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Evaluation of the Micropulverization and Benefication of Lignite

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EVALUATION OF THE MICROPULVERIZATION

AND BENEFICIATION OF LIGNITE

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EVALUATION OF THE MICROPULVERIZATION AND BENEFICIATION OF LIGNITE

by

C. W. Bouchillon and W. G. Steele

INTRODUCTION

Lignite has been mined and utilized as a boiler fuel on a regional basis for several years, primarily in North Dakota and Texas. The major U. S. lignite deposits occur in the Fort Union Region (North Dakota and Montana) and the Gulf Region (Texas, Louisiana, Arkansas, Tennessee, Mississippi, and Alabama) (1, 2). The Gulf Coast lignite resources have been estimated at thirty-five billion tons with the majority of this in Texas and Mississippi (3). Typical as-received analysis of Mississippi lignites is given in Table 1.

The research reported here is part of a cooperative program among the Mississippi Energy Research Center, Mississippi State University, the Mississippi Department of Energy and Transportation, the Mississippi Mineral Resources Institute, Mississippi Power and Light Company, and Ergon, Inc. The broad objective is to develop processes which will allow effective expansion of the use of lignite as an energy source. The three major areas of research under development are micropulverization, drying and beneficiation. Each of these three general processes has potential to improve lignite as a fuel.

The first area of this research program has concentrated on the micropulverization of lignite in a fluid-energy pulverizing system. Tests have been conducted with various operating parameters for the pulverization system. The samples from these tests have been analyzed for particle size, ash content, and heating value. Preliminary results show that lignite can be pulverized successfully in a fluid-energy apparatus as the initial stage in the production of a microfine lignite fuel for boilers currently using oil or gas.

Related work on ultrafine pulverized coal (4, 5) has shown that micropulverized coal (100% less than 44 microns) will cause less slagging than conventional pulverized coal when used in an oil or gas boiler. Figure 1, similar to the illustration presented by Margulies, et.al. (4), illustrates the basic concept favoring micropulverization. Other work on slag reduction of Canadian lignites has been reported (6). The tubes in oil and gas boilers are generally spaced closer than those in conventional coal-fired boilers. Because of this increased tube surface to flow area ratio, the larger ash particles in conventional pulverized coal will impact the tubes and cause slagging. However, the very fine ash particles in the micropulverized coal or lignite tend to follow the flow streams around the tubes resulting in less slagging and erosion. These small particles can then be collected by a scrubber, a baghouse or a precipitator.

An oil or water slurry is one of the primary methods being considered for transport and end use of pulverized coal. With regular pulverized coal slurries, either the boiler must be modified to minimize fouling or the coal must be beneficiated to reduce the ash content. The coal slurry technology is progressing at a rapid rate and preliminary commercialization efforts are underway (7, 8, 9).

Work has been performed in Australia on the use of ultrafine brown coal to make a coal-oil mixture to replace diesel fuel for engines (10).

Also micropulverized coal-oil mixtures made by Ergon, Inc. have been successfully burned with no significant ash deposits forming in the boiler (11).

The primary result of the pulverization tests reported here was that lignite with its high moisture content could be micropulverized. The fluid-energy mill used for micropulverization was described by Taylor (12). Basically, a ring of inward facing jets directed slightly off-center causes a vortex flow to be established and the particles grind or impact against each other to generate a lignite product with a mean diameter of 5 to 7 microns based on population and 20 to 25 microns based on volume.

The second area of research reported here concentrated on beneficiation or ash removal processes. With processes which have been used successfully in coal cleaning as a starting point for consideration, attention was given to gravimetric, agglomeration, particle size separation, and ion-exchange methods.

Comparison of the ash characteristics of various lignites was presented by White (13) and is presented in Table 2. The Gulf Coast lignites generally have a much higher silicate content and a significantly lower sodium content than the North Dakota lignites. This suggests that a mechanical separation may be more important than a chemical or ion-exchange process for the Gulf Coast lignites.

Previous work in gravimetric methods applied to micropulverized coals indicated that hydrocyclones may be effective for ash removal. The results are highly dependent upon the washability—or the ash distribution with specific gravity. Recent work by Keller and Simmons (14) indicates that for coals with good washability characteristics.

that good cleaning of -325 mesh coal in a true heavy liquid medium of Freon-113 can be accomplished in a 2 inch cyclone with an 85 psi pressure drop. Washability studies (15) indicate that some Gulf region lignites do have reasonably good washability characteristics while some exhibit almost no ash content variation with specific gravity.

