	23.43	89.69	91.99	15.42	10.31	8.01
	Conc 0.25-0.5 min	98.47	20.49	6.22	1.40	0.60	45.51	79.89	84.46	29.97	20.11	15.54
	Conc 0.5-1 min	93.85	19.53	6.64	1.35	0.56	66.47	69.91	77.73	44.20	30.09	22.27
	Conc 1-2 min	66.40	13.82	8.31	1.56	0.78	81.03	61.08	71.22	52.25	38.92	28.78
	Conc 2-5 min	41.35	8.60	14.08	2.36	1.59	89.53	51.76	62.89	52.42	48.24	37.11
	Tails	76.12	15.84	42.48	7.29	6.54						
	Total Feed	480.58	100.00	13.00	2.43	1.64						

Table 11.7 Flotation kinetics of 200-mesh Illinois No. 6 coal with the standard dosages.

	Sample ID	471-B1-04-W200										
	Dodecane	4.92 lb/T		MIBC		0.98 lb/T						
	pH	Initial 4.01		Final 4.07								
Size	Product	Weight	Yield	Ash	Tot.S	Pyr.S	CMR	Ash	Pyr. S	EI	Ash	Pyr. S
<u>Mesh</u>	<u>Name</u>	<u>g</u>	<u>g</u>	<u>g</u>	<u>g</u>	<u>g</u>	<u>g</u>	<u>Rel. g</u>	<u>Rel. g</u>	<u>g</u>	<u>Rel. g</u>	<u>Rel. g</u>
+400	Conc 0-0.25 min	16.52	11.16	6.50	3.29	0.68	11.47	91.99	94.83	6.31	8.01	5.17
	Conc 0.25-0.5 min	15.24	10.30	5.99	3.27	0.66	22.12	85.19	90.25	12.37	14.81	9.75
	Conc 0.5-1 min	18.22	12.31	5.96	3.24	0.63	34.85	77.09	85.02	19.86	22.91	14.98
	Conc 1-2 min	14.69	9.92	6.06	3.17	0.56	45.10	70.45	81.25	26.35	29.55	18.75
	Conc 2-5 min	20.25	13.68	6.40	3.26	0.65	59.18	60.79	75.20	34.38	39.21	24.80
	Tails	63.10	42.63	12.91	5.17	2.60						
	Feed +400	148.02	34.38	9.06	4.07	1.48						
-400	Conc 0-0.25 min	40.52	14.35	9.35	3.77	1.19	15.58	91.89	92.64	8.22	8.11	7.36
	Conc 0.25-0.5 min	44.00	15.58	8.66	3.74	1.15	32.63	83.72	84.91	17.54	16.28	15.09
	Conc 0.5-1 min	53.63	18.98	8.78	3.72	1.13	53.38	73.64	75.64	29.01	26.36	24.36
	Conc 1-2 min	40.86	14.47	9.24	3.78	1.20	69.11	65.55	68.17	37.27	34.45	31.83
	Conc 2-5 min	41.65	14.75	11.89	4.31	1.75	84.67	54.95	57.05	41.73	45.05	42.95
	Tails	61.80	21.88	41.49	8.33	6.05						
	Feed -400	282.46	65.62	16.53	4.83	2.32						
Overall	Conc 0-0.25 min	57.04	13.25	8.52	3.63	1.04	14.09	91.91	93.19	7.28	8.09	6.81
	Conc 0.25-0.5 min	59.24	13.76	7.97	3.62	1.03	28.81	84.05	86.24	15.05	15.95	13.76
	Conc 0.5-1 min	71.85	16.69	8.06	3.60	1.01	46.64	74.41	77.98	24.62	25.59	22.02
	Conc 1-2 min	55.55	12.90	8.40	3.62	1.03	60.38	66.64	71.43	31.81	33.36	28.57
	Conc 2-5 min	61.90	14.38	10.09	3.97	1.39	75.40	56.25	61.58	36.98	43.75	38.42
	Tails	124.90	29.01	27.05	6.73	4.30						
	Total Feed	430.48	100.00	13.95	4.57	2.03						

Table 11.8 Flotation kinetics of 200-mesh Illinois No. 6 coal with double dosage of collector.

Sample ID		474-B1-04-W200										
Dodecane		9.82 lb/T		MIBC		0.98 lb/T						
pH		Initial 3.87		Final 3.97								
Size	Product	Weight	Yield	Ash	Tot.S	Pyr.S	CMR	Ash	Pyr. S	EI	Ash	Pyr. S
Mesh	Name	%	%	%	%	%	%	Rel. %	Rel. %	%	Rec. %	Rec. %
+400	Conc 0-0.25 min	24.57	15.64	6.74	3.46	0.85	16.03	88.26	90.73	6.75	11.74	9.27
	Conc 0.25-0.5 min	21.58	13.74	6.26	3.38	0.77	30.17	78.68	83.38	13.56	21.32	16.62
	Conc 0.5-1 min	25.02	15.93	6.34	3.34	0.73	46.56	67.44	75.29	21.85	32.56	24.71
	Conc 1-2 min	19.49	12.41	6.40	3.20	0.60	59.32	58.60	70.16	29.48	41.40	29.84
	Conc 2-5 min	17.06	10.86	7.09	3.33	0.73	70.41	50.02	64.64	35.05	49.98	35.36
	Tails	49.37	31.43	14.29	5.52	2.96						
	Feed +400	157.09	35.67	8.98	4.03	1.44						
-400	Conc 0-0.25 min	49.57	17.51	10.12	4.10	1.52	19.21	90.18	89.21	8.42	9.82	10.79
	Conc 0.25-0.5 min	48.56	17.16	9.09	3.96	1.37	38.24	81.53	79.69	17.92	18.47	20.31
	Conc 0.5-1 min	58.13	20.54	9.43	4.04	1.45	60.93	70.79	67.60	28.53	29.21	32.40
	Conc 1-2 min	43.03	15.20	10.47	4.14	1.56	77.54	61.97	57.97	35.51	38.03	42.03
	Conc 2-5 min	33.16	11.72	13.86	4.63	2.09	89.85	52.97	48.07	37.93	47.03	51.93
	Tails	50.85	17.97	53.19	8.75	6.61						
	Feed -400	283.30	64.33	18.04	4.97	2.47						
Overall	Conc 0-0.25 min	74.14	16.84	9.00	3.89	1.30	17.98	89.76	89.58	7.56	10.24	10.42
	Conc 0.25-0.5 min	70.14	15.93	8.22	3.78	1.18	35.14	80.92	80.58	15.72	19.08	19.42
	Conc 0.5-1 min	83.15	18.88	8.50	3.83	1.24	55.42	70.07	69.46	24.88	29.93	30.54
	Conc 1-2 min	62.52	14.20	9.20	3.85	1.27	70.54	61.25	60.93	31.47	38.75	39.07
	Conc 2-5 min	50.22	11.40	11.56	4.19	1.63	82.38	52.34	52.10	34.48	47.66	47.80
	Tails	100.22	22.76	34.03	7.16	4.81						
	Total Feed	440.39	100.00	14.80	4.63	2.10						

Table 11.9 Flotation kinetics of 200-mesh Illinois No. 6 coal with double dosage of frother.

