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SOLVENT REFINING OF FOSSIL RESIN FLOTATION CONCENTRATE FROM WESTERN COALS

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ABSTRACT

Macroscopic fossil resin from the western coal fields is a unique resource in the United States. Such resinous coals are found in the states of Arizona, Colorado, New Mexico, Utah, Washington, Wyoming, etc. Among these, the Wasatch Plateau coal field in central Utah has a particularly high content of macroscopic coal resin. Many seams in this coal field have been reported to contain as much as 5% resin by weight. This fossil resin varies in color from lemon yellow to dark brown and has a market value of $0.40-0.70/lb, depending on its color and softening point. It is used as an additive in the adhesives, rubber, varnish, paints, coatings, and thermoplastics industries, and particularly in the ink industry.

Fossil resins have been recovered intermittently from the Utah coal field since 1929 by gravity and/or flotation processes. Resin concentrates thus produced can be refined by solvent extraction with the resin product finally recovered by evaporation of the solvent. Production, nevertheless, has been on a very small scale and the technologies used have limited the development of a viable fossil resin industry. Because of the lack of technology for the efficient recovery of resins from coal this valuable resource has been wasted, being burned together with coal for electric power generation. It is estimated that the fossil resin from the Wasatch Plateau coal field burned each year for electric power generation has a value of $100 million - equivalent to the value of the coal itself!!

In view of this situation, the University of Utah and Advanced Processing Technologies, Inc., with support from the US DOE, have initiated a research program on the continuous refining of these fossil resin flotation concentrates. The program includes batch extraction kinetics, product characterization, circuit design and construction, and continuous refining of the resin concentrate. It is expected that the successful completion of the project will provide much-needed proof-of-concept data for fossil resin refining and ultimately the establishment of a fossil resin industry.

INTRODUCTION

Certain bituminous coals of the Western United States are known to contain appreciable quantities of macroscopic fossil resin (resinite).[1-3] Such resinous coals are found in the states of Arizona, Colorado, New Mexico, Utah, Washington, Wyoming, etc. Among these, the Wasatch Plateau coal field in central Utah is of special value because of its particularly high content of macroscopic fossil resin and the resin's specific physical/chemical properties.[4,5]

Fossil resin is derived from terpenoid plant resins that have polymerized in situ and is of predominantly aliphatic character with high hydrogen and carbon content and low oxygen, nitrogen, and sulfur content when compared to the parent bituminous coal.[6,7] Fossil resin from Utah coal generally exhibits low density, a range of colors (mostly brown color), and good solubility in hexane and/or heptane.[6,7] It has been recovered intermittently on a small scale from the Utah coal field since 1929 by gravity and/or flotation processes. The resin concentrates thus produced are refined by solvent extraction. The resin particles show a basic amber color and

contain inclusions of what appear to be fine coal colloids. As the fine coal inclusions increase in the resin matrix, the darker the coal resins appear. Thus solvent refining is required to remove the fine coal inclusions and dark-color inducing compounds (hexane insoluble) from the resin concentrate to produce a premium resin product. Solvent-purified resins from the Wasatch Plateau coal typically have a molecular weight of about 1200 and a softening point of about 170°C. This product, at the present time, has a market value of about $0.40-0.70/lb as a chemical commodity and can be used in the adhesives, rubber, varnish, paints, coatings, and thermoplastics industries, and particularly in the ink industry.[6,7]

Because of the lack of technology for the efficient recovery of resins from coal this valuable resource has been wasted, being burned together with coal for electric power generation. It is estimated that the fossil resin from the Wasatch Plateau coal field burned each year for electric power generation has a value of $100 million - equivalent to the value of the coal itself!! The waste of this valuable resource is evident. In view of these factors, the waste of a valuable resource and the special product quality, Professor J. D. Miller and his research group at the University of Utah have made significant efforts to develop technology for a fossil resin industry in the western coal fields. As a result of these efforts, several new flotation technologies have been developed. Two U. S. patents were granted in 1988 [8,9] and another is pending.[10] Nevertheless, the development of a fossil resin industry remains uncertain. The primary reason for this situation is that coal producers who own this valuable resource are not convinced that such a venture to develop a fossil resin industry would be profitable. The research efforts and the technology development for the refining of resin concentrate are needed. In this regard, the University of Utah was awarded a DOE contract to support a proof-of-concept continuous resin refining research program that was administrated by the Utah Engineering Experiment Station and involved both the University of Utah and Advanced Processing Technologies, Inc. (APT). The research program includes batch extraction kinetics, product characterization, circuit design and construction, and continuous refining of the resin concentrate. It is expected that the successful completion of the project will provide much-needed proof-of-concept data for fossil resin refining and ultimately the establishment of a fossil resin industry.