In an effort to reduce the sodium content of North Dakota lignites Paulson, et.al. (16) have done extensive work in ion-exchange processes which result in a reduction in the sodium content of the ash—apparently the predominant ash component contributing to tube fouling problems in boilers.

MICROPULVERIZATION

The lignite used in these tests was obtained from a seam in Panola County, Mississippi which is relatively close to the surface. The samples were stored in polyethylene bags in barrels prior to the pulverization studies at the Ergon laboratory.

Tests were performed with both as-mined and air-dried moisture levels and with various conditions of pulverizer operation. The air-drying consisted of cracking the lignite into large chunks and spreading it out in a sunlit area in an attempt to remove some of the surface moisture. The eight test runs are given in Table 3. Both steam and air were used as fluid-energy pulverization mediums and the pressure at the inlet nozzle to the fluid-energy mill was set at either 40 psi or 65 psi. The lignite was successfully micropulverized in all of the runs.

A proximate analysis was performed on some of the pulverized samples, and the moisture, ash, volatiles, fixed carbon, and heating value for these samples are given in Table 4. A significant result of the air driven micropulverization was the reduction in the moisture. It is our opinion that this was accomplished primarily by the significant increase in surface area caused by the micropulverization. This allowed much of the surface moisture and some of the inherent moisture to be removed by the heated (200°F) air driving fluid. Another observation is that the air predrying of the raw lignite had no significant effect on the moisture content of the samples studied. Additional tests are planned later concerning lignite moisture reduction.

It is also of interest to note the reduction in ash content during the micropulverization process. The ash content was reduced by approximately 10 percent in the samples which were evaluated. This reduction is attributed to the very small particle size observed for some of the ash particles and the resultant blow-by of these fine particles in the separation apparatus of the laboratory micropulverization equipment. A venturi scrubber followed by an air cyclone separator was used for removal of the micropulverized particles from the air stream.

An optical microscope was used to observe, measure and photograph the micropulverized samples from both as-mined and air-dried pretreatment. Typical particles sizes for the hydrocarbon particles were 15-30 microns and 2-5 microns for the ash particles. Figure 2 is a photograph of the micropulverized lignite as taken with an optical microscope camera system. Similar results were observed with a scanning electron microscope except that more individual particle surface detail could be observed as is shown in Figure 3. The apparent matrix of ash and carbon in the larger particles is seen in this picture.

Partiele size analyses were conducted by PETC-DOE on a Bausch and Lomb automatic image analyzer and by Mississippi Power and Light Company on a HIAC particle analyzer. The results of the particle analysis done on the Bausch and Lomb automatic image analyzer as reported by Killmeyer (17) are presented in Figure 4. For the two samples evaluated by PETC-DOE the arithmetic mean sizes on a number basis were 5.24 and 4.14 microns with geometric mean sizes on a weight basis of 22.54 and 14.79 microns respectively. These results are comparable with the optical microscope observations discussed above.

Typical results of the particle size analysis performed on the HIAC particle analyzer (18) showed that the mean particle sizes based on population ranged from 6.4 to 5.1 microns and the particle size based on volume ranged from 24.6 to 15.7 microns in the six test runs. These results are also comparable with the optical microscope evaluation and the automatic image analyzer results discussed above. The particle sizes observed for the samples taken suggest that micropulverization of Mississippi lignites can produce a resultant particle size in a desirable range—even with the high moisture content in the as-mined material.

BENEFICIATION

The first evaluation of the micropulverized samples included optical microscopic examination. A typical picture is shown in Figure 2. This suggested that the smaller particles were probably silicates and the larger particles were mostly hydrocarbons.

A hydrocyclone (3" x 5°) was obtained and modified for use as a true heavy liquid medium device. Freon-113 was selected as the fluid because of its low viscosity (=0.6 cp) and high specific gravity (si.5).

The modifications were made in an effort to provide significant flows to the underflow so as to carry out the smaller, heavier particles and allow the larger lighter particles to "float" out the overflow. The results of these tests were not successful—largely because of the poor washability characteristics of the particular lignite samples being used. An improved operational predictive method has been developed and presented by Pirzadeh (19) in a Master of Science Thesis as a result of this project.