	Sample ID	477-B1-04-W200										
	Dodecane	4.92 lb/T		MIBC		1.95 lb/T						
	pH	Initial 3.84		Final 3.98								
Size	Product	Weight	Yield	Ash	Tot.S	Pyr.S	CMR	Ash	Pyr. S	EI	Ash	Pyr. S
Mesh	Name	%	%	%	%	%	%	Rel. %	Rel. %	%	Rec. %	Rec. %
+400	Conc 0-0.25 min	21.40	18.26	7.79	3.72	1.12	18.76	86.14	89.40	8.16	13.86	10.60
	Conc 0.25-0.5 min	18.23	15.55	7.25	3.49	0.89	34.84	75.15	82.23	17.07	24.85	17.77
	Conc 0.5-1 min	23.22	19.81	7.47	3.51	0.91	55.26	60.72	72.87	28.13	39.28	27.13
	Conc 1-2 min	15.82	13.50	7.80	3.54	0.95	69.13	50.46	66.26	35.39	49.54	33.74
	Conc 2-5 min	14.40	12.29	9.08	3.63	1.05	81.58	39.59	59.58	41.15	60.41	40.42
	Tails	24.14	20.60	19.73	8.15	5.59						
	Feed +400	117.21	36.08	10.26	4.52	1.93						
-400	Conc 0-0.25 min	44.43	21.40	11.81	4.52	1.95	23.23	86.52	85.98	9.21	13.48	14.02
	Conc 0.25-0.5 min	39.66	19.10	10.34	4.18	1.60	44.30	75.99	75.71	20.02	24.01	24.29
	Conc 0.5-1 min	47.35	22.80	11.40	4.42	1.85	69.17	62.12	61.56	30.73	37.88	38.44
	Conc 1-2 min	30.57	14.72	14.16	4.89	2.34	84.73	51.00	49.98	34.71	49.00	50.02
	Conc 2-5 min	21.15	10.19	22.99	6.27	3.80	94.38	38.51	36.98	31.37	61.49	63.02
	Tails	24.47	11.79	61.30	11.46	9.35						
	Feed -400	207.63	63.92	18.75	5.48	2.98						
Overall	Conc 0-0.25 min	65.83	20.27	10.50	4.26	1.68	21.51	86.43	86.89	8.40	13.57	13.11
	Conc 0.25-0.5 min	57.89	17.82	9.37	3.96	1.38	40.67	75.79	77.45	18.12	24.21	22.55
	Conc 0.5-1 min	70.57	21.72	10.11	4.12	1.54	63.83	61.80	64.57	28.40	38.20	35.43
	Conc 1-2 min	46.39	14.28	11.99	4.43	1.87	78.74	50.89	54.32	33.06	49.11	45.68
	Conc 2-5 min	35.55	10.94	17.36	5.20	2.69	89.47	38.78	43.01	32.48	61.22	56.99
	Tails	48.61	14.96	40.66	9.82	7.49						
	Total Feed	324.84	100.00	15.69	5.13	2.60						

Table 11.10 Flotation kinetics of 200-mesh Pittsburgh No. 8 coal with the standard dosages.

Sample ID		473-B2-04-W200										
Dodecane		1.92 lb/T		MIBC		0.30 lb/T						
pH		Initial 3.64		Final 3.71								
Size	Product	Weight	Yield	Ash	Tot.S	Pyr.S	CMR	Ash	Pyr. S	EI	Ash	Pyr. S
Mesh	Name	g	%	%	%	%	%	Rel. %	Rel. %		Rec. %	Rec. %
+400	Conc 0-0.25 min	8.34	5.46	4.75	2.42	1.03	5.57	98.15	96.62	2.20	3.85	3.38
	Conc 0.25-0.5 min	11.03	7.22	4.45	2.28	0.91	12.97	91.39	92.65	5.63	8.61	7.35
	Conc 0.5-1 min	20.99	13.74	4.21	2.30	0.90	27.08	82.81	85.19	12.27	17.19	14.81
	Conc 1-2 min	31.77	20.79	4.15	2.36	0.93	48.45	70.00	73.49	21.94	30.00	26.51
	Conc 2-5 min	29.04	19.01	4.33	2.48	1.03	67.95	57.79	61.75	29.70	42.21	38.25
	Tails	51.62	33.78	11.53	4.63	3.03						
	Feed +400	152.79	32.68	6.74	3.14	1.66						
-400	Conc 0-0.25 min	32.48	10.32	6.07	2.72	1.34	11.01	94.75	95.07	6.08	5.25	4.93
	Conc 0.25-0.5 min	41.38	13.15	5.48	2.62	1.22	25.12	88.71	89.34	14.46	11.29	10.66
	Conc 0.5-1 min	62.97	20.01	5.32	2.63	1.21	46.63	79.78	80.69	27.32	20.22	19.31
	Conc 1-2 min	69.10	21.96	5.12	2.78	1.29	70.29	70.36	70.60	40.89	29.64	29.40
	Conc 2-5 min	48.46	15.40	7.36	3.31	1.82	86.49	60.86	60.57	47.06	39.14	39.43
	Tails	60.29	19.16	37.89	10.03	8.85						
	Feed -400	314.68	67.32	11.93	4.19	2.80						
Overall	Conc 0-0.25 min	40.82	8.73	5.80	2.66	1.27	9.16	95.05	95.42	4.58	4.95	4.58
	Conc 0.25-0.5 min	52.41	11.21	5.26	2.55	1.16	20.99	89.28	90.09	11.08	10.72	9.91
	Conc 0.5-1 min	83.96	17.96	5.04	2.55	1.14	39.99	80.43	81.71	21.70	19.57	18.29
	Conc 1-2 min	100.87	21.58	4.81	2.65	1.18	62.87	70.27	71.28	34.15	29.73	28.72
	Conc 2-5 min	77.50	16.58	6.22	3.00	1.53	80.19	60.19	60.87	41.06	39.81	39.13
	Tails	111.91	23.94	25.73	7.54	6.17						
	Total Feed	467.47	100.00	10.23	3.85	2.43						

Table 11.11 Flotation kinetics of 200-mesh Pittsburgh No. 8 coal with double dosage of collector.