PHYSICAL/CHEMICAL CHARACTERIZATION OF RESIN CONCENTRATE

Fossil Resin Flotation Concentrate

Under a previous DOE-funded program, University of Utah and APT conducted a series of pilot-plant tests on the selective flotation of fossil resin from Wasatch plateau coal (both UPL coal and CO-OP coal mines) of south central Utah. About 200 lbs high grade fossil resin flotation concentrate (approximate 75% resin content) was generated from those pilot-plant flotation tests. The resin flotation concentrates were naturally dried, sampled and stored in one-kilogram plastic bags for future research use. The ash and moisture contents of the resin concentrate were found to be 1.23% and 1.03% by weight respectively.

Particle Size Distribution

The particle size distribution of the resin concentrate (Table 1) was determined by wet screening according to ASTM standard procedure (D 4749). It is evident that the resin concentrate has a relatively fine particle size distribution with more than 80% (by weight) being less than 200 mesh (74 microns) and about 64% (by weight) less than 38 micron (400 mesh). This tendency is attributed to the fact that fossil resin from Wasatch Plateau coal field is friable and is easily fractured into fines during mining, transportation, and preparation. For example, it is not unusual to find that the minus 28 mesh coal streams from a coal operation contain more

than 10% hexane-soluble resin, even when the run-of-mine coal contains only 3% resin. Such a fine particle size should be favorable for subsequent solvent refining due to the large surface area.

Table 1. Size distribution of the resin concentrate.

Particle Size Range		Weight	Cumulative weight	
(mesh)	(μm)	(%)	Plus (%)	Minus (%)
28-60	600-250	1.79	1.79	100.00
60-100	250-150	4.24	6.03	98.21
100-200	150-75	13.74	19.77	93.97
200-270	75-53	7.71	27.48	80.23
270-400	53-38	8.71	36.19	72.52
-400	-38	63.81	100.00	63.81
Total		100.00		

Petrographic Characterization

Since fossil resin fluoresces under blue light, the petrographic characteristics of resin types and coal component in the resin concentrate can easily be identified and classified by fluorescence microscopy. Under blue light fossil resin grains can be distinguished into four groups: green, green-yellow, yellow-orange and orange-brown which are similar to the hand-sorted resin types of yellow, amber, light-brown and dark-brown resin. The polished briquettes (pellets), in which the particles of the resin concentrate were mounted, were examined using an Axioplan Universal Microscope manufactured by Carl Zeiss, West Germany. For quantitative petrographic analysis, more than three hundred particles were counted. It was found from this petrographic analysis that the resin concentrate consists predominately of green-yellow (28.64%) and yellow-orange resins (34.97%) with some green resin (12.62%) and orange-brown resin (11.04%). A significant amount (12.72%) of fine coal particles are also found in the resin flotation concentrate.

BATCH SOLVENT EXTRACTION STUDY

Preliminary Solvent Extraction Tests

Fossil resin is a complex mixture of sesquiterpenoids and the solubility of these resin compounds depends on the solvent used. The determination of the extractable resin content in the concentrate by different solvents will provide an important criterion for the solvent refining and purification, process design, and to determine the quality of the refined resin products. The extractable resin content was determined with four solvents: ethyl acetate, hexane, heptane, and toluene. The extraction tests were conducted in a TX-6 Soxhlet extraction unit. In the tests, approximately 1-2 grams of the resin sample were placed in a single-thickness cellulose thimble with 60 to 70-ml of solvent. The extraction was carried out for at least 2 hours at boiling point of the solvent and then rinsed for another 4 hours. The results are given in Table 2.

It can be seen from Table 2 that the resin concentrate has the highest solubility in toluene. In most cases, the larger particles in the resin concentrate exhibit greater solubility. This is due to the fact that the coal contaminant is found in the fine size of the resin concentrate.