A gravimetric analysis of the samples was obtained. The results of gravimetric testing of the micropulverized lignite samples which were performed by PETC-DOE as reported by Killmeyer (17) are presented in Table 5. The following observations relative to the gravimetric data may be made. The difference in final cumulative weights and initial sample weights is attributable in part to the very small particles which passed through the filter paper and formed a sort of paste or film on the pan after evaporation of the heavy liquid as reported by Killmeyer (17).

One result of these tests which is surprising—yet significant—is the large concentration of ash which remains with the lighter fractions as shown in Table 5. If this result is because the ash is embedded within the particle material—even at this small size—then it will be difficult to provide significant beneficiation through gravimetric or inertial means. Conversely, if there are significant surface forces on the small sized ash particles which overcome the body forces of gravity or the inertial forces experienced in the centrifuge, then it may be possible to introduce some controlling surface force such as would occur with surfactants or electrostatics.

The pyritic sulfur is a small fraction (only about six percent) of the total sulfur and nearly all of that is contained in the particles with specific gravity greater than 1.8. In that the specific gravity of pyrite is approximately five, this result is not surprising.

The total sulfur content of the samples examined is of the order of 0.5 percent which is encouraging from an applications point of view. As with the ash content, the total sulfur is evenly distributed throughout the gravimetric range for the samples tested.

Further evaluation in an electron microscope indicated that there are indeed very small particles of ash material embedded in the 20-25 micron particles as shown in Figure 3. The lighter surfaces in the figure indicate a different characteristic of that surface—either due to higher surface reflectivity or to a different plating characteristic. This corroborates the results of the gravimetric analyses.

SUMMARY AND FUTURE WORK

In summary, fluid energy micropulverization of lignite to a product with a mean diameter of about 5 microns on a population basis and about 20 to 25 mcirons on a volume basis was successfully accomplished. Some drying did accompany the air-driven micropulverization at moderate temperatures (s200°F), however, this did not affect the equilibrium moisture of the product.

Gravimetric and visual observations indicated that the ash was somewhat uniformly distributed throughout the specific gravity ranges, thereby making beneficiation by mechanical means very difficult.

Based on informatin gained to date, further work is planned in all three of the major areas of micropulverization, drying, and beneficration. Continued developments on improved designs for the fluid energy micropulverizer are planned in order to further optimize the operational efficiency of the process.

Further work will continue on the drying techniques which produce permanent equilibrium moisture reduction. These will include combined micropulverization and drying processes as well as other continuous drying processes.

Additional studies planned for the benficiation processes include further evaluation of the ash distribution relative to particle size distribution for possible air classificiation techinques. Further work will also be done on ion-exchange methods to remove the organically bound metal ash content.

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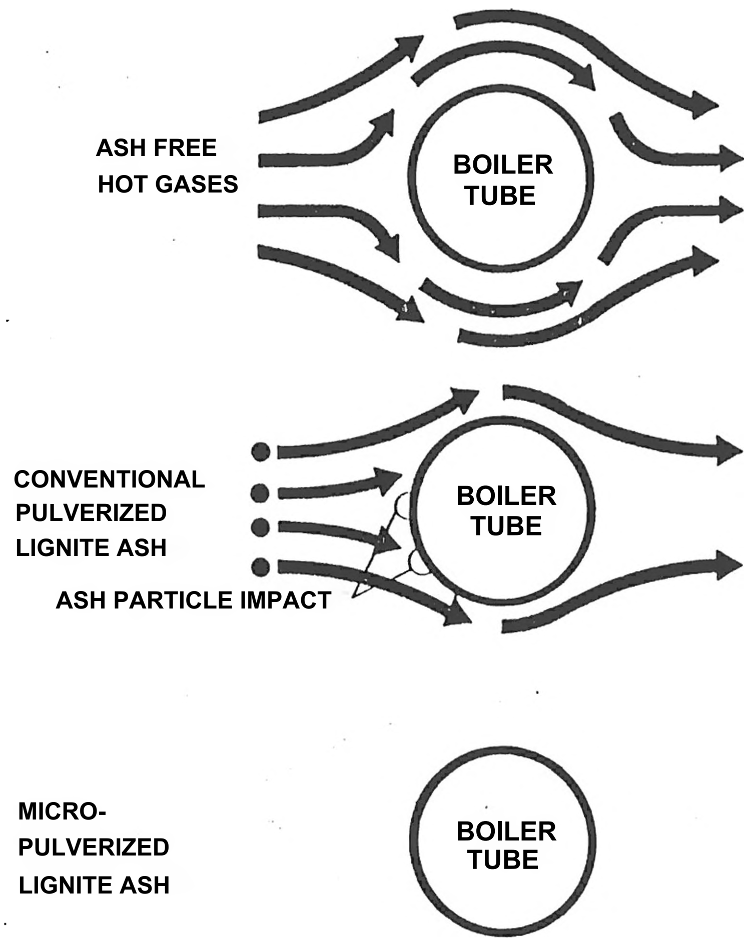

