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CoALiquid, INC.

ULTRASONIC ASH/PYRITE LIBERATION

FINAL TECHNICAL REPORT

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EXECUTIVE SUMMARY

Laboratory tests were conducted to investigate the effect ultrasonic pretreatment of coal has upon beneficiation. Ultrasonics "preconditions" coal prior to conventional or advanced physical beneficiation processes to enhance ash and pyrite liberation and improve combustible recovery.

Samples of coarse (28 mesh x 0), fines (200 mesh X 0), and micronized (325 mesh x 0) fractions of Illinois #6, Pittsburgh #8, and Upper Freeport coal were subjected to an ultrasonic frequency of 20 kHz, and then processed by conventional froth flotation. The fines fractions were further subjected to microbubble flotation. Comparative analyses of the beneficiation processes both with and without ultrasonic preconditioning were evaluated.

For all coals, ultrasonic pretreatment enhanced ash and pyrite liberation, and in most cases improved combustible recoveries. These results are attributable to the sonochemical and fragmentation effects caused by cavitation.

Samples of the Pittsburgh #8 and the Upper Freeport fines fraction (200 mesh X 0) were further subjected to various power levels of ultrasonic energy to study the process economics vis-a-vis combustible recovery, yield and concentrate ash. Ultrasonic pretreatment of the Upper Freeport coal proved to be more economical than the Pittsburgh #8.

The economic study suggests coals may be preconditioned with relatively low power usage, and therefore at a justifiable lower cost, while attaining increased yields and combustible recovery while lowering concentrate ashes.

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INTRODUCTION

The objective of this project was to develop a coal preparation concept which employed ultrasonics to precondition coal prior to conventional or advanced physical beneficiation processes such that ash and pyrite separation were enhanced with improved combustible recovery .

Experimental research activities involved a series of experiments that subjected three different test coals, Illinois #6, Pittsburgh #8, and Upper Freeport, ground to three different size fractions (28 mesh X 0, 200 mesh X 0, and 325 mesh X 0), to a fixed (20 kHz) frequency ultrasonic signal prior to processing by conventional and microbubble flotation. These same samples of coal were also processed by conventional and microbubble flotation without ultrasonic pretreatment to establish baseline conditions. The test matrix followed for each coal species is found in Figures 1 and 2.

Product ash, sulfur and combustible recovery data were determined for both beneficiation processes. Comparative analysis between the two series of experiments provided information about the overall effectiveness of each beneficiation process studied relative to the applied effects of ultrasonic preconditioning.

FIGURE 1

ILLINOIS #6 TEST MATRIX

FIGURE 2

PITTSBURGH #8 AND UPPER FREEPORT TEST MATRIX

BACKGROUND

Froth flotation has long been recognized as the most practical and cost effective method for cleaning -28 mesh coal. However, this size fraction of coal often contains ultrafine materials such as clay that may be carried into the froth product. As a result, low recovery and high product ash contents often occur when flotation is used to process such coal.

In order to overcome some of the deficiencies of the froth flotation process, a pre-beneficiation step, ultrasonic pretreatment, may be used. Ultrasound, applied at an optimum frequency, liberates ash and pyrites from the coal matrix that can be then removed via froth flotation.

Ultrasonics is widely used industrially to enhance chemical effects, emulsify immiscible liquids, degass and grind, to name a few(4,16,22). It is propagated through any medium (vapor, gas, liquid or solid), though most optimally transferred through a liquid(8). Crystal oscillators and magnetostrictive transducers are commonly used to generate the required sonic waves, with the latest technology involving the use of a new transducer material, Terfenol-D. Ultrasound may be utilized at varying frequencies depending upon the application.

Ultrasonics has found diverse application in many industries due to its simple construction, low set-up cost and low power consumption (compared with conventional methods). It is also possible to operate at lower pressures, temperatures, and for shorter times than previously required.

Ultrasound consists of oscillations which are propagated through an elastic medium with a supersonic frequency - exceeding 20 kHz. The frequency of a sound wave is characterized by a definite wavelength wherein there exists a dependence between sound propagation velocity, wavelength, and frequency(8).

Sound waves may either be transmitted continuously or pulsed depending upon the application. Of the two, pulsed is the more intense. Heuter & Bolt determined that there is a time delay between the application of sonics and cavitation, and the longer a liquid is exposed to intense sound, the lower the threshold pressure required for cavitation(31). Pulses for an extended period of time have been observed to be as effective as continuous. Additionally, an optimum time interval exists between pulses, which if not determined, will result in little or no effect(30). Currently, pulsed sound waves are used mainly in the medical field and for flaw detection.

The most promising practical uses of ultrasonics involves the mechanism of cavitation(31). Interest in cavitation may be traced as far back as 1865 where it was noted mostly for its damaging affects on ship propellers(38). Bubble formation, or cavitation, is also responsible for the condition of nitrogen narcosis which affects scuba divers.

When a liquid is subjected to rapidly alternating pressures of high amplitudes, cavitation results. Therefore, when high energy sound waves are applied to a liquid, cavitation occurs. Acoustically generated cavitation by sonics was first observed in the 1920's and is one mechanism by which ultrasound affects change in liquids and solids(12).

The onset of cavitation is influenced by temperature, pressure, viscosity, surface tension, number of bubble nuclei present, frequency and intensity of the sound wave, cohesive strength of the liquid, density of the liquid, and attenuation characteristics of bubbles.(22) The amount of energy released is specifically dependent upon the kinetics of growth and collapse of the bubbles, and the maximum radius a bubble achieves is dependent upon the vapor pressure of the liquid(31). Very fine non-wettable particles, such as coal, within a medium may have microfissures containing gas which can promote the origination of cavitation(8).

The quantity of energy available increases with the surface tension of the bubble and decreases with the vapor pressure of the liquid(12). Also, the pitting action of sonics increases with surface tension(31). Suslick, et al similarly observed in their research that as solvent volatility diminishes, the intensity of collapse, maximum temperature attained and reaction rate all increase(49). Optimum cavitation conditions differ for each application and are dependent upon the end results desired.

When the cavitation threshold is reached, that is, when ideal conditions for cavitation are present, a gaseous or vaporous cavity within the liquid or solid is excited to vibration in its natural elastic period and the bubble increases in size. This creates a reduction in the interior pressure(12). In addition, the pressure at the cavity wall boundary is increased due to the rarefaction and compression of the sonicated fluid until eventually, implosion occurs(8). At this instant, pressure of thousands of atmospheres and localized temperatures of hundreds of degrees occur(59).

This implosion produces microjets of liquid which are forced into a solid or between immiscible liquids at high velocities resulting in surface erosion in solids and disruption of barrier surfaces in liquids(50).

Ultrasonic cleaning has been used extensively in those industries where surface purity is of utmost importance to the efficiency of the operation, and also where contamination could prove detrimental to the apparatus(12). Any solid material that is bonded to a particle's surface, yet is different from that surface, may be removed by the scrubbing action of cavitation(22). Ultrasonic cleaning is simple and in certain applications, reduces cost of materials and labor(27).

Oscillating bubbles which originate during cavitation also contribute to the cleaning process by penetrating into small cracks and gaps(8). At lower frequencies, cavitation is primarily the means by which cleaning occurs, at high frequencies, oscillating bubbles are the primary method of cleaning(12). The high rate of oscillation of trapped bubbles rapidly fatigues material at its bond interface causing impurities to break off(22). Ultrasonic oscillations are especially beneficial in those instances where chemicals are used within the cleaning solution to enhance the cleaning process, since hydrodynamic currents produced during sonication facilitate mixing(8).

Another area of great interest has been the effect ultrasonics has upon chemical reactions and synthesis, otherwise known as sonochemistry. Reactions have been enhanced with the application of ultrasonics, reducing the rate several hundred times(28). Also, by cavitation, the chemically

enhanced solution/solvent is more effectively transferred to a solid interface by the microjet streaming and improved mass transfer which results(45,50). Also, chemicals have been shown to facilitate the fragmentation process either by the chemical effect of swelling or the disruption by chemical agents of the natural binding forces which hold the solid together(34).

In 1982, Mraz, et al found that the application of sonics significantly enhanced the dissolution of a mineral matrix in shale in the presence of hydrochloric acid. They deduced that ultrasonics reduced the particle size so that fresh sites for chemical attack were produced(36).

Current applications of ultrasonics include the comminution of coal. Present methods of grinding are extremely energy inefficient and equipment wear and maintenance is expensive. When ultrasonics is applied directly to the coal solid, the solid is strained past its elastic limit, therefore, suffering plastic deformation with each cycle of waves until breakage occurs(12). Suslick observed that with application of sonics to solids, a significant decrease in particle diameter of moderately sized particles resulted(50). This was confirmed by comminution research utilizing ultrasound performed for the Department of Energy(1,2,3). Greater control over particle size ranges was achieved. Additionally, when compared with conventional grinding, better removal of ash and pyrites resulted. Also, ultrasound was as much as 500% less energy intensive than conventional grinding.