Table 2. Extracted results for various size with four solvents.

Size (mesh)	Extracted weight	Solvent			
		Ethyl Acetate	Hexane	Heptane	Toluene
28-60	(%)	53.63	89.13	90.98	94.63
60-100	(%)	52.05	90.54	91.72	94.42
100-200	(%)	56.03	87.67	87.94	91.74
200-270	(%)	62.15	82.33	81.92	87.84
270-400	(%)	50.54	78.72	79.17	83.97
-400	(%)	63.44	66.85	66.24	73.92
Total	(%)	60.54	73.34	73.72	79.56

Characteristics of the Extracted Resin Products

The extracted resin products as obtained from preliminary solvent extraction tests on the composite resin concentrate were characterized in terms of their melting point, density, and chemical composition. The melting points of the extracted resin products were determined using 8100 Series Digital Melting Point Apparatus of Electrothermal Engineering Limited. Density measurements were carried out using the Autopyconometer 1320. The results from the composite concentrate are shown in Table 3.

Table 3. Characteristics of solvent extracted resin products from composite concentrate.

Solvent used for extracting resins	Melting point of extracted resin products (°C)	Density of extracted resin products (g/cm^3)
Ethyl acetate	141 - 142	1.048
Heptane	178 - 180	1.036
Hexane	140 - 142	1.034
Toluene	175 - 178	1.048
Resin concentrate	196 - 198	1.143

It should be noted that the melting point of heptane-extracted resin is higher than that of hexane-extracted resin. The increase in carbon chain length of the solvent results in a rise of the melting point for the refined resin products. Therefore, some long chain solvents such as nonane or decane should be considered as the solvent for resin refining. The results from elemental analysis are listed in Table 4. The ethyl-acetate extracted resin was found to contain more oxygen, whereas both hexane- and heptane-extracted resins were found to contain less oxygen than the product obtained from toluene extraction. The other elements were found to be quite constant for all products. However, the color of ethyl acetate extracted resin, hexane extracted resin, and heptane extracted resin were found to have a light-yellow color while the toluene extracted resin was found to be significantly darker. Because light-colored or yellow resin is preferable and of greater commercial value than the dark-colored resins, particularly in the ink industry, solvent

refining is a necessary step to purify resin concentrates and produce a light-colored resin product. Therefore, the identification of color inducing compounds in coal resins and their characterization are of great commercial interest.

Table 4. The elemental composition of resin concentrate and various products recovered from extraction with indicated solvent

Solvent	Element content %					Atomic ratio	
	C	H	N	S	O	O/C	H/C
Ethyl acetate	85.79	10.57	0.19	0.29	3.16	0.0276	1.48
Heptane	86.88	10.85	0.17	0.30	1.80	0.0155	1.50
Hexane	86.79	10.84	0.16	0.40	1.81	0.0156	1.50
Toluene	86.49	10.63	0.20	0.30	2.38	0.0206	1.47
Resin concentrate	84.66	9.57	0.45	0.35	4.97	0.0440	1.36

Solvent Extraction of Fossil Resin by Hexane/Toluene

In this case, solvent extraction of the fossil resin concentrate was carried out first by hexane then followed by toluene at room temperature. Table 5 lists the measured density, molecular weight, and proximate analysis of the two soluble fractions as well as the toluene insoluble residue. The number average molecular weights of the hexane and toluene extracted resins were determined by Vapor Pressure Osmometry (toluene as solvent). It is evident from Table 5 that most of the resin concentrate (61.3%) is hexane soluble and shows a yellow color, low density, high volatile matter, and low molecular weight. The hexane insoluble but toluene soluble resin (31.8%) has a darker color, higher density, slightly lower volatile matter, and a higher molecular weight. The toluene insoluble residue resembles coal and has a relatively high density (1.205 g/cm^3), ash content (3.1%), and very low volatile matter (58.9%).

