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COMBUSTION AND FUEL CHARACTERIZATION
OF COAL-WATER FUELS

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SELECTION AND PROCUREMENT OF CANDIDATE
COAL-WATER FUELS WITH COMMERCIAL POTENTIAL

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INTRODUCTION

Coal-water fuel mixtures (CWFs) which typically contain 70% pulverized coal, 30% water, and less than 1% chemical additives, have the potential of replacing fuel oil in commercial furnaces. Use of these coal-based fuels could lessen U. S. dependency on imported oil and reduce power generating costs.

Commercial development of CWF has been impeded by technical and economic uncertainties. The key technical question concerns the impact an ash-bearing CWF would have on furnaces originally designed for ash-free or very low ash fuels. Economic feasibility - in terms of boiler derating, equipment modification, and retrofit costs - will largely depend upon the degree to which CWF affects boiler performance. Resolution of these unanswered technical and economic questions is an essential prerequisite to the commercial development of CWFs as alternatives to fuel oil. In response to this need, the Pittsburgh Energy Technology Center (PETC) of the Department of Energy initiated a comprehensive effort in 1982 to develop the necessary performance and cost data and to assess the commercial viability of CWFs as applied to representative utility and industrial units.

Combustion Engineering, Inc. (C-E) was contracted to conduct the project. The effort comprised six tasks beginning with coal resource evaluation and culminating in the assessment of the technical and economic consequences of switching representative commercial units from oil to state-of-the-art CWF firing. Extensive bench, pilot and commercial-scale tests were performed by C-E to develop necessary CWF combustion and fireside performance data for the subsequent boiler performance analyses and retrofit cost estimates. Gulf Research and Development Company, Energy International, Inc., and the Adelphi Center for Energy Studies provided essential sub-contract support in various aspects of source coal characterization and selection, coal preparation and procurement, and the determination of detailed fuel properties. CWFs were prepared for this project by Advanced Fuels Technology, Atlantic Research, CoaLiquid, Ergon, and OXCE Fuel Company.

This report (Volume 2) provides a review of the fuel selection and procurement activities. Other results of the project are provided in the following volumes:

Volume 1 - Tasks 1 through 6	Final Summary Report
Volume 3 - Task 2	Bench-Scale Characterization of Chemical, Physical and Combustion Properties of Coal-Water Fuels
Volume 4 - Task 4	Commercial-Scale Atomizer and Burner Evaluation
Volume 5 - Task 5	Pilot-Scale Ash Deposition and Performance Testing of Coal-Water Fuels
Volume 6 - Task 6	Commercial Application and Economics of Coal-Water Fuels

RESEARCH APPROACH

The project was organized to respond directly to the probable needs of CWF users. Five major tasks were defined to establish the technical data base necessary for boiler performance analyses. Established boiler performance models then utilized these data, in a sixth task, to establish specific load-limiting factors and to determine consequent equipment modification and retrofit costs. Finally, economic feasibilities in terms of payback periods were estimated for several boiler types and CWF properties based upon fuel price differentials.

To ensure commercial relevance, criteria consistent with the expected market were defined for each task. Coal resources, beneficiation processes, and coal-water fuel producers were evaluated and selected on the basis of availability, suitability (as measured by test criteria), and other commercial considerations. Engineering evaluations of atomization, combustion, and fireside performance of the selected CWFs were based upon test equipment and procedures used regularly for coal-fired and oil-fired boiler performance tests. Overall program logic is reflected in the task responsibilities illustrated in Figure 1.

Selection of Test Fuels

The fuels tested were CWFs having formulations representative of those that are expected to become available commercially. The selected CWFs were made from five coal sources, two levels of beneficiation, and five commercial suppliers.

Based upon published data, twenty coals from the Eastern United States were initially selected and subjected to first level screening tests. Key criteria included commercial significance, beneficiation potential, ash content and quality, sulfur content, volatile matter content, and char reactivity. Since coal beneficiation is expected to play a major role in the commercial use of CWFs, the selection process considered the anticipated

FIGURE 1 OVERVIEW OF PROJECT ACTIVITIES

characteristics of the coal after beneficiation. As part of these screening tests, gravity separation (14 mesh at 1.3 specific gravity) and subsequent analysis of the two density fractions plus the raw coal were performed.

Based upon results of the first level screening tests, eight coals were selected for second level screening tests. These tests consisted of detailed "washability" analyses and detailed fuel analyses.

The results of the second level screening tests were used to select source coals for beneficiation and for preparation of CWFs for large-scale testing. Cedar Grove, Splash Dam, and Upper Freeport CWFs were tested at bench scale, pilot scale and full scale. A Lower Kittanning coal and an Alma seam coal were used to make CWFs for bench-scale and pilot-scale tests. These selected coals provided a range in fuel characteristics including varying volatile matter content and ash quality.

Program Objectives

The purpose of this project is to evaluate coal-water fuels (CWFs) as replacement fuels in oil-burning facilities. This involves not only a detailed investigation of the physical and combustion properties of the CWFs, but also an evaluation of the chemical, physical, and combustion properties of the source coal and their impact on CWF properties.

The initial phase of this research, therefore, involved an evaluation of the chemical, physical, and combustion properties of the source coals. Twenty coals were selected for the initial screening washability studies for the CWF program. After completion of the preliminary studies and an evaluation of test data, eight coals were selected for further detailed washability studies. These detailed studies were completed and the washability data evaluated.

Feed coals for the formulation of test CWFs were recommended. Each coal was to be beneficiated to two levels to evaluate the influence of ash on manufacturing and combustion of the CWFs. The levels of beneficiation were defined as follows:

1. 7% ash on a dry basis, $\pm$ 10%
2. 2% ash on a dry basis, $\pm$ 20%

Candidate Coal Selection

Table I is an excerpt from an American Electric Power report listing typical boiler fuel-quality characteristics for steam coal. In general, these parameters were used as "first-pass" guidelines for selecting sources of coal for CWFs. It should also be mentioned that only coals from major bituminous resources were considered for selection. The limits presented in Table I reflect the environmental emissions standards, combustion characteristics, and boiler fouling potential for coals normally associated with steam use. They may not, however, adequately reflect the desired combustion characteristics for coals to be used as CWF fuels.

The moisture added to the coal during CWF preparation effectively reduces the heating value of the fuel. This relationship between calorific value of the coal and the resulting calorific value of the CWF is illustrated graphically in Figure 2. Fuel oil typically has a calorific value of 19,000 Btu/lb with a specific gravity of 0.9. Obviously, this heating value cannot be matched with a CWF. However, a favorable comparison can be made with a minimum calorific value for steam coal given as 11,160 Btu/lb. Coals with heating values greater than 13,500 Btu/lb are preferred, when considering CWFs loaded up to 70% solids.

In addition to having adequate heating values, the candidate CWF coals must have good combustion properties. The ratio of fixed carbon to volatile matter, or fuel ratio, is a good indicator of combustibility. For adequate combustion performance, the fuel ratio of the coal should be less than 2.2.

The selection criteria used in identifying the twenty candidate coals for this study are summarized in Table II. These limits provide guidelines reflective of the environmental emissions standards, combustion characteristics, and boiler fouling potential for coals to be used as CWFs.

TABLE I

TYPICAL BOILER FUEL QUALITY CHARACTERISTICS

<u>Characteristic</u>	<u>Desirable Value for Electric Utility</u>
1. Total Moisture	10% Max.
2. Proximate Analysis Fuel Ratio FC/VM	2.2 Max.
3. Ash	15%
4. Total Sulfur (Air Dry Basis)	1.0% Max.
5. Calorific Value (Air Dry Basis)	11,160 Btu/lb Min.
6. Nitrogen (Dry Basis) Ultimate Analysis	1.7 Max., less than 1.6 Desirable
7. Grindability (Hardgrove Index)	45 Min.
8. Size Consistency	40 MM x 0 100% 2 MM x 0 30%
9. Ash Deformation Temperature	1,150°C Min.
10. Slagging Potential	Low

FIGURE 2 CWF FUEL HEATING VALUE

TABLE II
CWF FEED COAL PROPERTIES (BENEFICIATED)

Sulfur	< 1.2 lb SO ₂ /10 ⁶ Btu (0.8% sulfur @ 13,300 Btu/lb)
Ash	< 2%
Volatile Matter	> 29%
Moisture	< 7%
Heating Value	> 13,500 Btu/lb
Fuel Ratio (FC/VM)	< 2.2
TGA	< 7 minutes for 99% burnout
Flammability Index	< 1000°F
Ash Properties	
Softening Temperature	>2300°F (red, atm)
Fluid Temp,	>2500°F (red. atm)
Free Quartz (% of ash)	25% Maximum
Sodium Content (% of ash)	2% Maximum
Slurryability	Vendor approval

The coal properties listed in Table II were not used as absolute specifications for which to eliminate coals. Rather, the degree of conformity of coals to the Table II properties were weighed together with other selection guidelines.

Selection Criteria: Eight Coals for Detailed Studies

The criteria used for selecting the eight coals for detailed washability studies are relatively straightforward. They are discussed in descending order of importance as follows:

1. Ease of separation. This is related to the specific gravity of separation required to produce 2% ash coal. The most significant indicators are the ash contents and the recovery. If the ash content of the 1.3 specific gravity fraction is less than 2.4% and the recovery high (50% or greater), it is suspected that the Level 4 beneficiation can be accomplished.
2. Estimated recovery of the 2% ash fraction. Generally, the larger this value, the greater the yields and the easier the separation. However, differences in ash levels for the same gravity fractions can significantly alter the ease of separation among coals of similar estimated recoveries.
3. Heating value (moisture and ash free). It can generally be considered, in the case of CWFs, that a higher heating value coal should produce a higher value product for the same level of processing. Of course, in the final analysis, the CWF user/supplier must weigh the economic benefits of acquiring higher quality coals versus sacrificing CWF quality for lower coal costs.