At Energy Minerals and Research Company, ultrasonics was used to generate standing waves inside of a pipe using reflecting transducers. A particle laden stream is passed through the pipe(28). Differing density particles are then separated into various fractions and collected. It was determined by Pugin in January of 1987, that high intensity ultrasound sources such as an ultrasonic horn, tend to uncouple; that is, cavitation occurs only at the radiating surface and only marginal sonic intensity may be detected elsewhere in the surrounding liquid. He concluded that treatment of suspended particles should be conducted in a continuous flow cell as the reaction mixture is forced into the cavitation zone(43). This could be used beneficially on coal particles to separate out impurities. Energy Minerals and Research is also developing a process whereby ultrasonics aids in desulfurization. Results were extremely successful with process time decreasing a hundred-fold.

Suslick, et al, reported favorable results with ultrasonics and inorganic solids emphasizing fragmentation due to cavitation. They concluded that ultrasound increased dramatically the rate of intercalation of a variety of inorganic and organic solids and organometallic compounds. Reactivity of transition metals was also greatly enhanced. Optical viewing of the organic and inorganic solids showed badly eroded surfaces. Alternately, transition metals exhibited smoothing of the surface probably due to the malleable properties. They also deduced that shock waves produced during sonication resulted in fracturing of friable solids(50).

Similarly, Cook and Gaikwad applied ultrasonics during coal extraction and reported reduced ash within the extract, increased yield and a decrease in

particle size(19). Operating at a frequency of 60 kHz, sonication time was varied from 8 to 25 hours. Their experimentation also showed that rate of extraction and yield increased with an increase in temperature. Additionally, with a decrease in particle size, the yield was also increased suggesting that greater surface area is available for sonic irradiation.

Several patents exist documenting processes utilizing ultrasonics to remove ash and pyrites from coal water slurries, stabilizing COM for better burning, comminuting solids and wet grinding coal(20,54).

Currently, in Ames, Iowa, research is being performed with coal utilizing sonic frequencies in the audible range to improve liberation. This research has indicated that slight ash reduction results with a slight improvement in yield(13,14,15).

At West Virginia University, utilizing a batch process, ultrasonics has been shown to promote cavitation in a coal slurry, and thus improve flotation by the liberation of ash and pyrite from the coal particles. This increases the hydrophilicity of the impurities and sharpens flotation separation(47).

The proposed technology for this contract is based upon the fact that any substance has a natural frequency of vibration. When an external vibrating force of a certain amplitude reaches the resonant frequency of that substance, it causes the substance to resonate. The resonance, under certain circumstances, creates a force of sufficient magnitude to fracture

the substance itself. When coal particles are dispersed in water and subjected to an ultrasonic field, at a certain frequency, mineral matter in the coal matrix will resonate to an extent such that resonance results in physically fracturing mineral matter away from the coal particle.

Also, the cavitation that occurs when a liquid is subjected to ultrasonics produces surface erosion through bubble implosion, and oscillating bubbles which originate during cavitation contribute to the cleaning process by penetrating into small cracks and gaps, fracturing off impurities.

Finally, energy is dissipated at interfaces of small gas bubbles or suspended particulate matter that act as nuclei for high pressure gradients. The cavitation mechanism makes energy available at the interface of a solid particle suspended in liquid, and this energy is used to implant chemical reagents on the surface of the particle, thereby improving flotation.

EXPERIMENTATION

TEST COAL PREPARATION

The test coals selected for experimentation were Illinois #6, Pittsburgh #8, and Upper Freeport. These were chosen due to their widespread use and long-term availability. All samples were fresh run of mine.

Illinois #6 was obtained from Orient #6 mine located in Waltonville, Illinois at the Freeman United Coal Company. Upper Freeport was acquired from Limestone Pit mine located in Cadiz, Ohio at the Cravat Mine Coal Company. The Pittsburgh #8 sample, supplied by the Department of Energy, came from the Bruceton Mine in Pittsburgh, Pennsylvania.

Bulk samples of each coal were processed using a hammer-mill and classified into coarse (28 x 0 mesh) and fines (200 mesh X 0) fractions. After grinding, the resultant coal samples were purged with nitrogen to prevent oxidation. Figure 3 illustrates the grinding process in detail.

Some of the fines were further processed in a ball mill to produce micronized coal (325 x 0 mesh) and used immediately.

PROCEDURES

Figure 4 describes the procedure followed for all conventional flotation experiments. A Denver D-12 flotation machine with a 2.5 liter stainless steel cell was used for all tests. In each experiment, 100 grams of coal

SAMPLE PREPARATION PROCEDURE

FIGURE 3
12

FIGURE 4
FLowsheet of conventional flotation procedure

was slurried in water for one minute. Next, a collector (#2 fuel oil) was added and dispersed for one minute. At this point, either an ultrasonic enhancer was added and dispersed for another minute or the entire system was dispersed for three minutes. In experiments that required ultrasonic preconditioning, the sample was subjected to continuous ultrasonics for 10 minutes. The frother, MIBC (Methyl Isobutyl Carbinol), was then added to the cell and dispersed for one minute. In all conventional tests, a three minute product collection time was used. Samples of the product and reject were collected and analyzed.

Figure 5 illustrates the microbubble flotation process. Coal and water were slurried in a 55 gallon mix tank for five minutes. Next, the collector was added and mixed for 3 minutes. The frother was added and allowed to mix an additional 3 minutes. If an ultrasonic enhancer was used, it was added last and the system was allowed to mix three more minutes. If ultrasonic pretreatment was required, the slurry was passed through the ultrasonic chamber. A back pressure of 30 psi was maintained in the system to further enhance the ultrasonic transference. The slurry was then fed to the microbubble column flotation unit. After reaching steady state, samples of the product and reject were collected and analyzed.

BASELINE ANALYSES - TEST COALS

Table 1 shows complete analysis of a head sample of each of the test coals. All analyses were performed according to ASTM procedures at the University

FIGURE 5

MICROBUBBLE FLOTATION FLOW DIAGRAM

TABLE 1

PROXIMATE ANALYSES   ULTIMATE ANALYSES*

Coal ID	Ash Content (%)	Volatiles (%)	Moisture Content (%)	Fixed Carbon (%)	Higher Heating Value (BTU/Lb)	Carbon	Hydrogen	Nitrogen	Oxygen
Illinois #6	29.50	30.03	7.57	32.90	8,389	61.25	5.19	1.27	14.32
Pittsburgh #8	5.19	25.98	2.44	66.39	13,530	79.09	4.39	1.30	7.44
Upper Freeport	16.39	29.90	5.54	48.17	10,706	69.17	4.63	1.50	5.36

FORMS OF SULFUR*

Coal Identification	Pyritic (%)	Sulfate (%)	Organic (%)
Illinois #6	1.11	0.10	0.93
Pittsburgh #8	0.39	0.04	0.79
Upper Freeport	1.75	0.04	0.78

All sample analyses were performed by the University of Kentucky Energy Research Laboratory in Lexington, Kentucky.

*Reported on a dry, ash-free basis.

of Kentucky Energy Cabinet Research Laboratory. As is evident from this information, these coals represent wide impurity level variations.

Tables 2 through 4 show the washability analyses for each test coal. Tables 5 and 6 present the washability data for Pittsburgh #8 and Upper Freeport coals on a composite basis. This was performed to determine the potential recovery and to investigate cleaning characteristics of each coal. It appears from the data that for all test coals, the ash is finely disseminated in the coal. However, pyrites appear to be liberated as shown by low values obtained for the float fractions.

For each of the three test coals, a petrographic analysis of a head sample was performed. Two hundred and fifty grain counts were made for each of the samples analyzed. Table 7 shows the particle size distribution range for both free and included pyrites for each coal. For all test coals, the majority of pyrites (pyritic sulfur) exist in the less than 5 micron range. For Illinois #6 and Upper Freeport coals, the majority are free or not attached to the coal particle. For Pittsburgh #8, the pyrites are included, or attached to the coal particle. This information will be used as baseline pyrite data for comparison with the clean coal product data obtained after ultrasonic pretreatment.