Figure 1.

COMPARISON OF GAS STREAMLINES AND ASH TRAJECTORIES WHEN FIRING CONVENTIONAL VS. MICROPULVERIZED LIGNITE

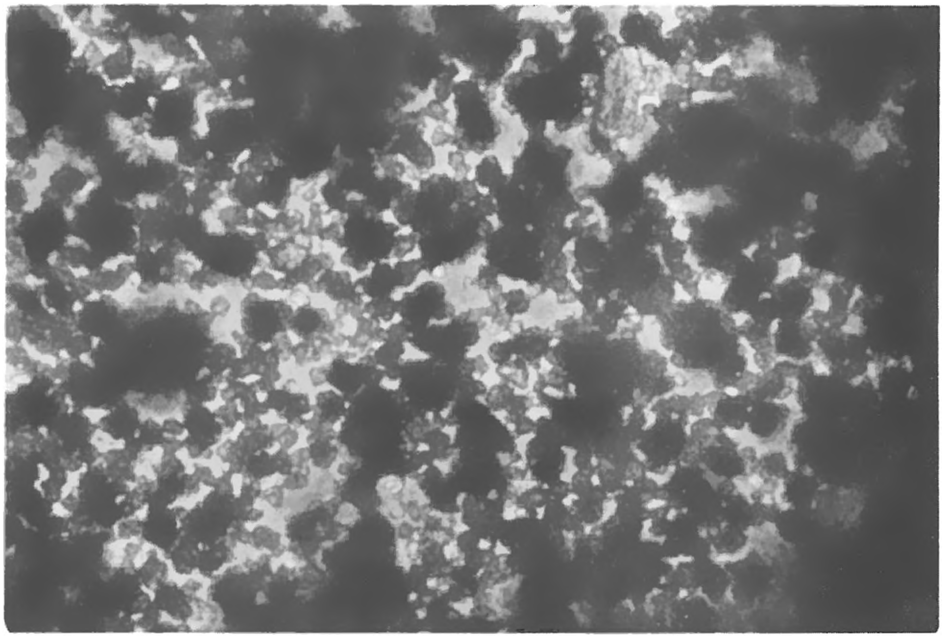

Figure 2. Micropulverized Lignite

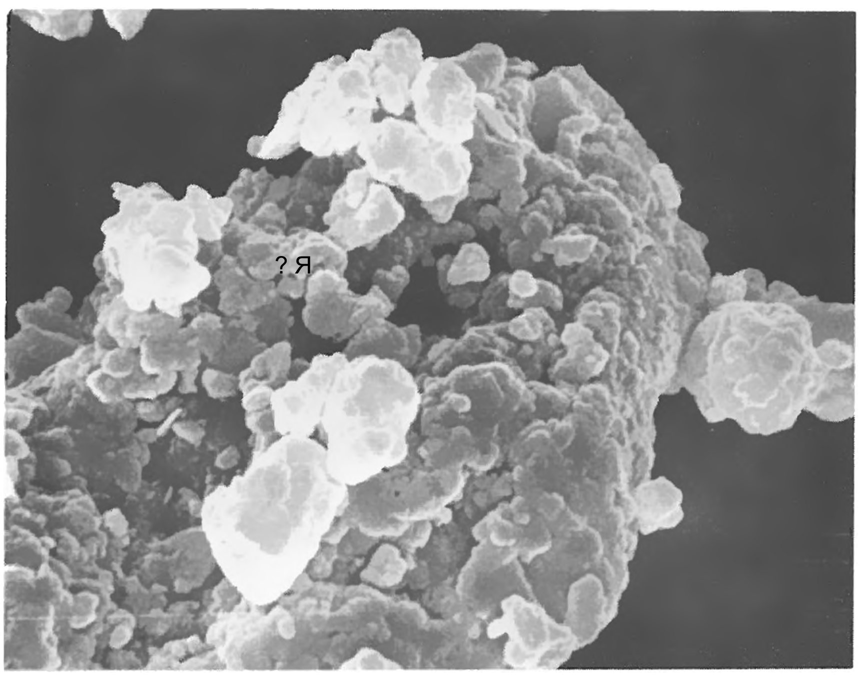

Figure 3. Scanning Electron Microscope View
of Micropulverized Lignite

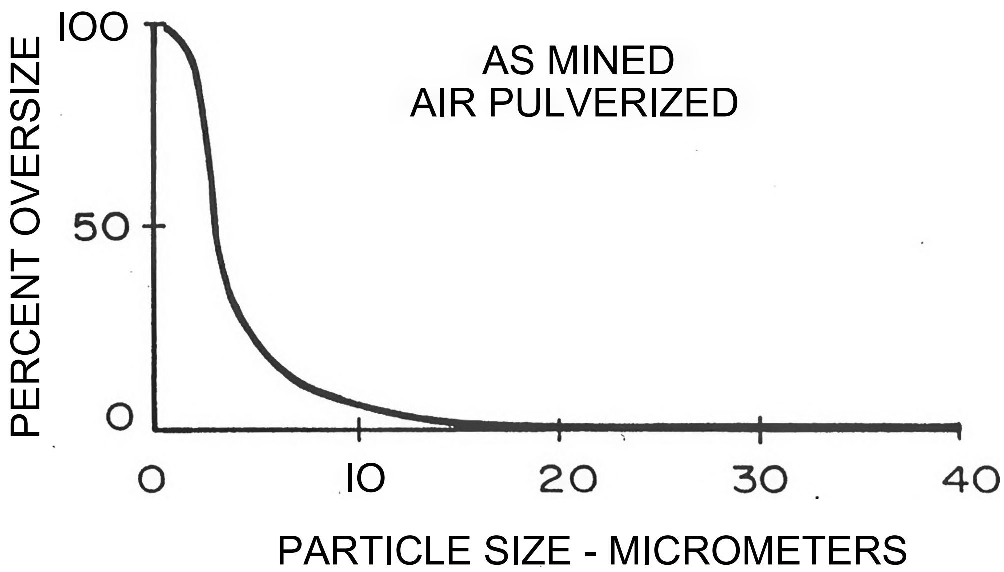

Figure 4. Results of the Bausch and Lomb Particle Size Analysis on a Population Basis for a Sample of Mississippi Lignite

TABLE 1

Typical As-Received Analyses of Mississippi Lignites (3)

	North Wilcox	South Wilcox	Claiborne
Moisture, %	44.0	43.0	42.0
Ash, %	12.0	12.0	14.0
Volatile Matter, %	25.0	24.0	30.0
Fixed Carbon, %	19.0	21.0	14.0
Sulfur, %	0.5	1.2	0.5
Higher Heating Value, Btu/lbm	5396.0	5509.0	5585.0

TABLE 2

Comparison of Ash Characteristics for Mississippi, Texas, and North Dakota Lignites and an Eastern Bituminous Coal (15)

	Mississippi	Texas			Eastern
	Claiborne	Wilcox	Claiborne	North Dakota	Bituminous
SiO_2	54.1	44.5	53.6	19.7	40.0
Al_2O_3	17.0	11.4	15.0	11.1	24.0
Fe_2O_3	6.9	7.4	6.6	9.1	16.8
TiO_2	1.0	1.1	0.8	0.4	1.3
P_2O_5	0.1	0.1	0.1	0.3	0.1
CaO	9.6	13.7	9.9	24.6	5.8
MgO	2.0	2.6	1.8	6.9	2.0
Na_2O	0.3	0.6	1.2	6.5	0.8
K_2O	0.5	0.6	1.2	0.4	2.4
SO_3	6.9	10.9	8.9	19.5	5.3
Ash Fusion Temperature (Softening)	2,340	2,280	2,200	2,290	2,250
Hardgrove Grindability	101	52	n. a.	35-70	55
Base/Acid Ratio	0.27	0.44	. 0.30	1.53	0.43