The criteria listed above were considered to be the most important factors for selection of the eight coals. All other criteria were considered to be of lesser importance and were applied in the final selection of the five coals for preparation into slurries.

Selection Criteria: Four CWF Feed Coals

The primary objective of the coal screening and selection phase of this research was to identify four coals that were viable candidates for use in CWFs and that can be successfully beneficiated to 7% and 2% ash. It is expressed or implied in this statement that a number of coal characteristics or properties be concurrently evaluated in order to identify "viable candidates." These include: washability characteristics, combustion properties and ash fouling potential, and characteristics effecting CWF manufacturing. Each of these is briefly discussed in the following paragraphs.

From a beneficiation standpoint, the coals must yield reasonable quantities of two percent ash product, preferably at coarser gradations (plus 28 mesh). Generally, coals which require grinding to a fine particle size for ash liberation lend themselves to more expensive, less efficient, and lower capacity physical cleaning methods. Based on the results of the detailed washability studies and proposals submitted by six candidate beneficiators, a 3/8 inch by 28 mesh size range was selected as being the most workable and cost effective choice for this project. Coals which yield the highest recoveries of two percent product for this size range were considered CWF candidate coals.

Another parameter given consideration in the selection process was the coal's combustion characteristics. As previously mentioned, the fixed carbon-to-volatile matter ratio provides a general indication of a coal's combustion character. Since this ratio, along with other standard ASTM analyses, provides only an indirect indication of combustion properties, two in-house Combustion Engineering (C-E) tests (Flammability and TGA) were conducted which provide more direct measurements. The relative values obtained from these tests were compared with those from C-E's data base to indicate a coal's flame stability, turndown characteristics, and fuel reactivity.

A third criterion used in selecting the CWF candidate coals was the slurry vendors' analysis of the coals from a manufacturing standpoint. At the request of one CWF manufacturer selected for this project, the following tests were conducted on each of the coals considered prime CWF candidates:

1. Ultimate Analysis,
2. Proximate Analysis,
3. Hardgrove Grindability,
4. Equilibrium Moisture, and
6. Ash Composition.

The exact nature of the analysis of these data is not known and is considered, by the vendor, to be proprietary. In addition, test CWF batches were prepared and evaluated by the vendors in order to further define the effect of the feed coal on CWF manufacturing.

It is readily apparent from the previous discussion that a purely analytical approach to selecting the candidate coals for this program could not be used. In some cases it is necessary to resort to more subjective and/or empirical methods in order to identify coals that are "viable candidates" for use in CWFs. Each of these criteria will be discussed in more detail later in this report with reference to particular coals.

DESCRIPTION OF COAL SELECTION

Procurement of Candidate Coals

Run-of-mine (ROM) samples were obtained from each of the 20 coal seams listed in Table III. Initially, each mine was contacted to confirm their commitment to participate in the project and to establish a date for sample collection. None of the mines visited were operating at full capacity and, in some cases, special arrangements were necessary to obtain the small sample required for this project. Excellent cooperation was afforded by each of the participating companies.

Sampling Procedure

The procedure for securing samples of each of the 20 candidate coals involves selecting a sample quantity which will satisfy two basic requirements: (1) an adequate amount is obtained, such that representative sample is assured; and (2) a sufficient amount of coal is obtained to allow both the screening and detailed washability studies to be conducted without resampling.

Guidelines for selecting representative sample amounts were based on the maximum nominal particle size of the coal. This relationship between particle size and minimum sample weight is illustrated in Figure 3. A maximum nominal particle size of six inches was anticipated for mines employing surface mining or conventional underground mining techniques. Utilizing the curves from Figure 3 as a guide, an eight-ton sample amount was considered adequate and was used as the requirement for all samples. Although this amount far exceeds that which was required to conduct the washability testing, it was necessary in order to ensure that a representative sample was obtained.

Table III

COAL SAMPLE ACQUISITION DATA

Coal Seam	Company/Mine	Mine Address	County	Mine Type	Sample Type	Date of Collection
1. Black Creek	Central Mining Co. Black Pond No. 3	207 Hwy. 195 Jasper, AL 35501	Winston	S	A	01/20/83
2. Mary Lee	Alabama By-Products Co. Mary Lee No. 1	P.O. Box 218 Goodsprings, AL 35560	Walker	D	A	01/20/83
3. Lower Elkhorn	Republic Steel-Coal Div. Republic	Elkhorn City, KY 41522	Pike	D	B	01/17/83
4. Lower Kittanning	Armco Coal Co. Mine No. 25	Montcoal, W. VA	Raleigh	S	A	01/18/83
5. Cedar Grove	Chafin Coal Co. Golden Brook	P.O. Box 338 Logan, W. VA 25601	Logan	D	B	02/24/83
6. West KY No. 11	Peabody Coal Co. River Queen	P.O. Box 289 Greenville, KY 42345	Muhlenberg	S	C	02/10/83
7. West KY No. 9	Brown Badgett, Inc. Busick	P.O. Drawer 617 Central City, KY 42330	Muhlenberg	D	B	02/10/83
8. Illinois No. 5	AMAX Coal Co. Leahy	P.O. Box 165 Campbell Hill, IL 62916	Perry	S	C	02/08/83
9. Illinois No. 6	AMAX Coal Co. Leahy	P.O. Box 165 Campbell Hill, IL 62916	Perry	S	C	02/08/83
10. Castle Gate D	Price River Coal Co. Mine No. 5	P.O. Box 629 Helper, UT 84526	Carbon	D	A	02/17/83
11. Splash Dam	Black Watch Coal Co. Bounty Mine No. 1	Rt. 1, Box 220 Grundy, VA 24614	Buchanan	D	B	02/22/83
12. Jaw Bone	Pittston Coal Co. McClure No. 1	McClure, VA	Buchanan	D	A	02/21/83
13. Hazard No. 4	Shamrock Coal Co. Greasy Creek	Big Laurel, KY P.O. Box 175	Dickenson Harlan	D	B	02/23/83
14. Elkhorn No. 3	Southeast Coal Co. Polly	Whitesburg, KY 41858	Letcher	D	A	02/23/83
15. No. 2 Gas	Armco Coal Co. Mine No. 7	Montcoal, W. VA	Raleigh	D	B	01/18/83
16. Pocohontas No. 3	Island Creek Coal Co. VA Pocohontas No. 1	Grundy, VA	McDowell Buchanan	D	B	02/22/83
17. Upper Freeport	BCNR Mining Corp. Newfield	Verona, PA	Allegheny	D	A	02/02/83
18. Pittsburgh No. 8	J&L Steel Co. Nemacolin	Carmichael, PA	Greene	D	A	02/03/83
19. Pittsburgh No. 8	Consolidation Coal Co. Franklin No. 125	Cadiz, OH	Harrison	S	A	02/03/83
20. Waynesburg No. 2	Delta Mining, Inc. Koontz Strip	Klondyke, MD	Garrett	S	A	02/17/83

FIGURE 3 SIZE-WEIGHT RELATIONSHIP FOR COAL SAMPLES

The eight-ton samples were obtained from each mine using bulk sampling bags containing approximately one ton each. The bags were held open and loaded using a front-end loader. The samples were secured from three general locations at a given mine, depending on the operating constraints imposed by the particular mine. These sample types are identified as follows:

- A. The sample as obtained from the ROM stockpile at the mine preparation facility.
- B. In the case of an underground mine producing from multiple seams, samples were obtained from the ROM conveyor of the desired coal seam.
- C. In the case of a surface mine producing from multiple seams, samples were obtained by diverting a haul truck obtaining coal from the desired seam to a remote location of the stockpile area. The samples were then loaded from this dumping area in order to minimize sample contamination.

Table III contains pertinent mine and sample information regarding each of the samples acquired. Mines are identified as "S" (surface) or "D" (deep) with the sample type identified as discussed above.

The 20, eight-ton coal samples brought back to GR&DC were split into smaller samples for testing and possible future use. A rigorous splitting procedure was used to ensure that all fractions would be representative of the parent coals.

Small representative quantities of all of the 20 coals were crushed to 14 x 0 mesh. A sink/float separation at a 1.3 specific gravity was performed on each coal using "Certigrav" as the heavy liquid. The fractions were dried and subjected to the analyses listed in Table IV.

Table IV

ANALYSES SCHEDULE (FIRST LEVEL WASHABILITY)

<u>Float</u>	<u>Sink</u>	<u>Test</u>
X		Ash Composition
X	X	% Ash
X	X	Btu
X	X	% Sulfur
X		Sulfur Forms
X		Acetic Acid Sol. Alkali
X	X	% Recovery
X		% Volatile Matter
X		Free Quartz
X		Ash Fusibility Temp. in Red. Atm.

Preliminary Screening Tests

Raw Coal Data

As mentioned previously, initial review of the properties of the 20 selected coals had been carried out using information supplied by the respective coal companies or obtained through the open literature. After selection and procurement of the 20 selected coals (eight-ton lots), each coal was analyzed again to ensure against large discrepancies in expected coal properties and actual coal properties. The results of this analysis indicated acceptable agreement between the data sets. The analytical results for the raw coal are presented in Table V.