OVERVIEW

For each coal, baseline experimental conditions were determined utilizing fuel oil or frother dosage curves. Table 8 shows the dosages of #2 fuel oil (collector) and MIBC (frother) used for each test coal. Each of the

TABLE 2
WASHABILITY
ILLINOIS #6

(28 MESH X 200)

SPECIFIC GRAVITY	WEIGHT (%)	WEIGHT (%) , CUM.	ASH CONTENT (%)	ASH CONTENT (%) , CUM.	SULFUR CONTENT (%)	SULFUR CONTENT (%) , CUM.	PYRITIC SULFUR CONTENT (%)	PYRITIC SULFUR CONTENT (%) , CUM.
FLOAT 1.40	46.8	46.8	9.0	9.0	1.72	1.72	0.57	0.57
1.40 X 1.50	23.9	70.7	15.1	15.1	1.85	1.76	0.78	0.64
1.50 X 1.60	7.5	78.2	24.6	24.6	2.10	1.79	1.08	0.68
1.60 X 1.70	1.7	79.9	33.3	33.3	2.09	1.80	1.38	0.70
SINK 1.70	20.1	100.0	72.3	72.3	4.36	2.31	3.90	1.34

(200 MESH X 0)

SPECIFIC GRAVITY	WEIGHT (%)	WEIGHT (%) , CUM.	ASH CONTENT (%)	ASH CONTENT (%) , CUM.	SULFUR CONTENT (%)	SULFUR CONTENT (%) , CUM.	PYRITIC SULFUR CONTENT (%)	PYRITIC SULFUR CONTENT (%) , CUM.
FLOAT 1.50	36.6	36.6	11.6	11.6	1.44	1.44	0.34	0.34
1.50 X 1.60	7.5	44.1	15.4	15.4	1.40	1.43	0.46	0.36
1.60 X 1.70	4.7	48.8	27.9	27.9	1.59	1.44	1.70	0.49
SINK 1.70	51.2	100.0	75.2	75.2	3.24	2.36	2.60	1.57

All analyses were performed at the University of Kentucky Center for Applied Energy Research.

TABLE 3
WASHABILITY
UPPER FREEPORT

(28 MESH X 200)

SPECIFIC GRAVITY	WEIGHT (%)	WEIGHT (%) , CUM.	ASH CONTENT (%)	ASH CONTENT (%) , CUM.	SULFUR CONTENT (%)	SULFUR CONTENT (%) , CUM.	PYRITIC SULFUR CONTENT (%)	PYRITIC SULFUR CONTENT (%) , CUM.
FLOAT 1.30	33.3	33.3	2.0	2.0	0.56	0.56	0.07	0.07
1.30 X 1.40	40.5	73.8	5.7	4.0	1.11	0.86	0.33	0.21
1.40 X 1.50	9.2	83.0	16.1	5.4	2.70	1.06	1.40	0.34
1.50 X 1.60	4.8	87.8	22.3	6.3	4.17	1.24	2.45	0.46
1.60 X 1.70	2.4	90.2	30.2	6.9	4.95	1.33	3.25	0.53
SINK 1.70	9.8	100.0	64.3	12.6	7.90	1.98	6.06	1.07

(200 MESH X 0)

SPECIFIC GRAVITY	WEIGHT (%)	WEIGHT (%) , CUM.	ASH CONTENT (%)	ASH CONTENT (%) , CUM.	SULFUR CONTENT (%)	SULFUR CONTENT (%) , CUM.	PYRITIC SULFUR CONTENT (%)	PYRITIC SULFUR CONTENT (%) , CUM.
FLOAT 1.30	0.1	0.1	-	-	2.15	-	-	-
1.30 X 1.40	5.3	5.4	10.50	10.30	1.70	1.67	0.75	0.74
1.40 X 1.50	60.1	65.5	13.30	13.05	1.91	1.89	0.93	0.91
1.50 X 1.60	18.2	83.7	20.50	14.67	2.64	2.05	1.68	1.08
SINK 1.60	16.3	100.0	43.20	19.30	10.65	3.45	7.30	2.09

All analyses were performed at the University of Kentucky Center for Applied Energy Research.

TABLE 4
WASHABILITY
PITTSBURGH #8

(28 MESH X 200)

SPECIFIC GRAVITY	WEIGHT (%)	WEIGHT (%) , CUM.	ASH CONTENT (%)	ASH CONTENT (%) , CUM.	SULFUR CONTENT (%)	SULFUR CONTENT (%) , CUM.	PYRITIC SULFUR CONTENT (%)	PYRITIC SULFUR CONTENT (%) , CUM.
Float 1.30	64.5	64.5	1.9	1.9	0.90	0.90	0.10	0.10
1.30 X 1.40	27.8	92.3	7.2	3.5	1.30	1.02	0.30	0.16
1.40 X 1.50	2.9	95.2	16.6	3.9	2.10	1.05	0.90	0.18
1.50 X 1.60	1.9	97.1	21.8	4.2	2.40	1.08	1.30	0.20
SINK 1.60	2.9	100.0	55.2	5.7	7.40	1.26	5.30	0.35

(200 MESH X 0)

SPECIFIC GRAVITY	WEIGHT (%)	WEIGHT (%) , CUM.	ASH CONTENT (%)	ASH CONTENT (%) , CUM.	SULFUR CONTENT (%)	SULFUR CONTENT (%) , CUM.	PYRITIC SULFUR CONTENT (%)	PYRITIC SULFUR CONTENT (%) , CUM.
Float 1.30	25.7	25.7	2.9	2.9	1.00	1.00	0.11	0.11
1.30 X 1.40	67.8	93.5	4.6	4.1	1.04	1.03	0.17	0.15
1.40 X 1.50	2.8	96.3	9.3	4.3	1.52	1.04	0.46	0.16
1.50 X 1.60	2.3	98.6	21.8	4.7	2.36	1.07	1.30	0.19
SINK 1.60	1.4	100.0	53.9	5.4	9.58	1.21	-	-

All analyses were performed at the University of Kentucky Center for Applied Energy Research.

TABLE 5
WASHABILITY
UPPER FREEPORT

(28 MESH X 0)

SPECIFIC GRAVITY	WEIGHT (%)	WEIGHT (%) , CUM.	ASH CONTENT (%)	ASH CONTENT (%) , CUM.	SULFUR CONTENT (%)	SULFUR CONTENT (%) , CUM.	PYRITIC SULFUR CONTENT (%)	PYRITIC SULFUR CONTENT (%) , CUM.
FLOAT 1.30	25.28	25.3	3.6	3.6	0.56	0.56	0.07	0.07
1.30 X 1.40	32.02	57.3	13.4	9.1	1.13	0.88	0.35	0.23
1.40 X 1.50	21.61	78.9	21.7	12.6	2.16	1.23	1.08	0.46
1.50 X 1.60	8.07	86.9	12.2	12.5	3.32	1.42	2.03	0.61
1.60 X 1.70	5.73	92.7	15.8	12.7	8.86	1.88	6.04	0.94
SINK 1.70	7.29	100.0	33.2	14.2	7.39	2.32	6.07	1.31

All analyses were performed at the University of Kentucky Center for Applied Energy Research.

TABLE 6
WASHABILITY
PITTSBURGH #8

(28 MESH X 0)

SPECIFIC GRAVITY	WEIGHT (%)	WEIGHT (%) , CUM.	ASH CONTENT (%)	ASH CONTENT (%) , CUM.	SULFUR CONTENT (%)	SULFUR CONTENT (%) , CUM.	PYRITIC SULFUR CONTENT (%)	PYRITIC SULFUR CONTENT (%) , CUM.
FLOAT 1.30	50.1	50.1	2.1	2.1	0.92	0.92	0.07	0.07
1.30 X 1.40	42.7	92.8	5.7	3.7	1.16	1.03	0.22	0.14
1.40 X 1.50	2.9	95.8	13.9	4.0	1.82	1.05	0.72	0.16
1.50 X 1.60	1.9	97.6	21.7	4.4	2.29	1.08	1.30	0.18
SINK 1.60	2.4	100.0	54.9	5.6	7.89	1.24	4.10	0.27

All analyses were performed at the University of Kentucky Center for Applied Energy Research.

TABLE 7
PETROGRAPHIC ANALYSIS

Sample	Pyrites	< 5 micron	5-10 micron	10-25 micron	> 25 micron
Illinois 6	Free	49.6	5.6	0.8	0
	Included	38.8	5.2	0.4	0
Pittsburgh 8	Free	43.2	1.6	0.8	0
	Included	50.4	3.0	1.0	0
Upper Freeport	Free	54.8	5.6	0.6	0
	Included	37.8	1.0	0.2	0

All analyses were performed at the University of Kentucky Center for Applied Energy Research

TABLE 8
FROTHER AND COLLECTOR DOSAGES FOR THE
TEST COALS

Coal Identification	Frother Conc. MIBC (lbs/ton of coal)	Collector Conc. #2 Fuel Oil (lbs/ton of coal)
Illinois #6	2.00	5.0
Pittsburgh #8	0.67	0.5
Upper Freeport	1.30 *	2.5 *
	2.00 **	5.0 **

* Coarse (28 mesh X 0) and Fines (200 mesh X 0) fractions
 ** Micronized fraction (325 mesh X 0)

test coals followed the test matrix for experimentation previously mentioned. Results from each test were evaluated based upon the following criterion:

1. Clean-Coal-Ash
2. Clean-Coal-Pyritic Sulfur
3. Combustible Recovery
4. Clean-Coal Yield
5. Ash-Separation Index
6. Pyritic Sulfur-Separation Index (Select Samples)
7. Ash Rejection
8. Pyritic Sulfur Rejection

Formulae used for these performance criterion are found in Appendix A.