	Sample ID	475-B2-04-W200										
	Dodecane	3.84 lb/T		MIBC		0.30 lb/T						
	pH	Initial 3.64		Final 3.78								
Size	Product	Weight	Yield	Ash	Tot.S	Pyr.S	CMR	Ash	Pyr. S	EI	Ash	Pyr. S
<u>Mesh</u>	<u>Name</u>	<u>g</u>	<u>%</u>	<u>%</u>	<u>%</u>	<u>%</u>	<u>%</u>	<u>Rel. %</u>	<u>Rel. %</u>		<u>Rec. %</u>	<u>Rec. %</u>
+400	Conc 0-0.25 min	8.66	10.34	4.68	2.35	0.98	10.62	93.27	93.08	3.70	6.73	6.92
	Conc 0.25-0.5 min	10.96	13.09	4.40	2.29	0.91	24.10	85.26	84.89	8.99	14.74	15.11
	Conc 0.5-1 min	16.52	19.72	4.40	2.30	0.92	44.41	73.19	72.46	16.87	26.81	27.54
	Conc 1-2 min	16.93	20.21	4.77	2.36	0.99	65.15	59.78	58.73	23.88	40.22	41.27
	Conc 2-5 min	11.60	13.85	5.95	2.44	1.15	79.19	48.32	47.80	26.99	51.68	52.20
	Tails	19.09	22.79	15.23	4.13	3.07						
	Feed +400	83.76	27.86	7.19	2.75	1.46						
-400	Conc 0-0.25 min	34.02	15.68	5.63	2.62	1.23	16.80	92.88	93.08	9.96	7.12	6.94
	Conc 0.25-0.5 min	36.13	16.66	5.39	2.60	1.20	34.88	85.64	85.91	20.79	14.36	14.09
	Conc 0.5-1 min	47.49	21.89	5.62	2.78	1.33	58.47	75.72	75.45	33.92	24.28	24.55
	Conc 1-2 min	39.38	18.15	6.54	2.94	1.52	77.84	66.14	65.57	43.41	33.86	34.43
	Conc 2-5 min	24.12	11.12	9.62	3.57	2.20	89.31	57.52	56.82	46.13	42.48	43.18
	Tails	35.78	16.49	43.21	10.41	9.59						
	Feed -400	216.92	72.14	12.40	4.10	2.79						
Overall	Conc 0-0.25 min	42.68	14.19	5.44	2.57	1.19	15.07	92.95	93.07	8.14	7.05	6.93
	Conc 0.25-0.5 min	47.09	15.66	5.16	2.53	1.13	31.75	85.56	85.74	17.49	14.44	14.26
	Conc 0.5-1 min	64.01	21.29	5.31	2.66	1.23	54.38	75.24	74.95	29.33	24.76	25.05
	Conc 1-2 min	56.31	18.73	6.01	2.77	1.36	74.15	64.95	64.43	38.58	35.05	35.57
	Conc 2-5 min	35.72	11.88	8.43	3.20	1.86	86.36	55.80	55.31	41.67	44.20	44.69
	Tails	54.87	18.25	33.48	8.23	7.32						
	Total Feed	300.68	100.00	10.95	3.73	2.42						

Table 11.12 Flotation kinetics of 200-mesh Pittsburgh No. 8 coal with double dosage of frother.

		Sample ID		478-B2-04-W200											
		Dodecane		1.92 lb/T		MIBC		0.60 lb/T							
		pH		Initial 3.55		Final 3.70									
Size	Product	Weight	Yield	Ash	Tot.S	Pyr.S	CMR	Ash	Pyr. S	EI	Ash	Pyr. S			
Mesh	Name	%	%	%	%	%	%	Rel. %	Rel. %	%	Rel. %	Rel. %			
+400	Conc 0-0.25 min	13.77	12.62	5.12	2.38	1.04	12.88	90.60	90.97	3.83	9.40	9.03			
	Conc 0.25-0.5 min	15.62	14.31	4.77	2.24	0.92	27.49	80.66	81.92	9.41	18.34	18.08			
	Conc 0.5-1 min	26.24	24.04	4.66	2.28	0.93	52.11	64.35	66.46	18.57	35.65	33.54			
	Conc 1-2 min	30.61	28.05	5.32	2.30	1.01	80.62	42.83	47.00	27.62	57.37	53.00			
	Conc 2-5 min	16.48	15.10	8.17	2.74	1.55	95.51	34.67	30.90	28.41	75.33	69.10			
	Tails	6.41	5.87	28.78	9.30	7.55									
	Feed +400	109.13	30.32	6.87	2.77	1.45									
-400	Conc 0-0.25 min	47.16	18.80	6.40	2.84	1.44	20.02	90.03	90.14	10.15	9.97	9.86			
	Conc 0.25-0.5 min	46.76	18.64	5.83	2.72	1.31	39.98	81.02	81.22	21.21	18.98	18.78			
	Conc 0.5-1 min	62.17	24.79	5.97	2.82	1.39	66.49	68.76	68.69	35.18	31.24	31.31			
	Conc 1-2 min	51.81	20.66	7.28	3.28	1.80	88.27	56.31	55.19	43.46	43.69	44.81			
	Conc 2-5 min	22.53	8.98	14.21	5.59	3.88	97.04	45.73	42.51	39.54	54.27	57.49			
	Tails	20.38	8.13	67.87	14.51	14.44									
	Feed -400	250.81	69.68	12.07	4.10	2.75									
Overall	Conc 0-0.25 min	60.93	16.93	6.11	2.74	1.35	17.78	90.14	90.31	8.07	9.86	9.69			
	Conc 0.25-0.5 min	62.38	17.33	5.56	2.60	1.21	36.04	80.95	81.39	17.43	19.05	18.61			
	Conc 0.5-1 min	88.41	24.56	5.58	2.66	1.25	81.95	67.88	68.33	30.28	32.12	31.67			
	Conc 1-2 min	82.42	22.90	6.55	2.92	1.51	85.88	53.58	53.74	39.60	46.42	46.26			
	Conc 2-5 min	39.01	10.84	11.66	4.39	2.90	96.55	41.53	40.45	37.00	58.47	59.55			
	Tails	26.79	7.44	58.52	13.26	12.79									
	Total Feed	359.84	100.00	10.49	3.70	2.36									

Table 11.13 Flotation kinetics of 200-mesh Upper Freeport PA coal with the standard dosages.