As can be seen from Table V, the selected group of preliminary coals covers a broad range of characteristics ensuring a reasonable representation of the available bituminous coal supply. The majority of coals listed in the table are normally cleaned before sale. However, the run-of-mine analyses show a wide range (approximately 40 percent to 5 percent) of ash contents. The volatile matter (dry) shows a similarly large variation ranging from 41 to 18 percent.

Based on the results shown in Table V, most of the coals meet the 'requirements' presented in Table I (Typical Boiler Fuel Quality...). The Pocahontas No. 3 and Illinois No. 5 coals have FC/VM Values higher than 2.2. Several of the coals have a high sulfur content. Most noticeable among these are the Western Kentucky and Illinois coals.

Test Results

Representative samples of the run-of-mine coals were crushed to minus 14 mesh and subjected to separation at a specific gravity of 1.3. The 1.3 float fractions were subjected to the various tests described in Table IV. The results of these tests are presented in Table VI, including the proximate and sulfur forms analyses for the 1.3 float fraction. A proximate analysis of the sink fractions is presented in Table VII for completeness. The ash content of

Table V

RESULTS OF RAW COAL ANALYSIS (RUN-OF-MINE)

<u>Coal Seam</u>	<u>Ash % dry</u>	<u>VM % dry</u>	<u>Sulfur % dry</u>	<u>Heating Value (Btu/lb) dry</u>	<u>Moisture % AR</u>
Cedar Grove	4.70	35.79	0.59	14,637	3.29
Lower Elkhorn	11.16	30.85	0.68	13,685	4.99
Black Creek	6.27	33.57	1.20	13,798	6.96
Splash Dam	21.73	27.15	0.57	11,822	3.30
Kentucky #11	20.84	26.01	4.70	11,188	9.15
Lower Kittanning	13.40	31.51	0.79	13,113	3.93
Pittsburgh #8 (OH)	14.18	34.47	1.37	12,331	7.50
Upper Freeport	18.55	32.34	1.18	12,290	4.97
Kentucky #9	11.26	39.52	4.73	12,810	8.42
Waynesburg #2 Split	9.83	30.77	2.13	13,914	3.37
Pittsburgh #8 (PA)	17.03	32.49	2.25	12,448	4.40
No. 2 Gas	34.03	24.65	0.85	10,195	4.26
Castle Gate 'D' plus Sub-seam 3	14.05	40.86	0.48	12,384	3.52
Hazard #4	40.00	27.41	1.07	8,381	9.12
Elkhorn #3	28.08	29.68	1.47	10,542	5.64
Illinois #6	17.55	35.75	3.92	11,372	10.06
Illinois #5	19.77	35.02	4.74	11,102	8.55
Mary Lee	38.95	23.65	0.76	8,667	5.24
Pocahontas #3	22.92	17.97	0.76	11,532	9.30
Jaw Bone	33.08	20.03	0.69	10,228	5.34

Table VI

PROXIMATE AND SULFUR FORMS ANALYSES OF 1.3 FLOAT FRACTION

<u>Seam</u>	<u>Ash % dry</u>	<u>VM % dry</u>	<u>Sulfur % dry</u>	<u>Heating Value (Btu/lb) dry</u>	<u>Sulfur (Wt%)</u>	
					<u>Pyritic</u>	<u>Organic</u>
Cedar Grove	1.56	37.06	0.60	14,935	0.02	0.58
Lower Elkhorn	1.47	36.13	0.63	15,225	0.04	0.59
Black Creek	2.27	35.88	0.80	14,441	0.00	0.80
Splash Dam	1.78	32.67	0.61	15,184	0.08	0.53
Kentucky #11	3.58	42.50	2.51	13,899	0.44	2.07
Lower Kittanning	2.40	37.26	0.78	15,016	0.01	0.77
Pittsburgh #8 (OH)	3.73	39.39	1.02	14,018	0.16	0.86
Upper Freeport	3.48	37.62	0.68	14,840	0.02	0.66
Kentucky #9	4.09	43.20	2.43	13,932	0.46	1.97
Waynesburg #2 Split	3.20	26.07	1.54	14,941	0.31	1.23
Pittsburgh #8 (PA)	3.58	32.66	1.04	14,470	0.08	0.96
No. 2 Gas	3.02	34.00	0.79	14,964	0.02	0.77
Castle Gate 'D' plus Sub-seam 3	4.18	45.78	0.55	14,059	0.02	0.53
Hazard #4	3.22	41.52	0.91	14,513	0.02	0.89
Elkhorn #3	2.64	42.96	1.06	14,861	0.15	0.91
Illinois #6	2.73	43.73	2.63	13,902	0.31	2.32
Illinois #5	2.78	43.91	2.51	13,676	0.23	2.88
Mary Lee	3.20	34.37	0.84	14,472	0.00	0.84
Pocahontas #3	3.45	22.63	0.59	15,228	0.00	0.59
Jaw Bone	4.77	26.50	0.72	15,028	0.05	0.67

Table VII

ASH AND SULFUR ANALYSES--1.3 SINK FRACTION

<u>Coal Seam</u>	<u>Ash % dry</u>	<u>Sulfur % dry</u>	<u>Heating Value (Btu/lb) dry</u>
Cedar Grove	11.06	0.52	13,471
Lower Elkhorn	23.18	0.80	11,346
Black Creek	13.27	1.84	12,620
Splash Dam	33.75	0.48	9,616
Kentucky #11	34.35	5.94	8,885
Lower Kittanning	23.04	0.79	11,569
Pittsburgh #8 (OH)	27.33	1.87	10,356
Upper Freeport	29.60	1.76	10,476
Kentucky #9	19.41	7.20	11,429
Waynesburg #2 Split	13.30	2.50	13,246
Pittsburgh #8 (PA)	29.41	3.30	10,672
No. 2 Gas	41.65	0.79	8,600
Castle Gate 'D' plus Sub-seam 3	17.12	0.45	11,737
Hazard #4	42.88	1.12	8,700
Elkhorn #3	32.35	1.51	9,719
Illinois #6	18.24	4.37	11,412
Illinois #5	21.98	4.54	11,281
Mary Lee	40.35	0.75	8,544
Pocahontas #3	25.92	0.62	11,268
Jaw Bone	37.02	0.65	9,494

the 1.3 specific gravity float coals range from approximately 1.5 to 4.8 percent, which suggests that not all coals can be cleaned to the required two percent ash level. A reduction in ash content yields a corresponding increase in volatile matter and heating value on an as-received basis. Conversely, sulfur levels are decreased due to the removal of pyrites.

Table VIII presents the results of ash fusion tests as well as a free quartz and soluble alkali analyses. The free quartz content of the ashes range from 4 to 20 percent. All of these values are below the minimum CWF feed specification of 25 percent. Also, the soluble alkali content is below the tolerable limit for all coals and is not a factor for additional consideration. With the exception of the Castle Gate "D" coal sample and the Illinois Nos. 5 and 6 samples, all of the coals have fluid temperatures (reducing) in excess of 2500°F, which is the minimum value given in the selection criteria. The maximum temperature noted was 2700°F. If the initial deformation temperature of a coal was greater than 2250°F, with the fluid temperature being greater than 2500°F, the coal was judged to be a potential candidate for the first round of tests.

Table IX lists the results of the mineral matter analyses of the ash for the 1.3 float fractions. The NaO contents of the coals are all within the acceptable range. The Fe_2O_3 content of the Kentucky No. 11, Kentucky No. 9, Waynesburg, Castle Gate, Illinois No. 6, and Illinois No. 5 coals all are relatively high.

Discussion of Selection Process

The criteria for selecting the eight coals have been given previously. To reiterate, they were: ease of separation, estimated recovery of two percent ash, and heating value.

Table VIII

ASH ANALYSIS--1.3 FLOAT FRACTION

Coal Seam	Ash % dry	Ash Fusibility		Free Quartz (% Ash)	Soluble Alkali (ppm)	
		Initial	Fluid		Na	K
Cedar Grove	1.56	2291°F	2508°F	17.6	56	18
Lower Elkhorn	1.47	2635°F	2700°F+	9.1	60	24
Black Creek	2.27	2357°F	2634°F	4.1	35	22
Splash Dam	1.78	2259°F	2700°F+	8.8	47	19
Kentucky #11	3.58	2375°F	2570°F	13.2	73	31
Lower Kittanning	2.40	2700°F+	2700°F+	11.0	66	26
Pittsburgh #8 (OH)	3.73	2600°F	2700°F+	17.7	86	28
Upper Freeport	3.48	2477°F	2700°F+	10.3	94	32
Kentucky #9	4.09	2428°F	2585°F	10.0	85	26
Waynesburg #2 Split	3.20	2662°F	2700°F+	7.4	58	18
Pittsburgh #8	3.58	2700°F+	2700°F+	14.0	80	28
No. 2 Gas	3.02	2660°F	2700°F+	13.0	84	34
Castle Gate 'D' plus Subseam 3	4.18	2121°F	2341°F	20.0	89	20
Hazard #4	3.22	2495°F	2700°F+	8.6	120	81
Elkhorn #3	2.64	2207°F	2550°F	7.9	93	35
Illinois #6	2.73	1975°F	2260°F	14.0	44	30
Illinois #5	2.78	1972°F	2256°F	15.0	116	32
Mary Lee	3.20	2485°F	2700°F	9.8	83	31
Pocahontas #3	3.45	2300°F	2594°F	13.0	89	63
Jaw Bone	4.77	2410°F	2699°F	12.0	49	40