RESULTS AND DISCUSSION

ILLINOIS #6

For each size fraction of Illinois #6 coal (28 mesh X 0, 200 mesh X 0 and 325 mesh X 0), the optimum frother (MIBC) and collector (#2 fuel oil) dosages were 2.0 and 5.0 lbs/ton of coal, respectively. Two additional frothers, 2-Ethyl Hexanol and Shur Coal 168 A (Sherex Chemical Company) were also tested to determine if any enhancements in bubble formation were noted, and thus increased rates of flotation. Kerosene was tried as an alternative collector to increase recovery and yields. From experimental data, it was determined that MIBC and #2 fuel oil produced the lowest

product-ash and pyritic sulfur contents, and highest combustible recoveries and yields.

Each of the size fractions were floated using conventional and/or column flotation without ultrasonics to establish baseline conditions. From this data, it was possible not only to compare beneficiation processes, but also to establish ash and sulfur-separation indices for comparison with ultrasonic pretreatment. Based upon initial test findings, only two size fractions (28 mesh X 0 and 200 mesh X 0) were chosen for conventional flotation after ultrasonic pretreatment. Since only fines (200 mesh X 0) were used in microbubble flotation initially, only fines were used for microbubble flotation in conjunction with ultrasonic pretreatment.

As stated previously, in order for cavitation to occur, optimum operating conditions within the slurry must be present. For Illinois #6, a myriad of modifying reagents were employed in order to alter intrinsic variables of the slurry, and thereby promote cavitation. These were chosen not only to alter these variables, but also to facilitate the removal of any impurities scrubbed off by cavitation. A list of the additives used and comments regarding their effects are found in Table 9.

Since it was important economically to quantify solely the effect which ultrasonics had upon coal beneficiation, future work with the other test coals involved only the addition of a frother and collector (MIBC and #2 fuel oil, respectively), with the addition of the "ultrasonic enhancer" in selected experiments, for comparison.

TABLE 9

CHEMICAL ID	PURPOSE	RESULTS** - AFTER ULTRASONIC PRETREATMENT
1. Calgon	Decrease Surface Tension	Increased Product Ash and Pyrite Content. No Effect Upon Combustible Recovery.
2. Ethylene Glycol	Improve Froth Resiliancy	Increased Product Ash Content. No Effect Upon Pyrites. Increased Combustible Recov.
3. Ultrasonic Enhancer*	Known Cavitation Promoter	Enhanced Product Ash and Pyrite Reduction. Increased Combustible Recovery.
4. Cupric Sulfate	Improve Sulfur Reduction	Enhanced Ash and Pyrite Reduction. Decreased Combustible Recovery.
5. Hydrogen Peroxide	Improve Sulfur Reduction	Enhanced Pyrite Reduction. No Effect Upon Product Ash or Combustible Recovery.
6. Dispersant A23K	Increase Vapor Pressure	Enhanced Ash Reduction. No Effect Upon Pyrites. Decreased Combustible Recovery.
7. Lipocol	Decrease Surface Tension	Enhanced Ash and Pyrite Reduction. Decreased Combustible Recovery.
8. PX 944	Decrease Surface Tension	Increased Product Ash and Pyrite Content. No Effect Upon Combustible Recovery.
9. Ammonium Hydroxide	Increase pH	Improved Combustible Recovery. Increased Product Ash and Sulfur Content.
10. Durkee Durem 300 K	Decrease Surface Tension	Enhanced Ash Reduction. Improved Comb. Recovery. No Effect Upon Pyrites.
11. Durkex 100	Decrease Surface Tension	Increased Product Ash Content. No Effect Upon Pyrites or Combustible Recovery.
12. Alum	Agglomerate Impurities, Reject Pyrites	Increased Product Ash Content. Enhanced Pyrite Rejection. Decreased Combustible Recovery.

*Proprietary.

Coarse (28 mesh X 0)

This size fraction was floated using conventional flotation only. From results shown in Table 10, when conventional flotation was used, only a moderate yield and combustible recovery were realized, and there was no reduction in pyritic sulfur. However, there was a 68% reduction in ash content when compared with feed coal data.

After pretreatment of the coal with ultrasonics at a fixed frequency of 20 kHz, Table 11 shows that there was a significant increase in combustible recovery (24%), the ash-separation index improved 105 points, and ash rejection improved 31% when compared with baseline conditions. The improvement in combustible recovery could be attributed to the microjet streaming ("jets of high pressure liquid spewed out when a bubble collapses unsymmetrically") (p. 23, (28)) that occurs during cavitation which better implants the collector upon the coal particle surface, and thus improves flotation of clean coal.

Fines (200 Mesh X 0)

For this size fraction, when conventional flotation was used, Table 10 again shows only a moderate yield and combustible recovery. The fines fraction had a much lower pyritic sulfur content after conventional flotation than the coarse fraction.

TABLE 10

BASELINE CONDITIONS - Illinois #6

No Ultrasonic Pretreatment

Flotation Process	Size Fraction*	Product, Ash (%)	Product, Pyritic Sulfur (%)	Combustible Recovery (%)	Yield (%)	Ash Separation Index	Pyrite Separation Index	Ash Rejection (%)	Pyrite Rejection (%)
Conventional Flotation	Coarse	9.58	1.09	70.50	68.40	435	**	65	**
	Fines	10.91	0.78	82.97	65.60	392	288	76	106
	Micronized	9.89	0.92	29.26	16.50	63	**	**	**
Microbubble Flotation	Fines	7.29	0.90	89.50	74.99	929	**	71	**

* Coarse - (28 Mesh X 0)

Fines - (200 Mesh X 0)

Micronized - (325 Mesh X 0)

** No data available for this size fraction.

Reagent Dosages: 2.0 lbs MIBC/Ton of Coal

5.0 lbs #2 Fuel Oil/Ton of Coal

Ultrasonic Frequency: 0 KHz

TABLE 11

Illinois #6

After Ultrasonic Pretreatment

Flotation Process	Size Fraction*	Product, Ash (%)	Product, Pyritic Sulfur (%)	Combustible Recovery (%)	Yield (%)	Ash Separation Index	Pyrite Separation Index	Ash Rejection (%)	Pyrite Rejection (%)
Conventional Flotation	Coarse	9.77	1.13	87.42	67.82	540	**	85	**
	Fines	7.14	0.65	90.34	70.01	803	952	83	239
Microbubble Flotation	Fines	5.32	1.05	95.41	78.20	1199	**	63	**

* Coarse - (28 Mesh X 0)

Fines - (200 Mesh X 0)

Micronized - (325 Mesh X 0)

** No data available for this size fraction.

Reagent Dosages: 2.0 lbs MIBC/Ton of Coal
 5.0 lbs #2 Fuel Oil/Ton of Coal

Ultrasonic Frequency: 20 KHz

When microbubble flotation was used, the product-ash content decreased significantly (33%), combustible recovery increased 8%, and yield 14% when compared with conventional flotation results.

As seen in Table 11, for conventional flotation, ultrasonic pretreatment of this fraction significantly improved product quality as is evidenced by a lower product-ash content (35%) and lower pyritic sulfur content (17%) when compared with similar tests which did not receive ultrasonic pretreatment. Also, combustible recovery improved 9% and yield 7%. Ash-separation index increased 411 points, ash rejection improved 9.2%, and the pyritic sulfur-separation index increased 665 points. For the pyrite rejection formula, low yields in conjunction with moderate refuse pyrite levels mathematically produced pyrite rejection data greater than 100%

In order to verify that ultrasonics was indeed fracturing particles, a particle size analysis of an 80%, -200 mesh coal sample was performed both before and after receiving ultrasonic pretreatment. It is obvious from Table 12 that when ultrasonics was used, there was an increase in the number of particles at the smallest micron ranges, as evidenced by the decrease in mass mean diameter.

Additionally, in order to quantify the effect ultrasonics had upon pyritic sulfur, a petrographic analysis was performed on a sample that had received ultrasonic pretreatment. Table 13 lists both free (unattached) and included (attached) pyrites for this sample as well as a sample which had not received ultrasonic pretreatment. This verifies that ultrasonics

TABLE 12
PARTICLE SIZE ANALYSIS

Illinois #6

NO ULTRASONICS

Mesh Size	Micron Size	Percent Cumulative Below	Percent Between Band Sizes
28	595	100.0	0.0
50	300	100.0	0.0
	212	100.0	0.0
100	150	100.0	0.8
150	106	99.2	6.1
200	75	93.0	14.7
270	53	78.3	17.0
400	38	61.2	15.2
	27	45.9	12.8
	19	33.1	9.8
	13	23.2	7.6
	9.4	15.5	6.6
	6.6	8.8	4.5
	4.7	4.3	4.3

Mass Mean Diameter = 29.91 Microns

AFTER ULTRASONIC PRETREATMENT - 20 KHz

Mesh Size	Micron Size	Percent Cumulative Below	Percent Between Band Sizes
28	595	100.0	0.0
50	300	100.0	0.0
	212	100.0	0.0
100	150	100.0	0.0
150	106	100.0	0.0
200	75	100.0	7.1
270	53	92.8	15.7
400	38	77.1	15.8
	27	61.2	13.2
	19	47.9	12.6
	13	35.3	10.5
	9.4	24.7	9.4
	6.6	15.3	7.3
	4.7	8.0	8.0

Mass Mean Diameter = 20.21 Microns

Analyses were performed using Microtrac Particle Size Analyzer.