		Sample ID		470-B3-04-W200											
		Dodecane		0.24 lb/T		MIBC		0.26 lb/T							
		pH		Initial 3.40		Final 3.51									
Size	Product	Weight	Yield	Ash	Tot.S	Pyr.S	CMR	Ash	Pyr. S	EI	Ash	Pyr. S			
Mesh	Name	%	%	%	%	%	%	Rel. %	Rel. %	%	Rel. %	Rel. %			
+400	Conc 0-0.25 min	9.22	7.40	5.52	1.14	0.35	7.88	96.37	97.62	5.50	3.63	2.38			
	Conc 0.25-0.5 min	11.47	9.21	5.20	1.13	0.34	17.71	92.12	94.77	12.48	7.88	5.23			
	Conc 0.5-1 min	19.34	15.52	4.95	1.03	0.24	34.34	85.30	91.33	25.67	14.70	8.67			
	Conc 1-2 min	26.28	21.09	5.24	1.03	0.24	56.86	75.48	68.61	43.47	24.52	13.39			
	Conc 2-5 min	25.37	20.36	7.76	1.19	0.42	78.03	61.45	78.73	56.76	38.55	21.27			
	Tails	32.91	26.41	26.21	3.98	3.25									
	Feed +400	124.59	26.02	11.26	1.86	1.09									
-400	Conc 0-0.25 min	42.89	12.11	6.27	1.44	0.64	13.03	94.10	95.56	8.59	5.90	4.44			
	Conc 0.25-0.5 min	48.31	13.64	6.08	1.41	0.61	27.74	87.67	90.80	18.54	12.33	9.20			
	Conc 0.5-1 min	69.70	19.68	6.04	1.34	0.54	48.96	78.44	84.68	33.64	21.56	15.32			
	Conc 1-2 min	74.94	21.16	6.45	1.36	0.57	71.68	67.84	77.81	49.49	32.16	22.19			
	Conc 2-5 min	56.02	15.82	9.44	1.71	0.93	88.13	56.25	60.41	57.53	43.75	30.59			
	Tails	62.29	17.59	41.17	7.68	6.89									
	Feed -400	354.15	73.98	12.88	2.54	1.75									
Overall	Conc 0-0.25 min	52.11	10.88	6.14	1.39	0.59	11.67	94.64	95.94	7.61	5.36	4.06			
	Conc 0.25-0.5 min	59.78	12.49	5.91	1.36	0.56	25.09	88.71	91.52	16.61	11.29	8.48			
	Conc 0.5-1 min	89.04	18.60	5.80	1.27	0.48	45.11	80.05	85.89	31.00	19.95	14.11			
	Conc 1-2 min	101.22	21.14	6.14	1.27	0.48	67.78	69.64	79.41	47.19	30.36	20.59			
	Conc 2-5 min	81.39	17.00	8.92	1.55	0.77	85.46	57.47	71.12	56.58	42.53	26.88			
	Tails	95.20	19.89	36.00	6.40	5.63									
	Total Feed	478.74	100.00	12.46	2.36	1.58									

Table 11.14 Flotation kinetics of 200-mesh Upper Freeport PA coal with double dosage of collector.

	Sample ID	476-B3-04-W200										
	Dodecane	0.48 lb/T		MIBC		0.26 lb/T						
	pH	Initial 3.30		Final 3.44								
Size	Product	Weight	Yield	Ash	Tot.S	Pyr.S	CMR	Ash	Pyr. S	EI	Ash	Pyr. S
Mesh	Name	%	%	%	%	%	%	Rel. %	Rel. %	%	Rel. %	Rel. %
+400	Conc 0-0.25 min	9.19	9.17	5.24	1.11	0.32	9.71	95.46	97.18	6.90	4.54	2.82
	Conc 0.25-0.5 min	11.04	11.01	4.97	1.09	0.30	21.42	90.28	94.03	15.45	9.72	5.97
	Conc 0.5-1 min	16.76	16.72	4.88	1.07	0.28	39.20	82.56	89.56	28.77	17.44	10.44
	Conc 1-2 min	19.27	19.22	5.50	1.07	0.28	59.52	72.55	84.30	43.82	27.45	15.70
	Conc 2-5 min	19.06	19.01	7.48	1.22	0.45	79.19	59.10	76.13	55.31	40.90	23.87
	Tails	24.92	24.86	25.14	3.93	3.19						
	Feed +400	100.24	28.24	10.57	1.82	1.04						
-400	Conc 0-0.25 min	35.74	14.03	6.00	1.40	0.60	15.01	93.07	94.89	9.90	6.93	5.11
	Conc 0.25-0.5 min	36.81	14.45	5.94	1.37	0.57	30.48	86.00	89.89	20.37	14.00	10.11
	Conc 0.5-1 min	50.49	19.82	5.92	1.38	0.58	51.70	76.33	82.91	34.61	23.67	17.09
	Conc 1-2 min	51.14	20.07	6.62	1.42	0.63	73.04	65.38	75.29	48.33	34.62	24.71
	Conc 2-5 min	38.77	15.22	9.44	1.82	1.03	88.72	53.55	65.76	54.49	46.45	34.24
	Tails	41.80	16.41	39.65	7.42	6.63						
	Feed -400	254.75	71.76	12.14	2.45	1.65						
Overall	Conc 0-0.25 min	44.93	12.66	5.84	1.34	0.54	13.50	93.68	95.36	8.86	6.32	4.64
	Conc 0.25-0.5 min	47.85	13.48	5.72	1.31	0.51	27.89	87.09	90.72	18.61	12.91	8.28
	Conc 0.5-1 min	67.25	18.94	5.66	1.30	0.50	48.13	77.93	84.25	32.38	22.07	15.75
	Conc 1-2 min	70.41	19.83	6.31	1.32	0.53	69.17	67.22	77.12	46.29	32.78	22.88
	Conc 2-5 min	57.83	16.29	8.79	1.62	0.84	86.00	54.98	67.87	53.87	45.02	32.13
	Tails	66.72	18.79	34.23	6.12	5.35						
	Total Feed	354.99	100.00	11.70	2.27	1.48						

Table 11.15 Flotation kinetics of 200-mesh Upper Freeport PA coal with double dosage of frother.

	Sample ID	480-B3-04-W200										
	Dodecane	0.24 lb/T		MIBC		0.52 lb/T						
	pH	Initial 3.41		Final 3.51								
Size	Product	Weight	Yield	Ash	Tot.S	Pyr.S	CMR	Ash	Pyr. S	EI	Ash	Pyr. S
Mesh	Name	%	%	%	%	%	%	Rel. %	Rel. %	%	Rel. %	Rel. %
+400	Conc 0-0.25 min	19.57	20.42	6.22	1.24	0.45	21.40	87.90	91.20	12.60	12.10	8.80
	Conc 0.25-0.5 min	20.13	21.01	6.04	1.20	0.41	43.45	75.82	82.94	26.39	24.18	17.06
	Conc 0.5-1 min	30.68	32.02	7.46	1.26	0.48	76.55	53.07	68.17	44.72	46.93	31.83
	Conc 1-2 min	16.38	17.09	11.80	1.64	0.89	93.40	33.86	53.69	47.09	66.14	46.31
	Conc 2-5 min	5.80	6.05	24.60	3.32	2.61	98.50	19.68	38.68	37.15	80.32	61.34
	Tails	3.27	3.41	60.55	12.71	11.84						
	Feed +400	95.83	27.22	10.50	1.82	1.05						
-400	Conc 0-0.25 min	57.95	22.62	6.61	1.58	0.78	24.08	87.80	89.79	13.86	12.20	10.21
	Conc 0.25-0.5 min	52.74	20.59	6.65	1.53	0.73	45.98	76.84	81.05	27.03	23.36	18.95
	Conc 0.5-1 min	75.70	29.55	7.65	1.70	0.90	77.08	58.20	65.57	42.65	41.80	34.43
	Conc 1-2 min	40.87	15.95	11.19	2.30	1.50	93.23	43.64	51.63	44.85	56.36	48.37
	Conc 2-5 min	13.42	5.24	23.78	4.90	4.09	97.78	33.49	39.18	36.96	66.51	61.82
	Tails	15.51	6.05	67.78	11.86	11.11						
	Feed -400	256.19	72.78	12.26	2.52	1.72						
Overall	Conc 0-0.25 min	77.52	22.02	6.51	1.49	0.69	23.34	87.83	90.06	13.40	12.17	9.94
	Conc 0.25-0.5 min	72.87	20.70	6.48	1.44	0.64	45.28	76.44	81.43	26.71	23.56	19.57
	Conc 0.5-1 min	106.38	30.22	7.60	1.57	0.78	76.93	56.95	66.11	43.04	43.05	35.89
	Conc 1-2 min	57.25	16.26	11.36	2.11	1.33	93.27	41.26	52.08	45.35	58.74	47.92
	Conc 2-5 min	19.22	5.46	24.01	4.42	3.64	97.98	30.13	39.18	37.16	69.87	60.82
	Tails	18.78	5.33	66.50	12.01	11.24						
	Total Feed	352.02	100.00	11.77	2.33	1.53						