Table IX

ASH COMPOSITION ANALYSIS--1.3 FLOAT FRACTION

Coal Seam	Mineral Ash Analysis (wt% dry)									
	<u>SiO₂</u>	<u>Al₂O₃</u>	<u>TiO₃</u>	<u>Fe₂O₃</u>	<u>CaO</u>	<u>MgO</u>	<u>K₂O</u>	<u>Na₂O</u>	<u>SO₃</u>	<u>P₂O₅</u>
Cedar Grove	42.78	29.25	2.08	9.07	4.76	2.11	1.66	1.15	5.25	0.10
Lower Elkhorn	54.75	26.72	2.01	6.31	2.31	0.98	1.99	1.01	2.41	0.15
Black Creek	42.39	31.29	1.74	8.08	5.54	1.89	2.85	0.42	3.64	0.23
Splash Dam	41.17	30.61	2.03	12.82	3.36	1.51	1.90	1.55	2.94	0.13
Kentucky #11	46.76	21.22	1.72	21.35	2.04	0.79	2.31	0.79	1.65	0.15
Lower Kittanning	57.94	29.87	2.26	3.49	1.90	0.79	0.73	0.85	1.56	0.15
Pittsburgh #8	56.74	25.25	1.37	10.36	1.46	0.48	2.15	0.74	0.48	0.21
Upper Freeport	47.74	29.21	1.99	8.73	3.39	1.59	2.00	0.64	3.08	0.36
Kentucky #9	49.85	20.82	1.88	20.08	1.54	0.70	2.65	0.64	1.29	0.07
Waynesburg #2	47.73	27.43	1.46	16.27	1.94	0.40	1.88	0.83	1.06	0.35
Split										
Pittsburgh #8	51.14	29.22	1.68	6.38	2.94	0.60	1.90	0.78	2.87	0.25
No. 2 Gas	50.26	31.13	1.82	7.49	1.46	0.95	2.48	0.92	1.46	0.09
Castle Gate D plus Subseam 3	41.82	17.80	1.47	7.51	9.80	6.24	0.42	1.01	10.90	1.05
Hazard #4	45.76	31.13	1.91	9.29	2.74	1.14	2.24	1.11	2.00	1.12
Elkhorn #3	37.96	28.57	1.62	15.33	4.34	1.11	2.05	1.38	4.37	0.86
Illinois #6	46.07	22.79	1.70	18.31	3.65	1.36	2.14	0.46	2.40	0.14
Illinois #5	46.64	21.22	1.73	16.26	3.78	1.61	1.78	1.08	3.03	0.20
Mary Lee	45.03	30.12	2.67	9.70	2.66	1.21	2.05	0.61	2.61	0.73
Pocahontas #3	52.75	24.65	1.48	7.76	2.66	2.62	2.98	0.78	3.09	0.10
Jaw Bone	51.47	27.75	1.67	8.13	1.82	1.61	2.86	0.54	1.71	0.20

The "estimated recovery at two percent ash" was obtained by assuming a linear recovery to ash content relationship. The estimated energy recovery was estimated in like fashion. As shown in Table X, the 1.3 float fractions have ash contents ranging from about 1.6 percent to 4.8 Percent. The sink material yielded a much greater variation. Recoveries ranged from one to 70 percent.

Ranking the coals, first by using "estimated recovery at two percent ash," and secondly, the "estimated energy recovery," yields expected results. The ranks roughly coincide with the 1.3 float recovery values. Table X lists all 20 coals with the properties which were given priority in making the eight-coal selection. In this table, the coals are ranked in order of preference. The first eight coals were recommended for the detailed study. All of the eight coals listed met the standards set by the selection criteria. It should be pointed out that the mineral analyses of the Kentucky No. 11 ash indicates a high level of Fe_2O_3 and Fe/Ca as compared to the other coals. These particular characteristics have historically been used to identify coals which give fouling and slagging problems. Nonetheless, this coal was included after additional geographic considerations were made.

Table X

ESTIMATED 2% ASH RECOVERY

Coal Seam	Est. Wt. Recovery at 2% Ash (%)	Est. Energy Recovery at 2% Ash (%)	1.3 Float Fraction		
			Recovery % Dry	Ash % Dry	Btu DAF
Cedar Grove	89.33	91.15	69.68	1.56	15,172
Lower Elkhorn	78.56	89.46	57.74	1.47	15,452
Black Creek	60.93	63.77	69.15	2.27	14,777
Splash Dam	53.52	68.88	47.63	1.78	15,459
Kentucky #11	38.11	42.77	68.21	3.58	14,415
Lower Kittanning	37.73	43.20	45.27	2.40	15,385
Pittsburgh #8 (OH)	32.51	37.20	60.64	3.73	14,561
Upper Freeport	25.56	30.89	44.48	3.48	15,383
Kentucky #9	27.85	29.11	56.96	4.09	14,526
Waynesburg #2 Split	25.81	27.72	41.30	3.20	15,435
Pittsburgh #8 (PA)	23.32	27.12	41.74	3.58	15,017
No. 2 Gas	19.34	28.39	29.21	3.02	15,430
Castle Gate 'D'	17.99	22.78	37.59	4.18	14,672
plus Sub-seam 3					
Hazard #4	12.09	20.30	19.47	3.22	14,996
Elkhorn #3	9.33	12.92	12.31	2.64	15,264
Illinois #6	7.11	8.70	9.71	2.73	14,292
Illinois #5	3.12	4.17	4.33	2.78	14,067
Mary Lee	2.64	4.41	4.23	3.20	14,509
Pocahontas #3	0.90	1.19	1.55	3.45	15,772
Jaw Bone	0.52	0.69	1.23	4.77	15,781

SECOND LEVEL SCREENING TESTS

The eight coals selected after the preliminary washability analysis were subjected to a second level of testing to further define beneficiation parameters. The results of the second level study were used in selecting the four candidate CWF coals and in identifying appropriate size and gravity fractions required to produce 7% and 2% ash beneficiated coals. A presentation of the data generated during this phase of the screening procedure follows.

Float-And-Sink Data

The eight coals were subjected to the series of float-and-sink testing and analytical procedures described in Table XI. The tests consisted of gravity separations of three top sizes (1-1/2" x 0, 3/8" x 28 mesh, and 28 mesh x 0) into seven density fractions, and subsequent analysis of the fractions for moisture, ash, recovery, and heating value (21 samples per coal). All sample preparation and testing was conducted in accordance with applicable portions of the ASTM standards.

Theoretical Recovery

The results obtained from the float-and-sink procedure just described, were used to project the recovery and corresponding properties for both 7% and 2% ash coal. These projections were made without consideration of plant performance characteristics and, therefore, may be considered ideal to theoretical in nature. 7% ash quality and recovery characteristics were derived from the data for 1 1/2 inch x 0 size coal given in the tables and are summarized in Table XII.

Table XI

SECOND LEVEL WASHABILITY ANALYSES

Eight Coals--Seven Specific Gravities--Three Top Sizes

Top Sizes	1-1/2" x 0
	3/8" x 28 Mesh
	28M x 0
Gravity Fractions	1.25 Float
	1.25 x 1.30
	1.30 x 1.35
	1.35 x 1.40
	1.40 x 1.50
	1.50 x 1.60
	1.60 Sink

Analyses on all size and gravity fractions (21 Samples per Coal)

% Moisture
% Ash
Btu
% Recovery

Table XII

THEORETICAL 7% ASH RECOVERY

1 1/2 INCH X 0 MATERIAL

<u>COAL I.D.</u>	<u>SEPARATION</u>	<u>RECOVERY %</u>	<u>ASH %</u>	<u>MOIST, %</u>	<u>HEATING VALUE, BTU/#</u>
Cedar Grove*	+1.60	100.0	4.16	1.96	14750
Lower Elkhorn	1.57	87.1	7.00	3.19	14070
Black Creek*	+1.60	100.0	5.23	3.96	13820
Splash Dam	+1.60	84.9	7.00	1.16	14410
Lower Kitt.	1.43	78.7	7.00	1.01	14300
W. KY #11	+1.60	95.7	7.00	2.92	12540
Pittsburgh #8	1.50	86.4	7.00	3.89	13250
Upper Freeport	1.37	68.4	7.00	1.76	14170

*Low ROM Ash Content

As an aid in determining the characteristics of the 2% ash coal, curves for yield, cumulative float ash, and cumulative sink ash were derived for the 3/8 inch x 28 mesh material and are illustrated in Figures 4 through 11. Theoretical properties of the 2% ash coals are given in Table XIII.

Findings

Five of the eight coals, Cedar Grove, Lower Elkhorn, Black Creek, Splash Dam, and Lower Kittanning yielded results indicating that 2% ash recoveries ranging from 25 to 86 percent can be expected under ideal conditions, with a 3/8 inch by 28 mesh gradation. However, two of these coals, Cedar Grove and Black Creek, typically have very low ROM ash contents (<7%) which lead to difficulty in obtaining 7% ash coal from these sources.