TABLE 3

Lignite Sample Descriptions

Run ID#	Sample #	Sample Location	Feedstock Condition	Grind Medium	Nozzle Psi	Pulverizer Temp (avg)	Moisture %	Ash %
1	82-123	Feedstock	as-mined	air	40	200°F	43.70	13.04
	82-126	Cyclone					30.44	13.36
	82-127	Tank 2					33.33	13.97
2	82-135	Cyclone	air-dried	air	40	200°F	31.32	12.04
	82-137	Feedstock					45.59	11.02
3	82-141	Feedstock	as-mined	steam	40	277°F	44.99	9.38
	82-143	Tank 1					14.44	17.80
4	82-157	Tank 2	air-dried	steam	40	221°F	17.69	15.08
	82-159	Feedstock					46.71	9.51
5	82-161	Tank 2	as-mined	air	65	200°F	28.57	14.12
	82-162	Feedstock					43.95	10.77
6	82-152	Tank 2	air-dried	air	65	200°F	20.15	20.84
	82-153	Feedstock					40.68	15.08
7	82-148	Tank 1	as-mined	steam	65	224°F	56.63	6.87
	82-149	Feedstock					46.63	10.70
8	82-146	Tank 1	air-dried	steam	65	218°F	65.91	8.06
	82-155	Feedstock					33.25	12.12

TABLE 4

Results of Study of Micropulverization of Mississippi Lignite

Sample Number	82-124	82-136	82-125	82-135
Description	As-mined	Air-dried	As-mined, air-pulverized	Air-dried, air-pulverized
As-received basis:				
Moisture, %	42.9	44.0	29.5	26.6
Ash, %	13.1	12.1	13.7	13.8
Volatiles, %	30.6	28.1	37.2	37.8
Fixed Carbon, %	13.4	15.8	19.5	21.8
Higher heating value, Btu/lbm	5300	4966	7452	7561
Dry basis:				
Ash, %	22.9	21.6	19.4	18.8
Volatiles, %	53.6	50.2	52.8	51.5
Fixed Carbon, %	23.5	28.2	27.8	29.7

TABLE 5

Micropulverized Mississippi Lignite Centrifuged Float-sink Results

Specific Gravity	Weight, Grams	Weight, %	Ash, %	Pyritic Sulfur, %	Total Sulfur, %
Sample 03 (Starting Weight 202 g)					
1.30 FL	16	8.5	13.8	.03	.64
1.30 x 1.40	32	17.2	14.9	.01	.51
1.40 x 1.60	116	61.4	17.6	.01	.45
1.60 x 1.80	17	8.9	23.5	.01	.45
1.80 SK	8	4.0	59.3	.49	.54
	189	100.0	19.0	.03	.52
Sample 04 (Starting Weight 199 g)					
1.30 FL	6	3.7	11.7	.02	.39
1.30 x 1.40	30	18.6	13.5	.01	.44
1.40 x 1.60	92	56.4	16.4	.02	.48
1.60 x 1.80	29	17.4	23.9	.01	.35
1.80 SK	6	3.9	61.3	.39	.67
	163	100.0	18.7	.03	.45

NOTE: Particles that escaped through the filters after float-sink testing were left on the pan as a film after evaporation of the heavy liquid. This material was not recoverable.

As reported by R. Killmeyer of DOE-PETC, January, 1983

TECHNICAL STATUS REPORT FOR
MMRI GRANT NO. 83-1OS

Micropulverized Lignite Drying

Graduate Student: Stephen Savelle
Directed by : Dr. W. G. Steele

While lignite has been mined and utilized on a regional basis for some years, the present energy situation has prompted interest in expanded use of lignite as an energy source. With this increase in interest has come an increase in the research and development effort devoted to lignite. This includes the adaptation of high-rank (bituminous) coal technology, such as mining and handling, and the study of problems unique to lignite, e.g. beneficiation or combustion. (1)

Of the various research areas, beneficiation is perhaps the most important in bridging the gap in fuel rank when considering lignite as a replacement energy source. Beneficiation is a general term, including everything from initial size reduction through drying and physical cleaning to chemical removal of impurities. Conventional size reduction techniques are well established, and chemical processes for lignite appear to be the farthest from commercial application, leaving physical cleaning and drying as the focus of most research effort. Since the largest inert in lignite is water, drying has the greatest potential for beneficiation. (2) This is not to say cleaning is unimportant; wide ranges of ash content in lignite mandate cleaning for fuel consistency.