TABLE 13

**PETROGRAPHIC ANALYSIS OF COAL SAMPLES
WITH AND WITHOUT ULTRASONIC PRETREATMENT**

Illinois #6

Sample	Pyrites*	< 5 micron	5-10 micron	10-25 micron	> 25 micron
Without Ultrasonic Pretreatment	Free	36.8	4.0	2.4	0
	Included	52.4	2.8	1.6	0
After Ultrasonic Pretreatment	Free	44.4	3.6	0	0
	Included	48.4	3.2	0	0

* Free refers to pyrites which are not attached to coal particles.
Included refers to pyrites which are attached to coal particles.

enhances liberation evidenced by the large number of finer "free" pyrite particles shown in the less than 5 micron range. No pyrite particles are shown above the 10 micron range as compared with the same range of the sample which did not receive pretreatment with ultrasonics.

Conversely, there are less included pyrite particles shown on the preconditioned sample as compared with the non-preconditioned sample which also indicates an enhanced liberation effect.

A scanning electron microscope (SEM) analysis of representative feed and product samples verified that ultrasonic preconditioning significantly reduced particle size. As seen in Exhibit 1, at a magnification of 180 X, the sonicated sample had a much smaller particle size than the feed sample.

Exhibit 2 shows a feed sample prior to cleaning, a product sample after microbubble flotation, and a product sample after microbubble flotation with ultrasonic precleaning. First, it is obvious that there is a decrease in particle size in the sample that received ultrasonic pretreatment. Secondly, there is a greater quantity of attached mineral matter on the sample which was cleaned using only microbubble flotation.

In order to verify the identity of the attached particles, an SEM particle analysis was performed. Exhibit 3 shows the analysis of particle "c" on the feed sample. This analysis shows that the attached particles contain a high percentage of silicates, sulfur and aluminum, all impurities normally associated with coal. Also shown in this exhibit is a coal sample, at the same magnification, after ultrasonic pretreatment and microbubble

EXHIBIT 1

ILLINOIS #6 COAL

180x

COAL SAMPLE (200 MESH X 0) PRIOR TO ULTRASONIC TREATMENT

180x

COAL SAMPLE (200 MESH X 0) AFTER ULTRASONIC PRETREATMENT

EXHIBIT 2
ILLINOIS #6 COAL
(200 MESH X 0)

PICTURE IDENTIFICATION
LEFT - Feed Sample   LOWER LEFT - Product Sample After Microbubble Flotation   LOWER RIGHT - Product Sample After Ultrasonic Pretreatment and Microbubble Flotation

Note: All are at a magnification of 500 X.

500x

EXHIBIT 3
ILLINOIS #6 COAL
(200 MESH X 0)

PICTURE IDENTIFICATION	
LEFT - Feed Sample	LOWER LEFT - Product Sample After Ultrasonic Pretreatment and Microbubble Flotation LOWER RIGHT - Scanning Electron Microscope (SEM) Particle Analysis

**Note: All are at a magnification
of 750 X.**

Particle C

flotation. Note the small amount of attached impurities on these coal particles.

Micronized (325 mesh X 0)

For the micronized coal, using conventional flotation, low combustible recovery and yield were reported, as seen in Table 10, due to the small particle size and inefficiency of conventional flotation with this size fraction. No further flotation was performed with this size fraction.

PITTSBURGH #8

For each size fraction of this coal (28 mesh X 0, 200 mesh X 0, and 325 mesh X 0), a frother dosage curve was generated. Optimum reagent dosages were selected and used for all experimentation. The fuel oil and MIBC dosage levels for these fractions were chosen to obtain maximum combustible recovery and low product-ash levels.

Coarse (28 mesh X 0)

The fuel oil and frother dosages for this fraction were 0.5 lbs/ton of coal and 0.67 lbs/ton of coal, respectively. From results shown in Table 14, conventional flotation produced excellent combustible recovery and yield data. When compared with the feed coal data, after conventional flotation, the product-ash content decreased by 16% and the pyritic sulfur by 21%.

TABLE 14
BASELINE CONDITIONS - Pittsburgh #8

No Ultrasonic Pretreatment

Flotation Process	Size Fraction*	Product, Ash (%)	Product, Pyritic Sulfur (%)	Combustible Recovery (%)	Yield (%)	Ash Separation Index	Pyrite Separation Index	Ash Rejection (%)	Pyrite Rejection (%)
Conventional Flotation	Coarse	4.35	0.31	93.18	92.37	358	343	25	22
	Fines	3.90	0.39	94.77	97.93	653	638	10	13
	Micronized	2.86	0.14	99.00	97.76	846	**	**	**
Microbubble Flotation	Fines	3.90	0.13	95.70	96.99	678	2313	16	24

* Coarse - (28 Mesh X 0)
Fines - (200 Mesh X 0)
Micronized - (325 Mesh X 0)

** No data available for this size fraction.

Reagent Dosages: 0.67 lbs MIBC/Ton of Coal
0.50 lbs #2 Fuel Oil/Ton of Coal

Ultrasonic Frequency: 0 KHz

As seen in Table 15, when ultrasonics was used, product-ash decreased 13%, pyritic sulfur decreased 32%, with no effect upon combustible recovery when compared with baseline conditions (no ultrasonic pretreatment). Also, the ash-separation index increased 24 points, the pyritic sulfur-separation index increased 427 points, and both ash and pyrite rejection improved after ultrasonic pretreatment.

An ultrasonic enhancer was used with this size fraction to determine the effects, if any, upon product-ash content, pyritic sulfur content and combustible recovery. Although no improvements were noted for product-ash or pyritic sulfur contents, due to the increase in combustible recovery, the ash-separation index increased 126 points, and the pyritic sulfur-separation index increased 364 points.

Fines (200 Mesh X 0)

The optimum experimental parameters chosen for this size fraction were 0.5 lbs fuel oil/ton of coal and 0.67 lbs MIBC/ ton of coal. These reagent dosages were used for both microbubble and conventional flotation. Experimental data for this series of conventional flotation experiments are presented in Tables 14 and 15. Again, excellent combustible recovery and yield data were obtained. Also, grinding the coal to this size fraction produced a product with less ash as compared with the coarse (28 mesh X 0) size fraction.

When the slurry was subjected to ultrasonic pretreatment, and then floated conventionally, product-ash content improved 13% and pyritic sulfur content

TABLE 15

Pittsburgh #8

After Ultrasonic Pretreatment

Flotation Process	Size Fraction*	Product, Ash (%)	Product, Pyritic Sulfur (%)	Combustible Recovery (%)	Yield (%)	Ash Separation Index	Pyrite Separation Index	Ash Rejection (%)	Pyrite Rejection (%)
Conventional Flotation	Coarse	3.80	0.21	89.71	91.30	382	770	27	41
	Fines	3.40	0.10	97.71	98.74	1385	3081	12	8
	Micronized	3.29	0.12	99.00	96.56	1546	**	30	**
Microbubble Flotation	Fines	3.29	0.12	91.20	96.58	626	1135	14	11

The following experiments were performed using the Ultrasonic Enhancer, for comparison.

Conventional Flotation	Coarse	4.03	0.18	96.91	95.32	508	1133	19	28
	Fines	3.58	0.05	99.00	98.98	1544	8849	11	12

* Coarse - (28 Mesh X 0)

Fines - (200 Mesh X 0)

Micronized - (325 Mesh X 0)

** No data available for this size fraction.

Reagent Dosages: 0.67 lbs MIBC/Ton of Coal
0.50 lbs #2 Fuel Oil/Ton of Coal

Ultrasonic Frequency: 20 KHz

74%. Combustible recovery, yield, ash rejection, and sulfur rejections improved minimally. The ash-separation index more than doubled after pretreatment with ultrasonics prior to flotation, and the sulfur-separation index increased to 3081 from 638.

An ultrasonic enhancer was also used for this size fraction during conventional flotation experiments. As seen in Table 15, pyrite reduction improved by 50%, combustible recovery and yield improved slightly, and there was no effect upon product-ash reduction. The ash-separation index did improve 159 points, and the pyritic-sulfur separation index increased 5768 points. No improvements in either ash or pyrite rejection were noted.

Microbubble flotation experiments were conducted on the fines fraction in order to perform comparative analyses between the two flotation processes. Without pretreatment, microbubble flotation did not improve product-ash content over conventional flotation. However, pyritic-sulfur content decreased significantly (67%). After ultrasonic preconditioning there were no differences between conventional or microbubble flotation data, as seen in Table 15. However, when comparing microbubble flotation with and without ultrasonic pretreatment, ash content did decrease 16% after ultrasonic preconditioning.

In order to verify that ultrasonics did have a fracturing effect upon this coal species, a particle size analysis of a feed and product sample were performed. Again, as seen in Table 16, based upon the decrease in mass mean diameter, and percent increase of particles in the smallest ranges, ultrasonics did have a fracturing effect upon this coal.