FIGURE 4 WASHABILITY CURVES FOR CEDAR GROVE COAL, 3/8" x 28 MESH

FIGURE 5 WASHABILITY CURVES FOR LOWER ELKHORN COAL, 3/8" x 28 MESH

FIGURE 6 WASHABILITY CURVES FOR BLACK CREEK COAL, 3/8" x 28 MESH

FIGURE 7 WASHABILITY CURVES FOR SPLASH DAM COAL, 3/8" x 28 MESH

FIGURE 8 WASHABILITY CURVES FOR LOWER KITTANNING COAL, 3/8" x 28 MESH

FIGURE 9 WASHABILITY CURVES FOR WESTERN KENTUCKY #11 COAL, 3/8" x 28 MESH

FIGURE 10 WASHABILITY CURVES FOR PITTSBURGH #8 (OH) COAL, 3/8" x 28 MESH

FIGURE 11 WASHABILITY CURVES FOR UPPER FREEPORT COAL, 3/8" x 28 MESH

Table XIII

THEORETICAL 2% ASH RECOVERY

3/8 INCH X 28 MESH MATERIAL

COAL ID	SP. GR SEPARATION	RECOVERY			ASH, %	MOIST., %	HEATING VALUE, BTU
		CRUSH	WASH.	TOTAL			
Cedar Grove	1.34	90.2	94.5	85.2	2.00	1.69	15060
Lower Elkhorn	1.345	88.3	73.5	64.9	2.00	1.60	15020
Black Creek	1.305	91.1	84.0	76.5	2.00	3.31	15870
Splash Dam	1.30	84.9	60.0	50.9	2.00	1.18	15340
Lower Kitt.	1.275	85.0	30.0	25.5	2.00	1.01	15300
W. KY. #11	1.30	86.6	61.9	53.6	3.62	3.67	14560
Pittsburgh #8	1.30	85.0	43.1	36.6	3.48	2.67	14440
Upper Freeport	1.30	85.0	49.1	41.7	3.49	1.15	14900

COAL BENEFICIATION

The purpose of the detailed washability studies was to identify those coals that are capable of being beneficiated to 2% ash at lowest costs. These coals will probably be separated at or near a 1.3 specific gravity. 2% ash beneficiation will also include a mineral matter liberation step prior to the gravity separation step. Due to the limitations of fine coal (minus 28 mesh) cleaning at low specific gravities, the coals will be crushed in a manner such that fines production is minimized.

A request for proposal was sent to sixteen potential beneficiators identified in Table XIV. A list of the eight coals, along with available washability data, and the schedule for delivery of each coal was included in the request. Five of the candidate beneficiators responded with formal proposals and cost estimates. Costs for completing the 2% ash cleaning ranged from \$231,000 to \$772,000 and averaged \$490,000.

Selection of Coal Beneficiator

Each proposal was reviewed and evaluated based on the factors imposed by overall CWF program objectives. Since the CWF program does not include an R&D effort directed toward coal preparation, acceptable proposals were limited to those employing conventional cleaning methods. Additional factors affecting final selection of the beneficiator are discussed below.

- o Facility capabilities and capacities were reviewed relative to their ability to meet the delivery schedule for 2% ash coal in a timely manner. The facilities' bulk handling and storage capabilities were also given due consideration.
- o The geographic locations of the facilities were considered with regard to additional shipping and handling cost that may occur as a result.

Table XIV

LIST OF CANDIDATE BENEFICIATORS

	Beneficiator	Location
1.	McNally Pittsburg, Inc.	Pittsburg, Kansas
2.	Roberts & Schaefer Co.	Pittsburgh, PA
*3.	Dravo Corporation	Pittsburgh, PA
*4.	Iowa State University	Ames, Iowa
5.	Pioneer Coal Preparation, Ltd.	Ripley, England
6.	University of Kentucky	Lexington, KY
7.	Consolidation Coal Co.	Pittsburgh, PA
8.	Penn. State University	University Park, PA
*9.	Michigan Tech. University	Houghton, Mich.
*10.	Colorado School of Mines	Golden, CO
11.	Armco Coal Co.	Middletown, Ohio
*12.	Mountain States R&D Co.	Tucson, AZ
13.	Battelle Memorial Institute	Columbus, Ohio
14.	Bituminous Coal Research Institute	Monroeville, PA
**15.	Electric Power Research Institute	Homer City, PA
16.	West Virginia University	Morgantown, W. VA.

*Submitted formal proposals

**Responded as a part of Dravo's proposal

- o The availability of the plants were reviewed in order to anticipate project delays or plan necessary lead times required.
- o The beneficiators' plant performance history and staff experience were examined in order to provide reasonable insight into performance that can be expected for this project.

The proposal submitted by the Energy and Minerals Resources Research Institute of Iowa State University (ISU) was selected as best fitting all the factors previously discussed. ISU proposed a relatively simple, commercially used cleaning circuit that offers a controlled performance at low specific gravity separations. Details of this proposal are discussed in the following section.

Coal Cleaning Methods

7% Ash Coal

Generally, adequate supplies of 7% ash coal are being produced in commercial coal preparation facilities. These facilities normally provide this cleaned product from a blend of different coal sources or seams. For purposes of this program, where a particular seam is to be isolated as the coal source, special handling arrangements were made with the company operating the cleaning facility in order to meet this requirement.

2% Ash Coal

As previously mentioned, ISU was selected as the 2% ash beneficiator for this program. The coal was acquired in the 7% ash range from the commercial cleaning plant and shipped either by truck or rail to the ISU pilot preparation facility at Ames, Iowa. Less than carload lots were delivered by truck; larger shipments were delivered by rail in hopper-bottom cars. To prevent contamination with other coal, each rail shipment was unloaded with an under-car conveyor with delivery directly into trucks. The coal was placed on a hard-surface lot for storage.

ISU personnel collected a head sample and analyzed for moisture, ash, total sulfur, and heating value. A second sample was crushed to a nominal 1/4 inch top size, float-sink tested at several specific gravities, and analyzed for moisture and ash to aid in predicting plant performance. Based on these analyses, expected yield, and previous experience, a target specific gravity of separation was established for cleaning the coal.

The coal was crushed in a once-through impact mill to a nominal top size of 1/4 inch and cleaned in an eight-inch heavy-media cyclone circuit (see Figure 12). The circuit scalps any plus 3/8 inch material for recrushing and deslimes the cyclone feed at 1/2 mm. Present drain and rinse screens and magnetic separators do not perform acceptably if the feed is not deslimed.

After approximately 15 minutes of operation at a five-ton per hour feed rate, samples of the product and refuse were collected; the clean coal was analyzed for moisture and ash content, and the yield to clean coal was determined. The quality and yield of the product were compared to the desired results. As necessary, the heavy-media density was adjusted and the test repeated. When desired product quality was attained in a preliminary test, the remainder of the coal was processed without varying operating conditions and samples of the product and refuse collected periodically.

Expected Clean Coal Properties

Typically, the distribution curve is used to evaluate the efficiency of a given coal-cleaning device. This curve is derived by plotting the percentage of each density fraction of the raw coal that was recovered in the washed coal against the mean densities of the density fractions. Once this curve has been experimentally obtained, it can be used in conjunction with the washability data for a raw coal to predict both the yield and quality of the cleaned coal.

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FIGURE 12 HEAVY MEDIUM CYCLONE CIRCUIT

The curve shown in Figure 13 is the distribution curve for the eight-inch cyclone at ISU, operating at a low specific gravity. This curve was used in conjunction with the washability data derived for each of the eight coals selected for this program. Table XV provides a summary of the estimated yields and qualities of each of these coals for a 3/8 inch x 28 mesh particle-size distribution. Results indicate that five of the eight coals can be used as suitable feeds for producing a 2% ash product, although the Lower Kittanning coal was marginal in this respect.

FIGURE 13 ISU 8-INCH CYCLONE DISTRIBUTION CURVE

Table XV

ESTIMATED 2% ASH RECOVERY

3/8 INCH X 28 MESH MATERIAL

COAL ID	SP. GR SEPARATION	RECOVERY, %			ASH, %	MOIST., %	HEATING VALUE, %
		CRUSH	WASH.	TOTAL			
Cedar Grove	1.34	85	76	64.6	2.00	1.68	15040
Lower Elkhorn	1.34	83	63	52.3	2.14	1.60	14990
Black Creek	1.30	86	58	49.9	1.97	3.33	15880
Splash Dam	1.30	80	44	35.2	2.14	1.18	15320
Lower Kitt.	1.275	80	25	20.0	2.61	0.97	15020
W. KY. #11	1.30	81	47	38.1	3.77	3.65	14530
Pittsburgh #8	1.30	80	34	27.2	3.85	2.64	14350
Upper Freeport	1.30	80	37	29.6	3.73	1.15	14860

CWF SELECTION

The suitability for the manufacture of coal water fuels was determined by the vendors utilizing their proprietary processes. The generalized candidate coal ratings are shown in Table XVI.

Fuel Specifications

The specifications developed for the test CWFs were primarily designed to address anticipated user's needs. Five basic slurry characteristics were addressed:

- Solids loading
- Particle topsize
- Rheology
- Uniformity
- Stability

Since testing procedures used in characterization of CWF had not been fully developed, the criteria used to define certain slurry properties are expected to be further refined. The specifications shown in Table XVII are based upon information which in several areas was limited. However, while the actual values and analytical techniques used to measure those slurry properties may vary, user's specifications in these areas are believed to be necessary. (Actual test fuels varied from these targets, as described in Volumes 3, 4, and 5.)