One technique for coal preparation shows promise in allowing improved beneficiation. Micropulverization, as in Ergon’s fluid energy mill, reduces coal particle size to undei thirty microns. There are several advantages to this method over conventional grinding. A larger area of the coal is exposed to aid in thermal drying. In fact, micropulverization itself has a noticeable drying effect on lignite due to surface area increase. Also, most or all of the ash is structurally freed from the coal, allowing more complete physical cleaning. Preliminary studies by others (1), verified by research at Missisippi State University (3), show that micropulverized lignite has a distinct size difference between the coal particles and the ash particles which could be the basis for an effective physical cleaning technique, e.g. heavy-medium cycloning. This technique is being studied at MSU currently.

As mentioned above, drying has great potential for coal beneficiation. Moisture in lignite consists of surface moisture in coal pores and inherent moisture bound in the molecular structure. Thermal drying is the only practical means of removing surface moisture and part of the inherent moisture (4). There is interest in the observation that the inherent moisture, once removed, appears to be unable to rebind with the coal molecules, thus becoming the more easily removed surface moisture. This observation, if ture, could be used to develop a high solids concentration slurry, using freed inherent moisture as part of the required water.

The use of micropulverized high-rank coals in a slurry has been shown to be feasible, and even beneficial (5). The use of lignite in slurries is made feasible by beneficiation techniques, with the removal of inherent moisture in the lignite one of the most important factors in achieving the solids concentration necessary for economic use of a lignite-water slurry. This warrants further studies in the removal of lignite inherent moisture and the properties of a slurry prepared with this lignite.

The main problem that is being addressed in this project is the reabsorption of the inherent moisture by the lignite particles during the slurry transport. Based on the findings from laboratory tests on reabsorption, the economics of the various drying techniques will be investigated.

In order to obtain a reasonable approximation for the cost of dried, pulverized lignite fuel, the overall process will be investigated from mining, drying and pulverization to water slurry transportation and power plant use. Assistance will be obtained from Ergon, Inc. and other informed sources in order to make reasonable estimates for the items other than drying. In addition to fuel costs, approximations will be made for capital equipment modifications necessary for the use of ultrafine lignite fuel.

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Micropulverized Lignite Beneficiation

Graduate Student: Shahyar Pirzadeh
Directed by : Dr. C. W. Bouchillon

The purpose of this research is to develop a system for mechanical beneficiation of micropulverized lignite. Such beneficiation systems typically use physical or mechanical methods to remove undesirable impurities from run-of-mine coal. These may also be useful in lignite beneficiation. Since there is a large density difference between the organic coal substance (hydrocarbons) and mineral matter (ash particles) associated with it, a gravity separation method may be effective in cleaning and improving the quality of a raw coal or lignite. The conventional hydrocyclone is a device which utilizes the concept of gravity separation and separates clean coal from heavier particles (ash) by centrifugal forces in a vortex.

The objective is to conduct an experimental study to design, fabricate, test, and evaluate the effectiveness of a beneficiation system using a hydrocyclone as the separation device. The test facility being utilized in this investigation will be constructed at Mississippi State University. It is a closed loop system consisting of a three inch cyclone furnished by CE-Bauer of Springfield, Ohio, a 1½ horsepower centrifugal pump which circulates a carrier fluid with a low viscosity and high specific gravity such as Refrigerant (Freon) 113, piping made of PVC piping, globe valves, an orfice (or venturi) flow meter, and a steel container to collect the underflow and the overflow of the cyclone (Fig. 1).

The study will be accomplished in the following three major parts.

1. A preliminary study of the problem, background, and literature review of beneficiation methods, cyclone design, and analysis of the fluid flow in the cyclone.

2. Test and data collection; including, heat of combustion evaluation, size distribution estimation using optical and electron microscopes, and separation tests. The heat of combustion and size distribution estimation will be carried out both before and after the cleaning (separation) process.
3. Data analysis, efficiency evaluation, and conclusion.