TABLE 16
PARTICLE SIZE ANALYSIS

Pittsburgh #8

NO ULTRASONICS

Mesh Size	Micron Size	Percent Cumulative Below	Percent Between Band Sizes
28	595	100.0	0.0
50	300	100.0	0.0
	212	100.0	2.4
100	150	97.5	5.9
150	106	91.5	10.5
200	75	81.0	15.2
270	53	65.7	14.4
400	38	51.3	12.6
	27	38.7	9.8
	19	28.9	8.2
	13	20.6	7.1
	9.4	13.5	5.6
	6.6	7.9	3.9
	4.7	3.9	3.9

Mass Mean Diameter = 36.80 microns

AFTER ULTRASONIC PRETREATMENT - 20 KHz

Mesh Size	Micron Size	Percent Cumulative Below	Percent Between Band Sizes
28	595	100.0	0.0
50	300	100.0	0.0
	212	100.0	0.0
100	150	100.0	4.1
150	106	95.8	8.1
200	75	87.7	10.7
270	53	76.9	13.5
400	38	63.4	11.1
	27	52.2	9.8
	19	42.3	9.9
	13	32.4	7.9
	9.4	24.4	7.7
	6.6	16.6	6.5
	4.7	10.1	10.1

Mass Mean Diameter = 25.16 microns

Analyses were performed using Microtrac Particle Size Analyzer

Micronized (325 mesh X 0)

For this fraction, 1.0 lb fuel oil/ton of coal and 0.67 lbs MIBC/ton of coal were used as the optimum reagent dosages. As seen in Table 14, micronizing the coal and floating conventionally, produced a product that when compared to the original head sample had 44.9% less ash and 64.1% less pyritic sulfur, while maintaining 99% combustible recovery.

As seen in Table 15, after ultrasonic pretreatment, product-ash content decreased minimally and pyritic sulfur decreased 36% when compared with baseline conditions. There was no effect upon combustible recovery. No microbubble flotation tests were conducted on the micronized fraction.

UPPER FREEPORT

For each of the three size fractions, a conventional flotation fuel oil dosage curve was prepared. From these curves, optimum reagent dosages were selected. These optimum results were used for both microbubble and conventional experimentation.

Coarse (28 mesh X 0)

An optimum fuel oil dosage of 2.5 lbs/ton of coal and MIBC dosage of 1.3 lbs/ton of coal were selected. Experimental results obtained are presented in Tables 17 and 18. These experiments were conducted both with and without ultrasonic pretreatment.

TABLE 17

BASELINE CONDITIONS - Upper Freeport

No Ultrasonic Pretreatment

Flotation Process	Size Fraction*	Product, Ash (%)	Product, Pyritic Sulfur (%)	Combustible Recovery (%)	Yield (%)	Ash Separation Index	Pyrite Separation Index	Ash Rejection (%)	Pyrite Rejection (%)
Conventional Flotation	Coarse	9.20	0.81	64.30	61.04	183	**	63	**
	Fines	13.95	1.38	69.60	66.75	115	118	46	45
	Micronized	6.40	0.73	89.40	79.88	552	**	55	**
Microbubble Flotation	Fines	10.30	0.55	68.70	91.75	245	181	14	5

* Coarse - (28 Mesh X 0)
 Fines - (200 Mesh X 0)
 Micronized - (325 Mesh X 0)

** No data available for this size fraction.

Reagent Dosages: Coarse and Fines Fractions - 1.30 lbs MIBC/Ton of Coal
 2.50 lbs #2 Fuel Oil/Ton of Coal

Micronized Fraction - 2.0 lbs MIBC/Ton of Coal
 5.0 lbs #2 Fuel Oil/Ton of Coal

Ultrasonic Frequency: 0 KHz

TABLE 18

Upper Freeport

After Ultrasonic Pretreatment

Flotation Process	Size Fraction*	Product, Ash (%)	Product, Pyritic Sulfur (%)	Combustible Recovery (%)	Yield (%)	Ash Separation Index	Pyrite Separation Index	Ash Rejection (%)	Pyrite Rejection (%)
Conventional Flotation	Coarse	9.26	0.85	88.53	79.23	335	**	50	**
	Fines	10.73	0.95	97.13	90.92	632	1381	41	74
	Micronized	5.80	0.50	98.10	84.25	808	**	53	**
Microbubble Flotation	Fines	9.20	0.67	90.90	96.29	562	522	12	8

* Coarse - (28 Mesh X 0)

Fines - (200 Mesh X 0)

Micronized - (325 Mesh X 0)

** No data available for this size fraction.

Reagent Dosages: Coarse and Fines Fractions - 1.30 lbs MIBC/Ton of Coal
 2.50 lbs #2 Fuel Oil/Ton of Coal

Micronized Fraction - 2.0 lbs MIBC/Ton of Coal
 5.0 lbs #2 Fuel Oil/Ton of Coal

Ultrasonic Frequency: 20 KHz

The data in Table 17 indicate that for those experiments conducted without an ultrasonic pretreatment step, poor combustible recovery and yield were obtained. When comparing with feed coal data, ash content did decrease 44% and pyritic sulfur decreased 54%.

As seen in Table 18, after ultrasonic pretreatment, neither product-ash content nor pyritic sulfur decreased; however, combustible recovery improved significantly (38%) as did yield (30%). Due to the improved recovery, the ash-separation index increased 152 points. The improvement in combustible recovery may be attributed to the fact that ultrasonic cavitation produced fresh surface sites for chemical, namely fuel oil, attachment (32). Also, the collector is more effectively transferred to the solid interface by the microjet streaming and improved mass transfer which results from ultrasonic cavitation. (41,45). Even though sonochemical effects were observed, it is postulated that the 20 kHz frequency and power level used produced insufficient energy required for coarse particle fracturing.

Fines (200 mesh X 0)

Optimum reagent dosages of 2.5 lbs fuel oil/ton of coal and 1.3 lbs MIBC/ton of coal were used for this series of experiments. Table 17 results again indicate that for conventional flotation, poor combustible recovery and yields were obtained.

After pretreatment with ultrasonics prior to conventional flotation, Table 18 shows that product-ash content decreased 23%, pyritic sulfur content

20%, combustible recovery improved 40% and product yield improved 36% after ultrasonics was used. Ash rejection was the same after ultrasonic pretreatment; however, pyrite rejection did improve 64%. The ash-separation index for this size fraction improved 517 points, and the sulfur-separation index increased 1263 points. Again, the improvements may be attributed to the sonochemical effects of ultrasonics.

Microbubble flotation experiments were again conducted in order to perform comparative analyses between the two flotation processes. Data in Tables 17 and 18 indicate that when ultrasonics was used in conjunction with microbubble flotation, product ash was reduced by 12%. After ultrasonic pretreatment, combustible recovery increased 32%, and clean coal yield 5%. The ash separation index did improve 317 points, and the sulfur separation index increased 341 points when an ultrasonic pretreatment step was included. Ash and pyrite rejection data were essentially the same after ultrasonic pretreatment.

In order to verify that ultrasonics was indeed fracturing this coal, a particle size analysis of untreated and treated coal samples was performed. It is obvious from Table 19 that particle size did decrease since the mass mean diameter decreased from 29 to 24 microns. This data also indicates that Upper Freeport is the hardest coal to fracture with ultrasonics, since there was only a 4.7 micron decrease in the mass mean diameter as compared with 11.7 microns for Pittsburgh #8, and 9.7 microns for Illinois #6.

TABLE 19
PARTICLE SIZE ANALYSIS

Upper Freeport

NO ULTRASONICS

Mesh Size	Micron Size	Percent Cumulative Below	Percent Between Band Sizes
28	595	100.0	0.0
50	300	100.0	0.0
	212	100.0	1.9
100	150	98.1	1.9
150	106	96.1	6.9
200	75	89.1	12.5
270	53	76.6	14.9
400	38	61.7	14.4
	27	47.2	11.4
	19	35.8	9.6
	13	26.1	7.7
	9.4	18.3	6.7
	6.6	11.6	5.2
	4.7	6.3	6.3

Mass Mean Diameter = 29.07 Microns

AFTER ULTRASONIC PRETREATMENT - 20 KHz

Mesh Size	Micron Size	Percent Cumulative Below	Percent Between Band Sizes
28	595	100.0	0.0
50	300	100.0	0.0
	212	100.0	0.0
100	150	100.0	1.3
150	106	98.6	5.4
200	75	93.2	10.5
270	53	82.7	14.2
400	38	68.4	14.4
	27	54.0	12.0
	19	41.9	10.9
	13	31.0	8.8
	9.4	22.2	8.0
	6.6	14.1	6.4
	4.7	7.7	7.7

Mass Mean Diameter = 24.41 Microns

Analyses were performed using Microtrac Particle Size Analyzer.

Micronized (325 mesh X 0)

For this series of conventional flotation experiments, optimum reagent dosages for this fraction were 5 lbs fuel oil/ton of coal and 2.0 lbs MIBC/ton of coal. The data in Table 17 indicate that "micronizing" this coal produced a product which contained less ash and pyritic sulfur than the other size fractions. Also, good combustible recovery and yield were obtained.