Table 5. Preliminary analysis of hexane/toluene extracted resins from resin concentrate

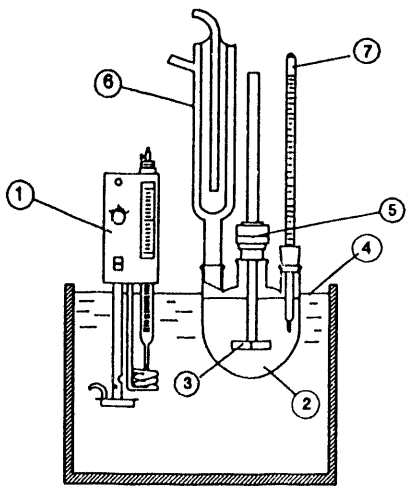
Extracted Fraction	Weight (%)	Ash (%)	Volatile Matter (%)	Density (g/cm^3)	Molecular Weight
Hexane soluble	61.3	0.0	100.0	1.023	1270
Hexane insoluble					
Toluene soluble	31.8	0.0	98.7	1.068	1700
Toluene insoluble	6.9	3.1	58.9	1.205	n.d.

It is also evident from Table 5 that toluene is a stronger solvent for resin extraction than hexane, and that a higher percentage of the resin can be recovered. Such a high extraction is expected due to the fact that hexane is a non-polar solvent while toluene is weakly polar with a permanent dipole and π-electron system that provides for a stronger interaction with resin. On the other hand toluene, being a more polar solvent, also extracts extraneous polar molecules, such as alkyl-substituted benzenes, naphthalenes and other aromatic compounds, imparting a darker coloration to the extracted resins as evidenced from FTIR and pyrolysis GC/EIMS analysis.[7]

Kinetics of Resin Extraction by Heptane

This research was designed to be conducted in such a way as to provide a fundamental understanding of the fossil resin refining process and to determine major factors which control resin extraction. Of course, the ultimate objective of the batch extraction study is to provide the basis for subsequent design and construction of a continuous resin refining circuit. A series of experimental tests were undertaken to determine the effect of temperature and particle size on resin extraction at low solids concentration. Resin extraction kinetics were determined by analysis of extracted resin solutions taken at periodic time intervals during the extraction process. Technical grade heptane (C_7H_{16}) was used as solvent for the kinetics study. Four monosize samples were prepared by wet screening of the resin concentrate. The heptane-soluble resin contents in all samples were determined with a TX-6 Soxhlet extraction unit at the boiling point of heptane (98 ^{0}C) for about 6 hours and the results are presented in Table 6. As shown in Figure 1, all resin extraction tests were carried out in a one liter round bottom 3-neck distillation flask. A thermometer, a condenser, and a stainless impeller were placed into the flask through the three neck openings. The impeller was connected to a motor through a Chesapeake stirrer connection at the center port. The agitation of resin/solvent solution was accomplished with a impeller/motor and control system to set the speed of rotation. The flask was submerged in a circulating water bath to control the extraction temperature with an accuracy of ±0.5 ^{0}C.

One liter of pure heptane was placed into the flask with about 1.1 to 1.5 grams of the resin sample at a desired temperature for each test. During the extraction process, approximately 3 ml of resin solution was taken from the suspension at pre-set time intervals by a syringe equipped with a prefilter. The heptane solution was analyzed for resin concentration by UV/Vis spectroscopy. All the data presented in the following sections were normalized on the basis of



1. Bath temperature controller
2. Extraction flask
3. Agitation impeller
4. Water bath
5. Chesapeaks connector
6. Solvent condenser
7. Temperature monitor thermometer

Figure 1. Schematic drawing of the resin extraction apparatus

Table 6. Heptane-soluble resin content of monosize samples

Monosize sample (mesh)	8x10	28x35	48x60	100x150
Heptane-soluble resin content (wt.%)	95.45	92.92	88.67	90.46

the heptane-soluble resin content of each monosize sample from the flotation concentrate.

The effect of particle size on the resin extraction rate at 20 °C and 500 RPM is presented in Figure 2. It is evident that the extraction rate decreases with an increase in extraction time and that there is a strong dependence of resin extraction rate on particle size. The extraction rate was found to decrease dramatically with an increase in particle size. For example, after 40 minutes of extraction, the percentage dissolved was approximately 20% for the 8x10 mesh resin particles while about 80% dissolved for the 100x150 mesh resin particles. After 2 hours of extraction, the 100x150 mesh sample had reached 92% of its ultimate extraction whereas only 40% had been extracted from the 8x10 mesh sample after 2 hours. The rate shows an inverse 1st-order dependence on particle size typical of diffusion or surface reaction controlled kinetics.