A summary of activities which have been accomplished is given below.

1. Some information, papers, articles, and books related to coal beneficiation methods and theory, application, and design of hydrocyclones have been collected and studied in order to prepare a literature review of the subject.
2. Using an adiabatic oxygen bomb calorimeter, a calorific value test was carried out on four different samples of lignite (as mined and pulverized) and the heat of combustion was determined for each sample.
3. A preliminary design of the experimental apparatus was prepared, and the appropriate test facility components were selected and acquired.
4. A calculation of flow losses in the system was made to determine the pressure drop and the pressure available at the inlet of the hydrocyclone.

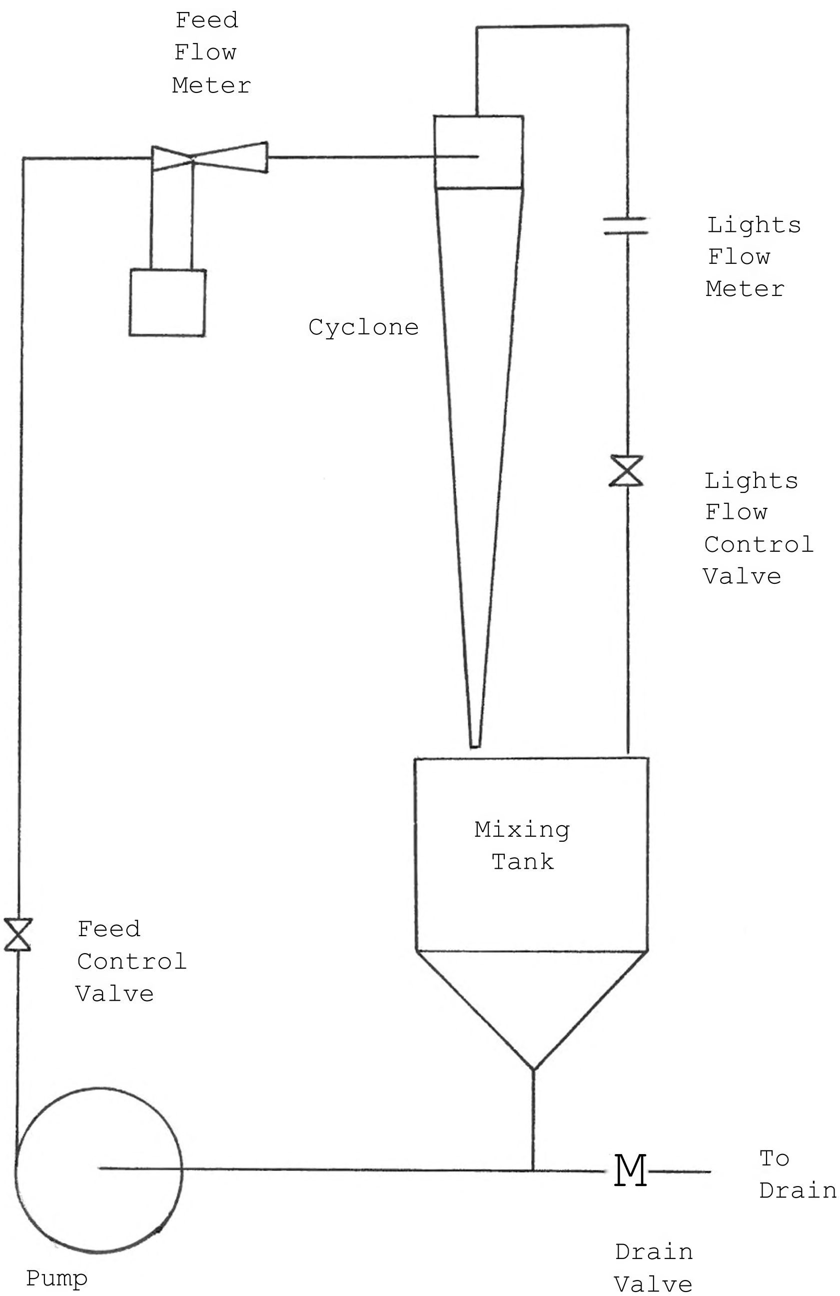

Figure 1. - Schematic of Experimental Apparatus for the Proposed Project