Figure 12.1 Combustible material recovery as a function of time for Upper Freeport PA coal ground to minus 100-mesh, minus 200-mesh and minus 325-mesh.

Time	100 MESH CMR %	200 MESH CMR %	325 MESH CMR %
0.5	42.7	24.2	22.5
1	21.7	23.2	20.6
2	13.4	24.8	23.4
3	9.0	8.7	9.0
5	8.3	9.7	5.7
10	2.3	5.6	3.6
20	0.6	1.7	1.6

Figure 12.2 Separation efficiency as a function of time for Upper Freeport PA coal ground to minus 100-mesh, minus 200-mesh and minus 325-mesh.

Time	100 MESH E _{LM}	200 MESH E _{LM}	325 MESH E _{LM}
0.5	30.7	19.8	17.3
1	45.4	38.7	40.0
2	54.1	56.0	56.2
3	59.8	61.3	60.9

Figure 12.3 Effect of frother conditioning time on the flotation rate constant and ultimate recovery of combustible material of Illinois No. 6 coal.

Cond. time mins.	Kinetic constant 1/min.	Ultimate recovery %
0.5	1.128	89.1
1	1.119	89.7
2	0.976	87.4
3	0.775	87.0

Figure 12.4 Effect of frother conditioning time on the flotation rate constant and ultimate recovery of combustible material of Pittsburgh No. 8 coal.

Cond. time mins.	Kinetic Constant 1/min.	Ultimate Recovery %
0.5	0.854	86.2
1	0.813	85.3
2	0.681	86.2
3	0.737	85.3

Figure 12.5 Effect of frother conditioning time on the flotation rate constant and ultimate recovery of combustible material of Upper Freeport PA coal.

Cond. time mins.	Kinetic Constant 1/min.	Ultimate Recovery %
0.5	0.9	86.7
1	0.88	86.4
2	0.75	85.8
3	0.68	84.8

Figure 12.9 Comparison of performance of mechanically agitated cell and flotation column for the rejection of non-pyritic minerals from Pittsburgh No. 8 coal.

1 STAGE CELL		2 STAGE CELL		0.65 LB/T MIBC		0.91 LB/T MIBC	
CMR %	NPAR %	CMR %	NPAR %	CMR %	NPAR %	CMR %	NPAR %
46.5	82.1	54.3	84.9	16.4	96.7	14.1	97.3
49.4	80.4	76.0	73.8	34.2	91.7	18.0	96.1
69.1	70.9	83.6	70.0	45.0	88.9	18.9	95.7
86.2	58.8	92.0	60.4	94.8	59.2	33.1	91.9
88.3	55.7			97.8	58.0	52.3	84.7
94.3	48.3					67.6	81.2
						64.0	80.7
						93.0	63.5

Figure 12.10 Comparison of performances of mechanically agitated cell and flotation column for the rejection of pyrite from Pittsburgh No. 8 coal.

1 STAGE CELL		2 STAGE CELL		0.65 LB/T MIBC		0.91 LB/T MIBC	
CMR %	NPAR %	CMR %	NPAR %	CMR %	NPAR %	CMR %	NPAR %
46.5	90.3	54.3	91.3	16.4	98.8	14.1	98.8
49.4	89.8	76.0	84.7	34.2	96.3	18.0	99.3
69.1	82.7	83.6	79.5	45.0	95.7	18.9	98.6
86.2	74.1	92.0	76.6	94.8	75.6	33.1	95.3
88.3	74.0			97.8	72.2	52.3	95.5
						67.6	91.3
						64.0	91.0
						93.0	73.9

Figure 12.11 Effect of bubble size on the rejection of pyrite from Pittsburgh No. 8 coal.

WASHABILITY		COARSE FRIT		MEDIUM FRIT	
CMR %	PSR %	CMR %	PSR %	CMR %	PSR %
96.4	87.7	99.3	66.9	99.8	59.1
95.1	90.1	85.3	81.7	98.5	61.4
77.1	96.2	82.7	84.5	96.5	55.1
67.7	97.3	74.8	88.6	91.4	61.1
68.4	98.2	71.0	88.1	83.4	80.4
		64.6	89.5	69.3	84.5
		57.1	92.0	58.2	88.6
		53.1	89.9	57.6	87.7
		51.4	91.8	54.7	90.4
		51.2	94.0		

Figure 12.12 Effect of bubble size on the rejection of non-pyritic minerals from Pittsburgh No. 8 coal.

WASHABILITY		COARSE FRIT		MEDIUM FRIT	
CMR %	NPAR %	CMR %	NPAR %	CMR %	NPAR %
96.4	59.4	99.3	55.9	99.8	55.6
95.1	63.0	85.3	71.0	98.5	58.3
77.1	81.2	82.7	72.7	96.5	58.3
67.7	85.4	74.8	74.9	91.4	63.8
68.4	88.3	71.0	77.8	83.4	73.0
		64.6	82.8	69.3	78.6
		57.1	84.3	58.2	83.3
		53.1	83.7	57.6	84.5
		51.4	85.2	54.7	85.7
		51.2	84.5		

Figure 12.13 Effect of wash water on the rejection of pyrite from Pittsburgh No. 8 coal - tests with coarse frit.

NO WASH WATER		5 CC PER MIN. WASH WATER	
CMR 1	PSR 1	CMR 1	PSR 1
99.3	66.9	97.8	72.2
85.3	81.7	95.7	65.9
82.7	84.5	94.1	74.4
74.8	88.6	84.0	83.4
71.0	88.1	82.6	85.4
64.6	89.5	71.3	89.1
57.1	92.0	64.0	91.3
53.1	89.9	56.3	89.3
51.4	91.8	53.8	92.6
51.2	94.0		

Figure 12.14 Effect of wash water on the rejection of pyrite from Pittsburgh No. 8 coal - tests with medium frit.