A solids loading specification was imposed to ensure test CWFs were representative of commercial products. Solids loading was generally determined by measuring moisture content. A range of 68 to 70% solids was established based upon a survey of commercially available CWFs. If all other properties are held constant, the benefits of higher solids loading are obvious. However, there are generally trade-offs between solids loading and CWF viscosity.

TABLE XVI

GENERALIZED CWF CANDIDATE COAL RATINGS

Coal	Washability @ 1.3 Specific Gravity		Ash, %	Combustion*	CWF Manufacturing	Comments
	Recovery, %					
Cedar Grove	85.2	2.00	Marginal	Acceptable	Low Fuel Reactivity	
Lower Elkhorn	64.9	2.00	Moderate	Marginal	Questionable Slurry-ability	
Black Creek	76.5	2.00	Good	Unacceptable		
Splash Dam	50.9	2.00	Moderate	Excellent	Reference CWF Coal	
Lower Kittanning	25.5	2.00	Moderate	Marginal	Oxidized Coal in Feed	
Western Kentucky #11	53.6	3.62	NA**	NA**	High Sulfur; Poor Ash Liberation	
Pittsburgh #8	36.6	3.48	NA**	NA**	High Sulfur, Poor Ash Liberation	
Upper Freeport	41.7	3.49	Good	Acceptable	High Sulfur; Poor Ash Liberation	

* Combustion Engineering TGA Method

** NA--Not Analyzed

TABLE XVII

PROPOSED SPECIFICATIONS FOR COAL WATER FUELS

PERCENT SOLIDS:	68-70 wt%
MAXIMUM PARTICLE SIZE:	Less than 0.0009% on 30 Mesh Screen Less than 0.050% on 60 Mesh Screen Less than 0.30% on 100 Mesh Screen
VISCOSITY:	1) Less than 2800 mPa.s @ 100 sec ⁻¹ , 20°C (Haake method). 2) Less than or equal to 1.1 power factor over shear range of 10-100 reciprocal seconds (Haake method).
pH:	7.5 to 9.0
STABILITY:	When the CWF is delivered, it must be pumpable; it must not be hardpacked.
UNIFORMITY:	Upon completion of preparation of the CWF, and following agitation or circulation if necessary, its total solids content shall not differ by more than 0.6% between the top 10% and the bottom 10% in each tank. Uniformity should last 60 days,
TOXIC COMPONENTS:	All toxic and/or hazardous components must be identified and their concentrations must be stated. The concentration of each toxic component must not exceed OSHA- or EPA-established limits.
NOTE:	The use of alkali metal salt additives is not recommended.

Specifications of fuel rheological properties are needed to predict pumping and atomization requirements. Unfortunately, CWF rheological characteristics are very complex and extremely difficult to predict. The most widely accepted viscosity measurement technique employs a Haake viscometer. This technique appears to provide good precision and consistent results at relatively low (100 sec^{-1}) shear rates. A maximum CWF viscosity of 2800 mPa.s at 100 sec^{-1} was specified using this technique. In addition, since CWF generally does not exhibit Newtonian characteristics, the rate of viscosity increase as a function of shear rate was considered. If the viscosity/shear rate relationships are described as a power law function,

$$\mu = KY^{n-1}$$

where μ = apparent viscosity
 Y = shear rate
 K = flow consistency index
 n = flow behavior index

Then n is a measure of the change in viscosity with shear rate. CWFs with flow behavior index exponents less than 1.0 are pseudoplastic (viscosity decreases with shear rate), and those with exponents greater than 1.0 are dilatant (viscosity increases with shear rate).

Extrapolation of the Haake viscosity data to predict atomization behavior at shear rates roughly two orders of magnitude higher is questionable, and present data do not show reliable correlation. The primary use of this low shear rate data is to estimate pumping characteristics. (Evaluation and development of high shear viscosity measurement techniques was being conducted by several organizations, and methods have since been proven.)

Identification of any toxic or hazardous substances contained in the CWF is necessary in order to ensure proper shipping, handling and disposal procedures. In general, CWF has been shipped and handled as nonhazardous material. The recommendation against alkali metal additives is an attempt to circumvent the deleterious effect of sodium based additives upon slagging and fouling tendencies of the coal mineral matter.

Slurry stability is a very broad category addressing changes in slurry properties with time. Various laboratory techniques have been developed to assess static rates. Although the need for long term static storage is still uncertain, an indication of settling rates and agitation requirements is necessary. Key characteristics such as settling with hardpack and substantial changes in viscosity with time are information essential for appropriate handling and utilization. The shear history to which a CWF is exposed can also have a significant effect on rheological properties. Information concerning the sensitivity of a slurry to shear is also very valuable.

A series of analyses were carried out on selected CWFs by C-E and GR&DC, and correlations were later made between this information and actual performance. The characterization tests are listed in Table XVIII and a brief description of the procedures in Table XIX.

At the beginning of the project, CWF vendors were sent letters of invitation to participate and proposed specifications (Table XVII). Also, visits were made to facilities of prospective suppliers. The vendors chosen were Advanced Fuels Technology (Sohio), Atlantic Research Corporation, Coal Liquids, Ergon and OXCE Fuel Corporation. These vendors felt that the proposed specifications were unrealistic, especially for CWF made from coals not previously used by them. The vendors would, however, make a best effort to meet the program goals. Each manufacturer had a quality control program to ensure the integrity of their products, and CWF for this project was ordered on that basis with the specifications being designated as desired properties.

Reference CWF Selection

In the project plan, a "reference slurry" was to be chosen as early as possible which would be representative of an average CWF for the purpose of testing various burner designs. The first coal chosen for this CWF was the Lower Kittanning from the Armco Coal Company, Mine No. 25, Montcoal, West Virginia.

TABLE XVIII

CHARACTERIZATION TESTS FOR COAL WATER FUELSGulf Research & Development

Percent Solids

Density

Particle Size Distribution

Viscosity

Variable Shear Rate

Constant Shear Rate

Effect of Temperature Change
(Freeze-Thaw)

Settling Rate, 20°C, 40°C, 60°C

Aging

Agglomeration

Abrasiveness

Corrosion

Combustion Engineering

Proximate

Ultimate

Higher Heating Value

Flammability Index

Ash Composition

Ash Fusibility

Sulfur Forms

Thermo-Gravimetric Analysis

Weak Acid Leaching

Scanning Electron Microscopy

TABLE XIX
PROCEDURES FOR CHARACTERIZATION OF CWF

Analysis	Procedure
Solids	A 10 g sample was weighed into a disposable 57 mm aluminum dish and heated @ 100°C @ 15" vacuum with small nitrogen flow until constant weight was achieved (minimum 2 hours).
Density	A Fisher pycnometer was utilized at ambient temperature.
Particle Size Distribution	<p>A 1000 g sample was diluted 1:1 with water and wet sieved through a series of screens (30, 60, 100, and 200 mesh). The screens were dried at 110°C, the material on the screens weighed and reported as % on the respective screen.</p> <p>The particle size distribution of the -200 mesh material was determined with a Leeds & Northrup Microtrac (7991-02) instrument.</p>
Viscosity	A Haake RV-100 with an MVIII sensor system was utilized, over a shear rate range of 0-190 sec ⁻¹ , at 20°, 30° and 40°C. Three flow curves at each temperature were recorded and data collected from the third acceleration curve. From this curve the viscosity at 100 sec ⁻¹ and the power law factor were calculated. A constant shear flow curve is also obtained at 100 sec ⁻¹ . Viscosity at higher shear rate ranges

TABLE XIX (CONT'D.)
PROCEDURES FOR CHARACTERIZATION OF CWF

<u>Analysis</u>	<u>Procedure</u>
	(5000 sec ⁻¹) was determined by Adelphi Center for Energy Studies.
Effect of Temperature Change (Freeze-Thaw)	The CWF was subjected to three freeze-thaw cycles and then settling rate, viscosity and particle size distribution determined.
Settling Rate	Settling rates of 20°C and 40°C were determined by periodically analyzing samples from the top, middle, bottom and drain of a 65 x 7.5 cm jacketed column. Settling rate at 60°C was determined by heating 1 liter container of the CWF for seven days and analyzing the top, center and bottom for solids content. Viscosity of total samples was also determined at 20°C.
Aging	Five-gallon containers were periodically observed for amount and nature of sediment.
Agglomeration	The particles retained on the screens from the particle size determination were photographed through a microscope (20X) and their physical composition observed.
Abrasiveness	A five-gallon sample was circulated in a pump loop for five hours and the abrasion of test elements determined.

TABLE XIX (CONT'D.)
PROCEDURES FOR CHARACTERIZATION OF CWF

<u>Analysis</u>	<u>Procedure</u>
Corrosion	Carbon and 316 stainless steel coupons were exposed to the CWF for periods ranging up to 100 days. After exposure, weight loss and physical appearance changes were determined.
Flammability Index	A measurement of the ignition temperature of a pulverized fuel in an oxygen atmosphere. This is a relative measurement which is a direct indicator of a pulverized fuels ignition stability and turndown characteristics.
Thermo-Gravimetric Analysis	A C-E in-house procedure used to assess fuel reactivity/char burnout characteristics. A 5 mg char sample (prepared either by ASTM D-271 volatile matter determination or in a Drop Tube Furnace system) is burned isothermally at 700°C in air atmosphere. The weight loss of the sample is measured as a function of time. This thermogram is used to predict combustion efficiency.
Weak Acid Leaching	A pulverized fuel sample was leached in a diluted solution of acetic acid at a controlled pH for 30 minutes. After the leaching period the slurry was filtered and the leachate analyzed. These data indicate the concentration of active alkali in the coal and can be used in assessing fouling potential.