As seen in Table 18, after treatment with ultrasonics, product-ash content decreased an additional 10%, pyritic sulfur decreased by 18%, and combustible recovery improved significantly by 32%. The ash-separation index was greatest for this size fraction, 552 without ultrasonics, and 808 after ultrasonic pretreatment. No microbubble experiments were conducted for this size fraction.

ECONOMIC ANALYSIS

In order to investigate economic viability, additional laboratory tests were conducted to determine the process costs, and approximate commercial scale-up costs for the ultrasonic pretreatment step. Scale-up economics for beneficiation techniques, such as column and conventional flotation, will not be included in this analysis.

Multitudes of frothers and collectors, at varying prices, are commercially available. For these series of experiments, MIBC and #2 fuel oil were utilized as the frother and collector, respectively. The actual dosages

used for each coal were those previously determined. Costs for MIBC and #2 Fuel Oil are not used in economic calculations.

The objectives of this study were to:

1. Determine the effect of ultrasonic energy on concentrate ash, combustible recovery, and yield for various energy inputs;
2. Determine the economic impact of ultrasonics on increased yields and combustible recoveries while lowering product ash levels; and
3. Determine the capital costs of a commercial-scale unit.

EQUIPMENT

The ultrasonic equipment used for this experimentation was purchased from Branson Ultrasonics located in Danbury, Connecticut. As seen in Figure 7, a variable power supply, Model W-350, was used in conjunction with a continuous processing cell. A converter, number 102, with a 2.0 gain booster and 2.8 gain horn were used to transfer the energy to the processing cell. This equipment allowed variable amplitude (0-4.2 kw) and wave intensities (0-3333.3 kw/m²) to be investigated.

EXPERIMENTATION

Sample Preparation

For these series of tests, previously prepared samples of Upper Freeport and Pittsburgh #8 fines (raw coal crushed to 200 Mesh X 0) were used. For further information on preparation of samples, see Figure 3.

Procedures

Figure 6 describes the procedure followed for all experimentation. A process flow diagram outlining the continuous ultrasonic pretreatment step may be found in Figure 7.

For each experiment, coal and water were slurried at 20% solids for 25 minutes to ensure complete wetting of the coal. From an economic standpoint, with the lab scale equipment available, processing costs would be prohibitive if lower solids were used. Collector, #2 fuel oil, was then added, and the slurry was mixed an additional 10 minutes. Next, the slurry was pumped through the ultrasonic conditioning chamber where varied amounts of energy, at a frequency of 20 KHz were applied to determine the effects of ultrasonics on a cost basis. A constant flow of 1.8 liters/min was maintained for each power level, which allowed treatment of 445 grams/minute of coal on a continuous basis. Unfortunately, the pump output capacity limited flowrates above 1.8 liters which hindered the ability to investigate kw-hr usages of less than 10 kw-hrs, or approximately \$0.60/ton of material processed.

FIGURE 6
FLOWSHEET OF CONTINUOUS ULTRASONIC PRETREATMENT PROCEDURE

FIGURE 7
CONTINUOUS ULTRASONIC PRETREATMENT SCHEMATIC

During ultrasonic processing, samples were collected for conventional flotation testing. A Denver Model D-12 with a 2.5 liter stainless steel cell was used for all flotation tests.

At each designated power level, 100 grams of coal was collected and diluted to 5% solids at the Denver cell. (A solids concentration of 5% was chosen to correspond with all previous conventional testing.) MIBC was then added and allowed to mix for one minute. For all conventional tests, a three minute product collection time was used. Samples of product and reject were collected and analyzed.

RESULTS AND DISCUSSION

Upper Freeport

The effect of ultrasonics power level variations may be found in Figure 8. The corresponding data in Table 20, showed that increased power levels of ultrasonics lowers concentrate ash levels while increasing combustible recovery and clean coal yield. In these series of experiments, the pump capacity would not allow testing of flowrates associated with energy usages levels below 10 kw-hr per ton of coal. At 10 kw-hr, energy level correlates to approximately \$0.60 of electricity usage for every ton of coal processed.

In order to recognize the benefits of ultrasonic pretreatment, an economic sensitivity analysis was performed to relate the savings that could be directly attributed to any increase in product yield for varying cost of

FIGURE 8

Upper Freeport

Effect of Ultrasonic Power Level
Variations on Combustible Recovery,
Clean Coal Yield and Concentrate Ash

Note: These experiments were conducted using ultrasonics at a frequency of 20 KHz.

TABLE 20
UPPER FREEPORT
EFFECT OF ULTRASONIC POWER LEVEL VARIATIONS

Power Level (kW-Hr/ton)	Concentrate Ash (%)	Combustible Recovery (%)	Clean Coal Yield	Cost per Ton
0.0	13.95	65.0	60.9	\$0.00
10.0	12.10	76.8	67.5	\$0.60
13.9	11.40	86.3	76.5	\$0.83
25.8	11.50	92.7	83.6	\$1.54

coal. For this laboratory scale analysis, 13.9 kw-hr of energy per ton of coal was chosen from Table 21, which would cost approximately \$0.83 per ton of coal processed. Even at this energy level, at a yield of 75%, a savings of \$267 per eight hour shift would be attained, if coal were selling at \$25.00 per ton. On a yearly basis, a plant processing as little as 160 tons of coal per shift in their flotation circuit, operating 2 shifts /day, 240 days/year would realize cost savings of \$129,000.

At an improved yield of 84%, a coal cost savings of \$554 for an 8 hour shift would be realized, if coal were selling at \$25.00 per ton. This would amount to an annual savings of \$266,000 for a plant operating 2 shifts/day, 240 days/year.

Pittsburgh #8

The same test scenario used for the Upper Freeport coal was also used for the Pittsburgh #8 coal. The Pittsburgh #8 is essentially a clean coal (5.19% ash), so improvement was not expected to be as noticeable as was for the Upper Freeport coal. Again, low pump output adversely effected usage of energy.

The effect of ultrasonic power level variation may be found in Figure 9. In conjunction, data obtained directly from this Figure is found in Table 22. Examination of this data indicates that ultrasonics are not needed to increase combustible recovery, yield or lower concentrate grade. Of interest for this coal would be the pyritic sulfur removal associated with the application of ultrasonics. Further investigation of the sensitivity

TABLE 21

Assumptions: 160 Tons of Coal (fines fraction, 200 Mesh X 0) processed per 8 hour day
 Cost of Kilowatt Hour is \$0.06 based upon Louisville Gas and Electric, April, 1990.

1 Day = 8 Hours

Clean Coal Yield without an Ultrasonic Pretreatment Step = 65%

Clean Coal Yields with an Ultrasonic Pretreatment Step = 75% and 85%

Ultrasonic Processing Costs @ 75% yield = \$0.83/Kw-Hr/ton of coal

Ultrasonic Processing Costs @ 84% yield = \$1.54/Kw-Hr/ton of coal

Cost of Coal/Ton	\$15.00	\$20.00	\$25.00	\$30.00	\$35.00	\$40.00	\$45.00	\$50.00	\$55.00
Cost of Coal/Day	\$2,400.00	\$3,200.00	\$4,000.00	\$4,800.00	\$5,600.00	\$6,400.00	\$7,200.00	\$8,000.00	\$8,800.00
Yield, 65% (tons/day)	104	104	104	104	104	104	104	104	104
Yield, 75% (tons/day)	120	120	120	120	120	120	120	120	120
Yield, 84% (tons/day)	136	136	136	136	136	136	136	136	136
Add'l Coal Processed @ 75% Yield, (tons/day)	16	16	16	16	16	16	16	16	16
Coal Cost Savings @ 75% Yield, (\$/day)	\$240.00	\$320.00	\$400.00	\$480.00	\$560.00	\$640.00	\$720.00	\$800.00	\$880.00
Ultrasonic Cost @ 75% Yield, (\$/day)	\$132.80	\$132.80	\$132.80	\$132.80	\$132.80	\$132.80	\$132.80	\$132.80	\$132.80
Coal Cost Savings @ 75% Yield, (\$/day)	\$107.20	\$187.20	\$267.20	\$347.20	\$427.20	\$507.20	\$587.20	\$667.20	\$747.20
Add'l Coal Processed @ 84% Yield, (tons/day)	32	32	32	32	32	32	32	32	32
Coal Cost Savings @ 84% Yield, (\$/day)	\$480.00	\$640.00	\$800.00	\$960.00	\$1,120.00	\$1,280.00	\$1,440.00	\$1,600.00	\$1,760.00
Ultrasonic Cost @ 84% Yield, (\$/day)	\$246.40	\$246.40	\$246.40	\$246.40	\$246.40	\$246.40	\$246.40	\$246.40	\$246.40
Savings per Day @ 84% Yield	\$233.60	\$393.60	\$553.60	\$713.60	\$873.60	\$1,033.60	\$1,193.60	\$1,353.60	\$1,513.60

FIGURE 9

Pittsburgh #8

Effect of Ultrasonic Power Level
Variations on Combustible Recovery,
Clean Coal Yield and Concentrate Ash

Note: These experiments were conducted using ultrasonics at a frequency of 20 KHz.