NO WASH WATER		10 CC PER MIN. WASH WATER		14 CC PER MIN. WASH WATER	
99.3	59.1	95.4	73.2	99.3	66.9
98.5	61.4	94.4	54.5	78.3	83.8
96.5	55.1	90.4	77.9	76.6	85.2
91.4	61.1	67.5	87.8	71.6	86.2
83.4	80.4	67.3	88.1	69.3	75.2
69.3	84.5			63.7	82.3
58.2	88.6			53.4	78.8
57.6	87.7			51.6	84.0
54.7	90.4				

Figure 13.6 Comparison of the particle size distribution of the rod milled research sample (95 percent passing 28 mesh) and Increment 1 of the weathered samples (inert, covered and open) for Illinois No. 6 coal.

Mesh	CUMULATIVE WT. PERCENT PASSING			
	St. Grain	Inert	Covered	Open
270				
200	36.4			
150	42.7	24.3	21.7	24.7
100	52.7	33.2	30.9	33.6
65	66.9	46.1	44.3	46.3
48	80.8	63.1	61.6	63
35	89.4	86.5	25.8	27.2
28	92.8	99.8	99.8	99.9
20	94.5	99.8	99.9	100.0
14	95.3	100.0	100.0	
10	95.9			
8	96.1			

Figure 13.7 Effect of the weathering time on the weight percent of material in different size intervals obtained by sieving the minus 28 mesh material of Illinois No. 6 coal weathered under open mode.

Weathering time, months	WEIGHT PERCENT IN SIZE INTERVAL			
	28 x 35 M	48 x 65 M	65 x 100 M	100 x 150 M
0.5	35.4	17.3	12.2	7.7
1.0	31.3	16.8	11.7	7.5
1.5	27.5	18.5	16.1	11.2
2.0	30.9	18.7	13.6	9.4
2.5	31.9	17.7	13.6	9.2
3.0	29.4	18.4	15.4	10.7
4.0	17.8	21.5	16.1	10.9
5.0	16.9	22.1	16.9	11.9
9.0	24.8	20.3	16.5	11.5

Figure 13.8 Effect of weathering on the size distribution of the 1/4-inch x 28 mesh Illinois No. 6 weathered coal samples (inert and open samples Increment 1, and open samples Increment 7 and 9).

Mesh	CUMULATIVE WEIGHT PERCENT PASSING			
	Incr.1 (Inert)	Incr.1 (Open)	Incr.7 (Open)	Incr.9 (Open)
150	0.18	0.82	1.24	1.58
100	0.22	0.73	1.46	1.83
65	0.28	0.87	1.72	2.13
48	0.36	1.05	2.02	2.6
35	0.48	1.35	2.41	3.6
28	1.29	2.8	5.39	7.7
20	9.00	11.0	13.5	17.1
14	18.4	20.6	22.8	26.9
10	33.3	35.6	37.3	42.1
8	48.5	50.8	52.0	56.4
7		59.7	61.4	64.1
6	78.6	69.8	70.6	73.2
5		81.6	82.3	82.7
4			92.5	91.7
3.5	88.8	91.5		97.8

Figure 13.9 Comparison of the particle size distribution of rod mill ground research samples (95 percent passing 28 mesh) and Increment 8 of the weathered samples (inert, covered and open) for Pittsburgh No. 8 coal.

Mesh	CUMULATIVE WT. PERCENT PASSING			
	St. Grin.	Inert	Covered	Open
270	30.9	33.8	31.9	34.8
200	9.6	41.0	39.5	42.2
150	50.2	51.2	50.3	52.2
100	64.5	63.2	63.1	64.3
65	82.2	79.6	79.7	79.8
48	95.4	99.7	99.8	99.8
35	99.5	100.0	100.0	100.0
28	99.9			
20	99.9			
14	100.0			
10				
8				

Figure 13.10 Effect of weathering on the size distribution of 1/4-inch x 28 mesh weathered samples (inert and open samples of Increment 1, and open samples of Increments 8 and 9) for Pittsburgh No. 8 coal.

Mesh	CUMULATIVE WT. PERCENT PASSING			
	Incr.1 (Open)	Incr.1 (Open)	Incr.8 (Open)	Incr.9 (Open)
150				
100				
65				
48				
35	0.2	0.12		
28	0.05	0.57	0.04	1.07
20	8.2	6.7	6.6	7.7
14	16.9	14.5	14.6	15.6
10	31.2	27.7	28.3	28.5
8	45.0	41.9	42.4	41.3
7	53.3	50.2	50.6	49.2
6	63.2	60.7	61.1	59.8
5	74.4	73.0	73.4	72.1
4	85.2	84.7	85.3	84.6
3.5			95.2	96.0

Figure 13.11 Comparison of the particle size distribution of rod mill ground research sample (95 percent passing 28 mesh) and Increment 8 of the weathered samples (inert, covered and open) for Upper Freeport PA coal.

Mesh	CUMULATIVE WT. PERCENT PASSING			
	St. Grain.	Inert	Covered	Open
270				
200				
150	32.5	23.5	21.5	20.4
100	44.3	31.0	29.1	27.1
65	57.8	41.7	40.4	36.7
48	71.7	55.3	54.1	49.5
45	87.8	77.9	73.9	68.9
28	97.3	99.3	98.5	98.0
20	99.6	100.0	100.0	100.0
14	99.9			
10	100.0			
8				

Figure 13.12 Film flotation partition curves of as-received samples of Increment 3 for Illinois No. 6 weathered under inert and open conditions.

LV mN/m	Inert	Covered	Open
36.5	0	0	0
40.5	1.8	1.3	0
45	4.9	5.2	1.5
48	9.8	9	4.1
51	18.9	16.3	8.5
54.5	20.2	20.1	13.3
58.8	28.3	26.3	18.1
64.5	55.4	36	26.4
72.7	59.2	58.9	46.7

Figure 13.13 Film flotation partition curves of deslimed samples of Increment 3 for Illinois No. 6 coal samples weathered under inert and open conditions.

LV mN/m	Inert	Covered	Open
33.0	0.0	0.0	0.0
36.5	2.9	1.1	0.7
40.5	13.5	3.7	4.4
45.0	22.4	12.0	11.2
48.0	30.1	17.5	19.9
51.0	36.6	24.3	24.4
54.5	47.8	30.6	30.0
58.8	59.1	37.9	36.5
64.5	65.1	51.6	45.9
72.7	71.8	61.2	51.2

Figure 13.14 Film flotation partition curves of deslimed Illinois No. 6 coal coal samples weatherd under open mode for 0.5, 1.5 and 5 months.