The overall approach for selecting a coal for CWF was to select run of mine coals that met the combustion and cleanability criteria and then submit them for vendor evaluation. Upon approval by the vendor, large quantities of the coal cleaned to 7% ash would be purchased and sent to the various vendors for production of 20 gallon batches. These small batches were to meet the proposed set of specifications which included a minimum solids loading of 70% and a maximum viscosity of 2800 mPa.s at 100 sec⁻¹. A 1-1/2 ton ROM sample of Lower Kittanning coal was sent to one producer, where a stable CWF was produced containing 70.8% solids and having acceptable rheological properties. On the basis of these results, 800 tons of the Lower Kittanning coal were purchased and cleaned to an ash level of 6.4% in a heavy media plant with a fine coal circuit employing froth flotation. Appropriate quantities of the cleaned coal were sent to four commercial CWF manufacturers. None could produce an acceptable CWF at the required 70% solids loading. One vendor, based on previous experience, was able to produce a stable CWF at 66% solids loading that stored, pumped, atomized and combusted well (Table XX).

The other vendors were not asked to investigate or develop a formulation to solve the problem. During this development effort, it was found that the beneficiated coal was much more oxidized and exhibited a 2% higher equilibrium moisture than the original sample. These results indicated that the effective solids loading (bone-dry) of a CWF would be lowered by over 2% (such as from 70% to 68%) by these two factors and that a CWF of 66-68% solids loading could be produced, which was later demonstrated (Table XX).

This difference in behavior of the two coals raised serious questions as to the influence of beneficiation upon slurriability. To answer these questions, several investigations were undertaken by others addressing

- (a) changes in mineral matter during beneficiation,
- (b) changes in surface properties as a result of beneficiation, and
- (c) confirmation and relevance of the equilibrium moisture and oxidation findings.

TABLE XX

ANALYSIS OF LOWER KITTANNING CWF

	Original	Beneficiated	Batches
	1/83 ROM		
	<u>Coal</u>	<u>10/12/83 (715 Gal)</u>	<u>11/3/83 (4250 Gal)</u>
Solids, %	70.7	68.0	66.1
Viscosity mPa.s @ 100 sec ⁻¹ (Haake)	1600	2830	1200
Power Factor 10-100 sec ⁻¹		0.69	0.84
Particle Size Distribution			
<30 mesh, %		100.00	100.00
<60 mesh, %		99.92	99.88
<100 mesh, %	99.7	99.04	99.28
pH		8.15	7.95

Based on the information developed, it was concluded that the reason the beneficiated Lower Kittanning coal would not consistently produce a 70% solids CWF with acceptable viscosity is associated with a volumetric property or maximum packing fraction. It appears that the polar groups on the surface of the highly oxidized coal immobilize through hydrogen bonding a sufficient quantity of water to significantly increase the effective volume and decrease the maximum packing fraction so that an acceptable 70% solids CWF could not be produced. The highly oxidized condition resulted from the inadvertent inclusion of some outcrop coal in the coal supply.

The next coal evaluated as feed for the reference slurry was the Splash Dam. Four hundred tons of coal beneficiated to approximately 6% ash were purchased and representative quantities furnished to the vendors for production of small batches of CWF. Properties are summarized in Table XXI.

These data demonstrated that several manufacturers could produce acceptable CWF from the Splash Dam coal, and it was chosen as the reference coal.

Representative data for the Splash Dam CWFs are shown in Tables XXII and XXIII. In general, densities ranged from 1.203 to 1.211, the pH varied from 7.7 to 9.6, the mean volume of the minus 200 mesh coal ranged from 14 microns to 24.5 microns, the viscosity decreased with increasing temperature, higher temperature increased the rate of settling but did not induce hard pack, freeze-thaw cycles caused some separation but essentially original properties were restored with gentle mixing, some particle agglomeration occurred, and five gallon sample stability was indicative of large-scale storage behavior. Data on other CWFs are given in Volumes 3, 4, and 5.

In order to preserve the anonymity of CWF manufacturers, an alphabetic code letter for each was used. To each CWF made for this program, an identification label which provides an abbreviated description of the CWF was assigned. For example, SD64C refers to a CWF for which the source coal is Splash Dam, the ash level is 6.4%, and the manufacturer is company C. Source coals are abbreviated as follows: UF = Upper Freeport, SD = Splash Dam, CG = Cedar Grove, LK = Lower Kittanning, and AL = Alma Seam.

TABLE XXI

PROPERTIES OF SMALL BATCHES OF CWF PRODUCED FROM
SPLASH DAM COAL BENEFICIATED TO 6% ASH

<u>Vendor</u>	<u>Solids Content %</u>	<u>Viscosity</u>		<u>Particle Size % of CWF</u>		<u>Stability</u>
		<u>mPa.s @, 100 sec⁻¹</u>	<u>Power Law Factor 10-100 sec⁻¹</u>	<u>+30 Mesh</u>	<u>-200 Mesh</u>	
A	68.9	1405	1.4	0.001	95.6	Softpack
C	71.2	1010	1.3	0.003	95.2	Softpack
E	67.9	1209	0.98	0.004	99.6	Softpack

TABLE XXII

PROPERTIES OF LARGE BATCHES OF SPLASH DAM CWF

Manufacturers	A	C	D	E
Solids Content (%)	68.9	70.1	70.7	66.4
Particles +30 Mesh(%)	0.001	0.001	0.001	0.015
Particles -200 Mesh (%)	87.8	94.7	96.7	99.6
Initial Sediment	Soft	Soft	Soft	Soft
After Stirring	Dispersed	Dispersed	Dispersed	Dispersed
Density @ 25°C	1.203	1.211	1.204	1.205
pH	9.6	8.8	7.9	7.7
Viscosity @ 100 sec ⁻¹ (mPa.s):				
20°C	1495	635	837	858
30°C	1153	463	436	608
40°C	981	341	370	554
Power Law Exponent between 10 and 100 sec ⁻¹ :				
20°C	1.0	1.4	1.5	1.2
30°C	0.9	1.3	1.1	1.0
40°C	0.9	1.2	1.1	1.3

TABLE XXIII

STABILITY SETTLING COLUMN DATA
VENDOR C SPLASH DAM CWF

Temperature 20°C
Position, % Solids

Day	1	2	3	Bottom
Feed	70.1	70.1	70.1	70.1
1	70.3	70.2	70.4	70.5
2	70.1	69.8	70.0	70.3
3	69.8	69.6	69.5	70.5
9	70.4	69.9	70.4	71.6
16	70.5	70.1	70.1	70.9

Temperature 40°C

Feed	70.1	70.1	70.1	70.1
1	69.6	69.6	69.8	70.6
2	70.2	70.1	70.1	69.1
3	70.0	70.1	68.8	70.4
9	69.5	69.7	70.1	71.6
16	69.6	70.0	69.9	73.7

Temperature 60°C*

7	67.7	71.8	73.3
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*1-Liter Plastic Container - Heated, Frozen, Sectioned, Analyzed

CWF PREPARATION AND SUPPLY

The purpose of this activity was to provide representative samples of CWFs required for the test program. Activities consisted of coordination and supply of the appropriate fuels, in the required quantities and, in the proper time frames. The work was divided into four parts:

1. Coal Acquisition - This activity included the acquisition and beneficiation of parent coals in the quantities required for evaluations and tests, which include that required for the 18 candidate CWFs selected for bench-scale testing, the coal required for the 5 CWFs tested during the combustion evaluation, and the coal required for the 12 CWFs evaluated during the performance testing.
2. Beneficiation of Test Coals - The clean coals for preparing the CWFs for the large-scale combustion tests were produced from the beneficiation of the selected raw coals at coal cleaning plants.
3. CWF Preparation - Clean coals were shipped to the selected slurry manufacturers who prepared CWF in the required quantities for the tests.
4. CWF Supply - Representative test samples of each CWF were shipped to appropriate testing facilities for laboratory analysis or combustion testing as required during execution of the project.

Batches of CWF were procured from five CWF vendors, who used feed coals obtained by Gulf Research and Development Company (GRDC). The coordination of scheduling, shipping and storing was also done by GRDC, to match the test schedules.

Four coals were obtained to produce eighteen small batches of CWF for bench-scale screening tests. Three out of these four coals were used to produce large batches of CWF for the commercial-scale combustion tests under

Task 4. A beneficiated microfine coal was obtained in addition to the four coals to produce twelve CWFs for the pilot-scale slagging and fouling evaluation under Task 5.

Most coals were washed by the coal companies, while several batches were beneficiated by CWF vendors or Iowa State University. Three coals were shipped directly to C-E for baseline dry pulverized coal tests. Figures 14, 15 and 16 illustrate the histories of the Splash Dam, Cedar Grove, and Upper Freeport coals used in the project. The Alma coal used for microfine CWF production was shipped directly from the mine/cleaning plant to the CWF manufacturer to C-E.

FIGURE 14 HISTORY OF SPLASH DAM CWF PROCUREMENT

FIGURE 15 HISTORY OF CEDAR GROVE CWF PROCUREMENT

FIGURE 16 HISTORY OF UPPER FREEPORT CWF PROCUREMENT

CWF TRANSPORTATION TEST

Gulf Research & Development Company designed and assembled a data acquisition system which recorded the following conditions during transportation of two CWFs over 2700 miles: outdoor ambient temperature, CWF temperatures at five levels, vibration in the vertical plane, and pressure in the tank.