TABLE 22

PITTSBURGH #8

EFFECT OF ULTRASONIC POWER LEVEL VARIATIONS

Power Level (kW-Hr/ton)	Concentrate Ash (%)	Combustible Recovery (%)	Clean Coal yield	Cost per Ton
0.0	3.8	94.5	93.4	\$0.00
9.0	3.6	95.3	94.2	\$0.54
13.3	3.7	95.8	94.2	\$0.80
25.3	3.7	96.7	94.3	\$1.50

analysis seen in Table 23, confirmed that ultrasonics does not increase yield sufficiently to justify usage.

Capital Costs

Methods of up-scaling an ultrasonic device were discussed with Branson Ultrasonics. For all calculations, the ultrasonic unit was considered as an add-on to an existing coal preparation plant circuit. Cost figures could only be "ballpark" estimated.

A full scale unit design and fabrication would take from one to two years to complete and would cost well over \$100,000 dollars. Another alternative would be to use off-the-shelf units and power supplies. For this method, it would take 6 to 8 of the Model 184 Systems at an approximate cost of \$5,000 each, or \$30,000 to \$40,000. When considering the Upper Freeport sensitivity analysis, and cost of coal at \$25.00 per ton, the ultrasonics would save \$534.40 for each 16 hour day. A full scale unit costing \$150,000 would have a pay back period of 280.7 working days, or slightly over one year. Utilizing existing systems at total cost of \$40,000 would have a pay back period of only 75 days, or less than one third of a year.

TABLE 23

Sensitivity Analysis

Assumptions: 160 Tons of Coal processed per 8 hour day
 Cost of Kilowatt Hour is \$0.06 based upon Louisville Gas and Electric, April, 1990.
 1 Day = 8 Hours
 Clean Coal Yield without an Ultrasonic Pretreatment step = 93.4%
 Clean Coal Yield with an Ultrasonic Pretreatment step = 94.2%

Pittsburgh #8										
Cost of Coal/Ton	\$15.00	\$20.00	\$25.00	\$30.00	\$35.00	\$40.00	\$45.00	\$50.00	\$55.00	
Cost of Coal/Day	\$2,400.00	\$3,200.00	\$4,000.00	\$4,800.00	\$5,600.00	\$6,400.00	\$7,200.00	\$8,000.00	\$8,800.00	
Yield, 93.4% (tons/day)	149.44	149.44	149.44	149.44	149.44	149.44	149.44	149.44	149.44	
Yield, 94.2% (tons/day)	150.72	150.72	150.72	150.72	150.72	150.72	150.72	150.72	150.72	
Increase in Amount of Coal Processed at 94.2% Yield (tons/day)	1.28	1.28	1.28	1.28	1.28	1.28	1.28	1.28	1.28	
Cost Savings (\$/day)	\$19.20	\$25.60	\$32.00	\$38.40	\$44.80	\$51.20	\$57.60	\$64.00	\$70.40	
Ultrasonic Process Costs (\$/Kw-hr/ton of coal)	\$0.54	\$0.54	\$0.54	\$0.54	\$0.54	\$0.54	\$0.54	\$0.54	\$0.54	
Ultrasonic Cost Per Day	\$86.40	\$86.40	\$86.40	\$86.40	\$86.40	\$86.40	\$86.40	\$86.40	\$86.40	
Savings per Day	(\$67.20)	(\$60.80)	(\$54.40)	(\$48.00)	(\$41.60)	(\$35.20)	(\$28.80)	(\$22.40)	(\$16.00)	

SUMMARY

CONCLUSIONS

Due to the acid rain legislation currently under consideration, coal burning utilities are searching for cost effective methods of reducing ash and sulfur contents, thus avoiding stiff penalties and possibly earning tax credits. Current post combustion methods of reducing pollutants, including scrubbers, are very expensive. Thus more economical alternatives are needed.

A technology which can provide a cost effective means of reducing impurities prior to combustion, where disposal is much less expensive and handling much easier, is certainly desirable.

Generally, utilities require a pulverized (80% 200 mesh x 0) coal for furnace firings. Though froth flotation is recognized as the most practical and cost effective method of cleaning this size fraction prior to combustion, ultrafine impurities may remain attached to the coal particle, reporting with the product, resulting in low recoveries and high ash and sulfur contents.

Based upon research conducted during this contract, both conventional and advanced froth flotation processes may be enhanced by the use of an ultrasonic preconditioning step prior to flotation.

Ultrasonic energy, applied at a frequency of 20 kHz:

1. fractured the coal, as verified by particle size analysis of treated and untreated coal samples;
2. liberated ash and pyrites from the coal matrix as evidenced by petrography and scanning-electron-microscopy data;
3. surface polished the coal thru the scrubbing action of cavitation, rendering it hydrophobic, thus improving combustible recovery and yield;
4. seems to implant flotation chemicals on the coal surface due to the microjet streaming and intense mixing occurring during cavitation, as evidenced by improved combustible recovery and yield;
5. in every case, improved sulfur separation, and in all but one case, improved ash separation;
6. in most cases, decreased product-ash and pyrite contents.

Improvements in product quality were noted for each of the test coals, Illinois #6, Pittsburgh #8, and Upper Freeport, even though these coals varied widely in ash and sulfur content. This further substantiates the benefits of using ultrasonics on all coal species.

Based upon economic analyses, a sensitivity analysis showed that as expected, ultrasonics is not justified for Pittsburgh #8, based solely on

yield. However, factoring in the sulfur and ash reduction handling and disposal costs, the cost of ultrasonics might very well be justified.

Conversely, the use of ultrasonics as a precleaning step prior to beneficiation of Upper Freeport was justifiable purely on a process cost basis. Additionally, capital cost outlay for a 320 ton per day plant could be as low as \$40,000 with payback in less than one year.

RECOMMENDATIONS

- * Although excellent results were obtained without the use of modifying reagents, the additive experiments performed with Pittsburgh #8 coal, essentially a clean coal, demonstrate the effects an ultrasonic enhancer could provide. Further research should be performed on the use of additives to promote cavitation, enhance transference, and therefore produce a cleaner product. The cost of the additives should justify the improvement in product quality.
- * Further, research should be performed to investigate whether the micro-jet streaming and hydrodynamic currents produced during cavitation better adsorbs flotation chemicals onto coal surfaces, thus reducing the quantity of flotation chemicals required and lowering process costs.
- * In order to further reduce ultrasonic processing costs, research should be performed to determine whether similar ash and sulfur reductions are realized when higher concentrations of solids are processed. This would

again reduce ultrasonic processing costs.

- * Since it is theorized that there exists an optimum resonance frequency which promotes fracturing of dissimilar materials, research should be performed at various frequencies in the ultrasonic range.
- * Further testing at lower energy levels, less than 10 kw-hr/ton of coal, should be investigated.
- * Tests to determine reagent consumption versus energy input should be investigated.
- * A sensitivity analysis investigating the effect that ultrasonics has upon pyrite reduction and utility economics should be performed.
- * Comparative analysis of ash and sulfur handling and disposal post combustion costs versus pre-combustion removal costs with ultrasonic pretreatment should be performed.

APPENDIX A

PERFORMANCE CRITERION FORMULA

Criteria	Formula
1. Clean Coal Ash, %	$\frac{[\text{wt of crucible \& ash residue}] - [\text{wt of crucible}]}{\text{wt of analysis sample used}} \times 100$
2. Clean Coal Pyrite, %	Performed by University of Kentucky, Energy Cabinet Laboratory
3. Energy Recovery, %	$\frac{\text{Clean Coal Wgt\%} \times \text{Clean Coal Heating Value}}{\text{Feed Coal Heating Value}}$
4. Ash Separation Efficiency Index	$\frac{\text{Clean Coal Wgt\%} \times \text{Refuse Ash(\%)}}{\text{Clean Coal Ash(\%)}}$
5. Pyritic Sulfur Separation Efficiency Index	$\frac{\text{Clean Coal Wgt\%} \times \text{Refuse Pyr.S(\%)}}{\text{Clean Coal Pyr.S(\%)}}$
6. Ash Rejection, %	$\frac{[100 - \text{Clean Coal Wgt\%}] \times \text{Refuse Pyr.S(\%)}}{\text{Feed Coal Ash (\%)}}$
7. Pyritic-Sulfur Rejection %	$\frac{[100 - \text{Clean Coal Wgt\%}] \times \text{Refuse Pyr.S(\%)}}{\text{Feed Pyr. S (\%)}}$
8. Ash Rejection, %	$\frac{[100 - \text{Clean Coal Wgt\%}] \times \text{Refuse Ash(\%)}}{\text{Feed Coal Ash (\%)}}$
9. Pyritic Rejection, %	$\frac{[100 - \text{Clean Coal Wgt\%}] \times \text{Refuse Pyrite(\%)}}{\text{Feed Pyrite (\%)}}$

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