	OPEN TIME, MONTHS		
	0.5	1.5	4
0.0	0.0	0.0	0.0
1.3	0.7	1.5	
4.3	4.4	3.6	
12.9	11.2	9.1	
14.6	19.9	10.7	
19.7	24.4	14.0	
25.0	30.0	18.0	
34.8	36.5	27.6	
48.0	45.9	34.5	
56.3	51.2	45.1	

Figure 13.15 Film flotation partition curves of deslimed Pittsburgh No. 8 coal weathered under open for 0.5, 2 and 5 months.

LV mN/m	OPEN TIME, MONTHS		
	0.5	2.0	5.0
33.0	3.2	2.4	0.1
36.5	8.2	9.0	2.1
40.5	14.2	12.9	7.6
45.0	21.9	20.7	16.1
48.0	29.6	32.3	14.9
51.0	37.7	38.9	21.3
54.5	56.2	55.0	30.8
58.8	65.5	65.5	44.0
64.5	80.0	75.4	59.2
72.7	85.6	83.7	70.9

Figure 13.16 Film flotation partition curves of deslimed Upper Freeport PA coal weathered under open mode for 0.5, 2.0 and 5 months.

LV mN/m	OPEN TIME, MONTHS		
	0.5	2.0	5.0
33.0	0.0	1.8	0.2
36.5	1.4	5.4	4.7
40.7	12.3	15.7	15.1
45.0	31.8	36.2	24.4
48.0	56.7	51.4	38.3
51.0	68.7	69.7	51.2
54.5	84.6	80.5	66.7
58.8	89.8	91.2	75.1
64.5	96.3	94.6	85.7
72.7	98.6	97.8	90.0

Figure 13.17 Induction time curves indicating the percentage of fruitful contacts as a function of contact time in milliseconds for 100 x 150M Illinois No. 6 weathered samples stored under open mode. Two different sample preparation procedures were used.

Contact time milliseconds	FRUITFUL CONTACTS, PERCENT	
	Not deslimed	Deslimed
100	15	36
150	--	42
175	--	40
200	40	56
250	45	56
275	50	64
300	55	60
300	50	70
375	60	70
375	60	--
450	56	76
460	70	86

Figure 13.18 Induction time curves indicating the percentage of fruitful contacts as a function of contact time in milliseconds for 100 x 150M Illinois No. 6 weathered samples Increment 1 stored under inert, covered and open modes.

<u>Contact time</u> <u>milliseconds</u>	<u>FRUITFUL CONTACTS, PERCENT</u>		
	<u>Inert</u>	<u>Covered</u>	<u>Open</u>
4.4	40	--	--
5.0	20	--	--
10	40	15	--
20	50	40	30
25	50	--	--
40	55	45	35
50	55	--	--
60	55	30	50
70	--	--	55
75	70	--	--
80	60	60	50
100	60	70	60
125	60	--	--
150	70	--	60
160	--	--	65
200	75	--	--
250	70	--	70

Figure 13.19 Induction time curve indicating the percentage of fruitful contacts as a function of contact time in milliseconds for 100 x 150M Illinois No. 6 weathered sample of Increment 13 stored under open mode.

<u>Contact time</u> <u>milliseconds</u>	<u>Fruitful contacts</u> <u>percent</u>
500	20
700	17
900	24
1000	25
1100	30
1200	25
1300	33
1400	33
1500	35
1600	40
1700	40
1750	48
1800	45
1900	41

Figure 13.20 Effect of weathering time on the flotation yield for Illinois No. 6 coal stored under inert, covered and open modes.

<u>Weathering</u> <u>Time, months</u>	<u>FLotation YIELD 1/4 INCH X 28 M</u>			
	<u>Inert</u>	<u>Open</u>	<u>Inert</u>	<u>Open</u>
0.5	68.6	56.7	82.8	83.3
1.0	66.3	53.2	--	--
1.5	63.8	53.6	--	--
2.0	--	50.1	--	--
2.5	53.5	42.9	--	--
3.0	--	42.8	--	--
4.0	63.5	36.4	83.0	--
5.0	--	--	--	--
6.0	48.3	29.1	--	81.4
9.0	45.7	30.0	81.5	70.4
11.0	36.3	23.6	76.7	71.7
13.0	46.8	25.4	--	--

Figure 13.21 Effect of weathering time on the flotation yield for Pittsburgh No. 8 coal stored under inert and open modes.

Weathering Time, mon	FLOTATION YIELD, PERCENT			
	28 M X 0		1/4 inch x 28 M	
	Inert	Open	Inert	Open
0.5	62.1	55.3	93.6	94.1
1.0	63.1	47.0	--	--
1.5	--	--	--	--
2.0	68.5	55.5	--	--
2.5	--	--	--	--
3.0	64.8	49.1	--	--
4.0	--	--	--	--
5.0	59.6	51.8	93.8	93.7
6.0	59.5	26.9	--	--
7.0	41.9	12.8	93.5	91.0
9.0	45.3	10.0	92.2	68.4
11.0	56.8	19.0	93.2	83.7
13.0	53.1	21.7	--	--

Figure 13.22 Effect of weathering time on the flotation yield for Upper Freeport PA coal stored under inert and open modes.

Weathering Time, months	FLOTATION YIELD, PERCENT			
	28 M x 0		1/4 inch x 28 M	
	Inert	Open	Inert	Open
0.5	89.1	84.7	92.5	91.7
2.0	87.0	83.3	--	--
5.0	88.2	70.2	91.4	87.0
7.0	78.3	39.8	--	--
9.0	76.8	10.5	88.9	84.6
11.0	69.2	19.5	88.8	86.2
13.0	67.7	25.8	--	--

Figure 13.23 Comparison of the effect of weathering on the combustible matter recovery for Illinois No. 6, Pittsburgh No. 8 and Upper Freeport PA coals weathered under open condition.

Weathering Time, months	COMBUSTIBLE MATTER RECOVERY, PERCENT		
	Illinois No. 6	Pittsburgh No. 8	Upper Freeport PA
0.5	70.4	63.0	87.7
1.0	65.4	52.4	--
1.5	69.7	--	--
2.0	62.0	61.4	83.6
2.5	53.6	--	--
3.0	52.2	54.5	--
4.0	45.7	57.4	--
5.0	--	55.9	74.5
6.0	35.0	28.8	--
7.0	--	12.4	41.6
8.0	--	--	--
9.0	33.8	9.5	10.7
11.0	29.1	20.7	20.5
13.0	31.2	23.5	27.1

Figure 13.24 Induction time, standard flotation and film flotation yields for Illinois No. 8 weathered samples stored under open mode as a function of weathering time.

<u>Weathering time</u> <u>months</u>	<u>Induction time</u> <u>milliseconds</u>	<u>Std. flot. yield</u> <u>percent</u>	<u>Film flot. yield</u> <u>percent</u>
0.5	60	56.7	56.3
1.5	200	53.6	51.2
4.0	250	36.4	45.1
6.0	250	29.1	--
9.0	1000	--	22.3
11.0	1520	23.6	12.0
13.0	1750	25.4	17.5

Figure 13.25 Induction time, standard flotation and film flotation yields for Pittsburgh No. 8 weathered samples stored under open mode as a function of weathering time.