A number of resin extraction tests were performed at different temperatures in order to determine the effect of temperature on the extraction rate. The resin extraction rate was found to increase with an increase in extraction temperature. The effect of temperature is very significant. The results are presented in Figure 3 for the 48x60 mesh sample where it is evident that the extraction rate rises significantly with temperature. Almost complete extraction was observed in 20 minutes at 60 °C while only 25% extraction was observed at 0 °C. As expected higher temperatures should be considered for the continuous extraction circuit in order to maximize yield for a short extraction time. An activation energy of 15.5 kJ/mole can be calculated based on the initial extraction rates. The magnitude of the activation energy indicates that the extraction process is mainly controlled by a surface reaction mechanism involving the dissolution of resin molecules into the heptane solution. As such, energetics of associated solvation reactions are expected to account for the observed activation energy.

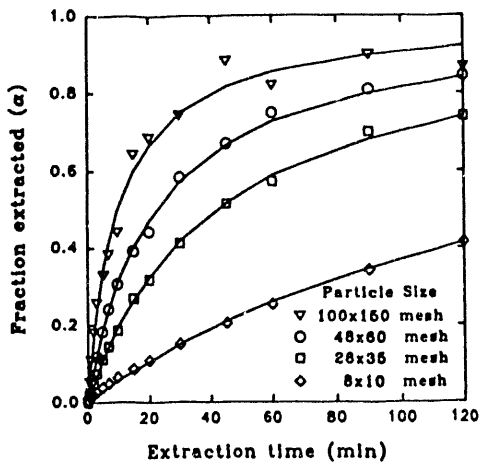


Figure 2. Effect of resin particle size on the rate of extraction with heptane at 20 °C.

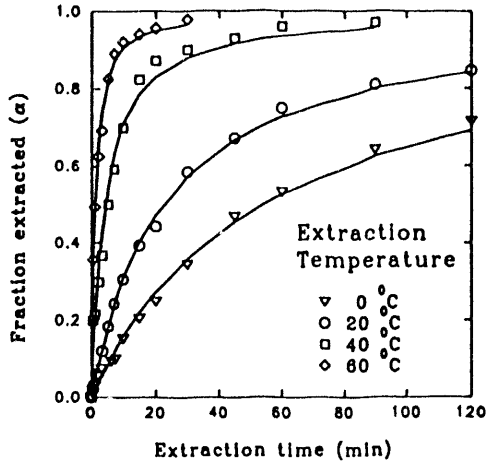


Figure 3. Effect of temperature on the rate of extraction with heptane for the 48x60 mesh sample.

DESIGN OF CONTINUOUS RESIN REFINING CIRCUIT

Based on the kinetic study and environmental/safety requirements for the solvent extraction operation, the continuous resin refining circuit was designed to provide health and safety compliance, high efficiency, compact size, and easy operation. The complete refining circuit includes resin feeder, solvent feed and circulation, extraction equipment, residue separator, spray dryer and product collection, compressed air-heater, and solvent recycling system.

SUMMARY AND CONCLUSIONS

Solvent extraction studies indicate that two major factors contribute to the natural color variation of the fossil resin: (1) relative abundance of chromophores (mostly heteroatoms and unsaturated compounds); and (2) finely dispersed inclusions of colloidal coal macerals. It has been found that the color of the resin changes from light to dark with an increase in the degree of unsaturation, heteroatomic content (O, N, S), and molecular weight, as well as with an increase in the colloidal coal maceral inclusions.

The rate of resin extraction from the resin concentrate is significantly affected by both particle size and extraction temperature. The finer the particle size the higher the extraction rate. The rate of heptane extraction will significantly increase with an increase in extraction temperature (from 0 °C to 60 °C). Therefore a moderate extraction temperature (about 60 °C) should be considered for the continuous extraction circuit in order to maximize yield for a short extraction time.

Process technology development for the refining of resin concentrates to produce a premium resin product is in progress at the University of Utah. Nevertheless, the development of a fossil resin industry still remains uncertain. It is clear that the creation of a fossil resin industry is no longer limited by process technology. Nor is it limited by economic considerations. Other peripheral factors appear to account for the reluctance of the Utah coal industry to move forward on this initiative.