The data acquisition system was battery-powered and required no attention during transit. Except for some components, the data acquisition system was housed in two weatherproof and tamperproof cabinets. This system was affixed to a 1500-gallon tank which was mounted on a flatbed truck trailer. Data were collected and stored continuously during transit. Then, at the destination the data were "dumped" via telephone line to a personal computer at GRDC in Harmarville, PA.

A portable, skid-mounted, 1500-gallon tank, 48-inch diameter by 16 feet, with horizontal profile was hauled on a flatbed trailer. CWF was loaded through a manway in the top and drained through a bottom nozzle. Samples were obtained through the manway. Instrumentation had been inserted upward through a 3 inch port on the bottom surface of the tank. Data acquisition components were thermocouples, accelerometer, pressure transmitters, vibration meter, data logger, tape recorder, and telephone modem. The monitoring system is depicted in Figure 17.

The "heart" of the data acquisition system was a small, portable data logger which collected all measurements. It logged data continuously during transit, integrated data each hour (per each log cycle), then stored the integrated data in its random access memory (RAM) and on a cassette tape for backup.

In the laboratory, a static test was performed in order to compare settling at constant temperature and no vibration with that settling which may have occurred during transportation. An elongated steel drum was constructed to simulate the depth (48 inch or 120 cm) of CWF in the 1500-gallon tank. Samples were taken through a port in the center of the lid. Data were collected during two shipments of coal-water fuel in September and October 1984.

FIGURE 17 CWF TRANSPORTATION MONITORING SYSTEM

Monitoring of Transportation Conditions

A coal-water fuel containing 2.2% ash, made from Splash Dam coal by Vendor C, was shipped to TRW in September 20-25, 1984. For the first time transportation conditions were monitored during the nearly 2700-mile trip. Measurements included:

- o outdoor ambient temperature
- o internal temperatures at two levels within the CWF
- o vibration of the tankful of CWF
- o tank pressure

Data from the above-mentioned measurements are presented in Figures 18, 19, and 20. All graphs were plotted using hourly integrated data; i.e., data were collected every minute then averaged for an hourly point.

Due to corrosion, only 2 of 5 internal thermocouples functioned. Corrosion on three wire leads caused opened circuits and, therefore, no temperature readings were recorded.

As expected, the CWF did not experience the extremes of ambient temperatures, which ranged from 14°C (57°F) to 34°C (93°F) (see Figure 18). Temperature in the mid-top of the CWF (36 inches from bottom of tank) varied over a narrower range than that near the tank surface (top and bottom, not side of tank). Temperature of CWF near the top surface (44 inches from bottom or 4 inches down from top) ranged from 16°C (61°F) to 37°C (98°F), while temperature near the center ranged from 26°C (77°F) to 34°C (93°F). These two temperatures over the 6 day period are plotted in Figure 19.

Vibration data for this first trip (Trip 1) are plotted in Figure 20. These integrated values include both minor vibrations and large shocks. The zero values correlate with the times when the truck was not moving. Based on the experience of the GRDC personnel, a value above 2 units represents travel over rough highway while values below 2 units represents travel over smoother

FIGURE 18 AMBIENT TEMPERATURE DURING TRIP 1

FIGURE 19 INTERNAL TEMPERATURES DURING TRIP 1

FIGURE 20 VIBRATION DURING TRIP 1

sections of highway. According to the manufacturer of the vibration detector/meter (Bruel & Kjaer), temperature fluctuations had no significant effect on vibration measurement.

To place these measurements into perspective, a comparison was made with data from other dynamic vibration tests. Table XXIV was obtained from an EPRI Report which reported on other vibration studies. By assuming vibration frequencies of 100-200 Hz, the values in Figure 20 indicate that the vibrations experienced during Trip 1 were in the range of truck travel over unimproved roads.

The internal tank pressure was measured at a point 8 inches below the surface. The tank pressure varied from a low of 1.8 psig to maximum of 4.7 psig correlating very well with the changes in ambient temperature.

A system based on differential pressure measurements was designed to infer settling as a result of density changes. Initially the measurement of pressures was planned at four different levels by use of one differential pressure cell and a rotating selector valve with connecting lines. Due to mechanical problems and time and money constraints, satisfactory instrumentation was not developed; however, the concept appears to be sound.

In October 1984, a second CWF (SD64F) was shipped from Vendor F in the East to the TRW test site in California. Like the first trip, this shipment was continuously monitored for temperature and vibration. A GRDC technician traveled with the CWF and sampled it periodically at four different levels for subsequent analysis of solids content. The settling data are reported in Table XXV.

Transportation of these two CWFs from the East Coast to California had no detectable effect upon their observed properties. Comparison of the stability data from static and transit samples showed that very little settling occurred. The settling behavior observed in the laboratory paralleled the behavior observed during transportation.

Table XXIV
VIBRATION MEASURED IN FIELD

<u>Transportation Mode</u>	<u>Frequency Range (Hz)*</u>	<u>Peak Acceleration Level (g's)*</u>	<u>Vibration Velocity in GRDC Test (inches/sec)</u>	
			<u>at 100 Hz</u>	<u>at 200 Hz</u>
Ship	0 - 20	0.4 - average 0.9 - peak	-	-
Rail	0 - 20	0.9 - average 2.0 - peak	-	-
Truck on highway	0 - 100	0.5 - average 2.0 - peak	1.2	-
Truck on unimproved roads	0 - 200	0.5 - average 3.0 - peak	-	0.9

* "Coal-Water-Slurry Evaluation, Volume 2: Laboratory and Combustion Test Results", Electric Power Research Institute, EPRI-CS-3413, Project 1895-3, Final Report, February 1984.

TABLE XXV

Transportation Settling Test - Trip 2
(CWF in drum on 10/11/84, 70.5 wt% solids)

	Weight % Solids Sampled by GR&DC				Sampled 10/17 by TRW [*]	
					<u>Depth</u> ^{**}	<u>% Solids</u>
Date	10/12	10/13	10/14	10/16		
Depth**						
6.5 inches	70.7	70.6	70.6	70.9	7	70.5
19	70.9	71.0	70.9	71.1	19	70.4
33.5	71.2	71.2	71.1	71.1	30	70.8
41	71.5	70.8	71.2	70.6	42	71.1

* TRW used their sampler; 70.9 weight % solids after sparging.

** Depth from top surface of CWF.

CONCLUSIONS

Five of the eight coals selected for detailed washability studies were capable of being cleaned to 2% ash. These source coals include the Cedar Grove, Lower Elkhorn, Black Creek, Splash Dam, and Lower Kittanning. General ratings of the coals relative to their washability, combustion, and suitability as CWF feeds are presented in Table XXVI.

The CWF vendors expressed concern over the need to produce high-solids CWFs from all coals. The problem was twofold: a reliable slurriability criteria did not exist as part of the coal selection process; and production of CWFs with high solids loading level could create significant problems in control of rheological and stability characteristics of different CWF batches.

Efforts to identify any coals, other than the Splash Dam, that completely met the initial targets for this program, were unsuccessful. Therefore, the following changes in the program were made:

1. Relaxation of the CWF solids loading requirement to 65 percent. This permitted evaluation of the Lower Kittanning Coal in CWF.
2. If the solids loading cannot be relaxed, then the washability requirements must be reevaluated. If two percent ash coals need not be produced, the field of prospective coals is greatly enhanced.

Based upon experience to date, other conclusions can be made as to the state of CWF technology.

1. Development of specifications and test methods will entail collection of test data from a large number of CWFs. The tests utilized in this program are useful, and vendors have developed their own quality control methods that are considered part of their technology. Many vendors will hold shipment if the CWF does not meet their private specifications.

Table XXVI

GENERALIZED CANDIDATE COAL RATINGS
FROM WASHABILITY EVALUATION

COAL ID	WASHABILITY		COMBUSTION	CWF MANUFACTURING	COMMENTS
	7% ASH	2% ASH			
Cedar Grove	Marginal	Good	Good	Good	Low ROM Ash; selected for testing
Lower Elkhorn	Good	Good	Moderate	Marginal	Questionable Slurring Cap.
Black Creek	Marginal	Good	NA	Marginal	Low ROM Ash
Splash Dam	Good	Moderate	Good	Excellent	Selected for Reference CWF Coal
Lower Kitt.	Good	Marginal	Good	Marginal	Selected for testing
W. KY #11	Good	Formidable	NA	NA	Did not meet targets
Pittsburgh #8	Good	Formidable	NA	NA	Did not meet targets
Upper Freeport	Good	Formidable	NA	NA	Later selected for testing

2. Vendors ordinarily perform development work prior to undertaking CWF production with a new coal, because formulations are somewhat coal specific. This is similar to what is found in the refining industry, where changes in crude oil make adjustments in operating conditions necessary.
3. In some cases, "specifications" should be regarded as "desired properties," the most important of which are viscosity and stability.
4. The bench-scale tests utilized in this program represent an initial contribution to the eventual ability to predict transportation, storage, pumping and combustion behavior of CWF.
5. Transportation of CWF by truck over long distances can be done with no deleterious effects.
6. All CWFs procured were of usable quality for the combustion tests.

Considering the fact that CWF fuels were relatively new, it was not too surprising that properties varied with coal selection. This has improved with further developments in the technology.

- END -

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