<u>Weathering time</u> <u>months</u>	<u>Induction time</u> <u>milliseconds</u>	<u>Std. flot. yield</u> <u>percent</u>	<u>Film flot. yield</u> <u>percent</u>
0.5	40	55.3	85.6
2.0	40	55.5	83.7
5.0	40	51.8	70.9
7.0	100	12.8	--
9.0	375	10.0	89.5
11.0	500	19.0	92.8
13.0	200	21.7	91.6

Figure 13.26 Induction time, standard flotation and film flotation yields for Upper Freeport PA weathered samples stored under open mode as a function of weathering time.

<u>Weathering time</u> <u>months</u>	<u>Induction time</u> <u>milliseconds</u>	<u>Std. flot. yield</u> <u>percent</u>	<u>Film flot. yield</u> <u>percent</u>
0.5	7.5	84.7	98.6
2.0	7.5	83.3	97.8
5.0	7.8	70.2	90.0
7.0	8.0	39.8	--
9.0	13.5	10.5	94.5
11.0	14.1	19.5	95.6
13.0	10.0	25.8	93.3

Figures 14.2 & 14.3 Release analysis and tree analysis results showing ash rejection and pyritic sulfur rejection as a function of C_{PR} for Illinois No. 6 coal.

WASHABILITY			STANDARD		
<u>1-Yield</u>	<u>PSR</u>	<u>Ash</u>	<u>1-Yield</u>	<u>PSR</u>	<u>Ash</u>
0.0	--	--	0.0	--	--
15.0	91.0	61.4	25.0	57.7	57.7
18.8	92.5	67.6	30.7	61.8	61.8
34.9	95.9	80.5	52.1	74.4	74.2
42.0	96.7	85.7	68.0	82.8	82.6
65.5	98.3	93.7	100.0	100.0	100.0

RELEASE ANALYSIS B1-03			TREE ANALYSIS B1-03		
<u>1-Yield</u>	<u>PSR</u>	<u>Ash</u>	<u>1-Yield</u>	<u>PSR</u>	<u>Ash</u>
0.0	--	--	0.0	--	--
12.3	58.3	49.0	5.8	29.8	30.1
14.9	67.6	58.1	6.2	31.2	31.9
18.6	73.9	64.8	9.4	37.6	39.0
22.0	78.2	69.3	11.2	41.2	43.4
29.4	81.4	73.6	17.8	53.8	55.8
50.2	89.2	84.1	17.9	54.0	56.0
67.6	93.5	90.3	18.3	54.6	56.7
90.2	98.7	97.4	19.0	55.4	57.6
100.0	100.0	100.0	19.4	55.7	57.9
			46.8	74.7	75.8
			51.2	80.5	81.6
			57.1	81.0	82.1
			70.0	88.4	89.6
			73.5	90.1	91.3
			94.6	98.4	98.3
			100.0	100.0	100.0

TREE ANALYSIS B1-02		
<u>1-Yield</u>	<u>PSR</u>	<u>Ash</u>
0.0	--	--
8.9	38.6	38.9
11.4	42.0	42.9
20.0	51.9	53.0
28.2	60.6	61.8
38.2	70.3	71.8
38.6	70.7	72.2
40.0	71.5	73.0
45.1	74.2	75.4
59.5	81.5	82.0
74.5	88.9	89.4
88.6	95.6	96.3
89.3	95.9	96.6
96.5	98.8	98.9
98.5	99.5	99.6
99.3	99.8	99.8
100.0	100.0	100.0

Figures 14.4 & 14.5 Release analysis and tree analysis results showing ash rejection and pyritic sulfur rejection as a function of 100-yield for Pittsburgh No. 8 coal.

WASHABILITY			1-STEP FLOAT		
<u>1-Yield</u>	<u>PSR</u>	<u>Ash</u>	<u>1-yield</u>	<u>PSR</u>	<u>Ash</u>
0.0	0	0.0	0.0	0.0	45.3
11.5	88	65.7	8.4	43.8	59.2
13.0	90	69.3	20.0	58.6	73.1
31.0	96	84.1	38.0	73.4	96.4
39.0	97	87.7	93.0	95.5	100.0
46.0	98	90.5	100.0	100.0	
0.0	100	100.0			
RELEASE ANALYSIS B2-03			TREE ANALYSIS B2-04		
<u>1-yield</u>	<u>PSR</u>	<u>Ash</u>	<u>1-yield</u>	<u>PSR</u>	<u>Ash</u>
7.6	41.4	48.2	0.5	2.4	2.8
12.9	57.7	61.0	12.2	49.3	48.4
18.4	68.1	69.4	18.8	56.5	55.5
39.8	83.4	82.6	32.9	71.4	69.9
54.6	88.8	88.0	41.8	80.0	78.7
70.2	93.7	92.9	43.7	81.7	80.3
84.2	97.0	96.5	45.1	82.8	81.5
97.0	99.6	99.5	46.2	83.5	82.3
			50.1	88.7	87.7
			68.2	91.6	90.8
			81.5	96.0	95.5
			90.0	98.1	97.9
			91.6	98.5	98.4
			93.5	98.8	98.8
			99.2	99.9	99.9

Figures 14.6 and 14.7 Release analysis and tree analysis results showing ash rejection and pyritic sulfur rejection as a function of 100-yield for Upper Freeport FA.

WASHABILITY			RELEASE ANALYSIS B3-03		
<u>1-Yield</u>	<u>PSR</u>	<u>Ash</u>	<u>1-yield</u>	<u>PSR</u>	<u>Ash</u>
0.0	--	--	0.0	--	--
12.0	91.9	60.7	6.7	41.8	34.8
19.0	94.3	74.6	10.3	59.6	48.2
28.8	97.0	84.8	13.0	70.1	56.3
29.5	97.0	85.2	25.1	82.2	71.0
45.1	98.1	91.4	42.4	89.6	82.0
100.0	100.0	100.0	63.1	94.7	90.4
			83.3	98.3	96.4
			100.0	100.0	100.0
TREE ANALYSIS B3-04					
<u>1-yield</u>	<u>PSR</u>	<u>Ash</u>			
0.0	--	--			
10.9	56.7	44.5			
12.7	60.6	48.4			
15.7	65.5	52.6			
26.6	78.7	67.4			
34.6	88.1	77.8			
35.9	89.0	79.0			
38.1	90.0	80.4			
40.9	91.0	82.1			
44.8	92.3	84.1			
51.4	94.1	87.4			
59.7	96.5	91.4			
62.4	96.9	92.2			
67.3	97.6	93.5			
76.9	98.5	95.5			
86.6	99.3	97.9			
100.0	100.0	100.0			

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