ACKNOWLEDGMENTS

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RECOVERY AND UTILIZATION OF FINE CLEAN COAL IN A THERMAL DRYER SYSTEM

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ABSTRACT

Two specific problems exist at a large number of coal preparation plants in the United States that use thermal dryers for producing product coal, cyclones for first-stage recovery of coal fines, and second-stage wet scrubbers to remove coal carry-over from the dryer exhaust gas. The first problem involves a need for eliminating the common practice of sacrificing clean ultra-fine coal captured in the scrubbers. The second problem involves a need for mitigating over-dry fine coal dusting from in the dryer product.

The second problem, controlling fine coal dusting, has been met by applying a solution of surfactants and process water to the over-dry coal fraction, de-dusting the product coal. To date, the problems associated with the recovery and use of fine clean coal from dryer scrubber effluent have not been solved. The program, reported in this paper, demonstrates a simple process improvement, involving use of a belt press, that will simultaneously solve both the de-dusting and the dryer scrubber effluent recovery issues.

This program proposes to use a combination of a clean coal thickener with a squeeze belt press to recover the ultra-fine coal in dryer scrubber effluent before it is mixed in with the balance-of-plant tailings. As an additional essential part of this program, we propose to demonstrate that the coal-water mixture (CWM) produced from the scrubber sludge of a thermal dryer can be used as a dust suppressant. The net effect of these two coal circuit changes will be to integrate the thickener underflow into the thermal dryer circuit. This will essentially close the loop and permit maximum efficiency from the system, by recycling a former waste stream (sludge) as an effective dust suppressant.

DISCUSSION OF THE PLANT OPERATIONS PROBLEM REGARDING COAL FINES

With any coal drying system a small amount of low ash, ultra fine coal is collected in wet and/or dry exhaust gas cleaning processes. Coal fines collected in dry particulate collection systems are usually recycled and mixed with the dry product coal, resulting in a dust generation problem. Dilute wet scrubber effluent is generally sent to a thickener; the resultant underflow sludge, containing clean coal, is sent to settle in on-site ponds.

Historically, the effluent from coal thermal dryer exhaust gas scrubbers has been mixed with tailings from the coal cleaning plant. Consequently, this low-ash coal becomes mixed with and diluted by a much larger flow of high ash tailings. There has been insufficient economic return to justify recovering clean coal from such combined thickener underflow streams or impoundments.

Recently, however, increased quantities of fines have been produced (or predicted to be produced) in coal preparation plants. These underflow fines have come from the increased use of mechanical mining equipment, which results in the use of wet cleaning methods to clean this fine coal fraction. Predictions of increased fines production have come due to the 1990 CAA amendments, which portend greater deep cleaning of coal. Consequently, recovery and use of these increased coal fines will eventually become an economic necessity.

Figure 1 illustrates a conventional coal processing plant flow sheet, dividing the plant into a thermal dryer circuit and the balance of plant. Process water produced in the thickener is needed for both wet scrubber and de-dusting operations in the dryer circuit, as well as in the balance of plant. Ultra-fine clean coal mixture is returned to the central continuous thickener, where it is mixed with high-ash tailings from the balance of plant. The resultant high-ash sludge is pumped to a clarification pond for final dewatering.

Figure 2 details the improved plant flow sheet. The fundamental improvement in this second design involves segregating the clean coal fines mixture from the tailings mixture, instead of mixing them in a common thickener. This separation of recoverable coal from high-ash tailings allows a new, fine coal dewatering loop to be added, ultimately producing a concentrated coal-water mixture (CWM) that is used to de-dust the over-dry thermal dryer product. Equipment used in the new dewatering loop consists of a fine coal thickener, which serves as both a scrubber holding tank and a first-stage concentrator; followed by a belt press, which performs the final stage of dewatering. Only minimal changes are required to utilize CWM in place of water as the de-dusting agent. This flow sheet will essentially close the loop and permit maximum efficiency from the system, by recycling a former waste stream (sludge) as an effective dust suppressant.

In summary, an improved coal preparation plant utilizes a thickener/belt press combination to dewater an ultra-fine clean coal mixture. This commercially viable, low-risk process improvement will simultaneously solve two major fine coal issues that apply to all thermal dryer loops: a requirement for de-dusting the over-dry fine product coal, and finding a viable means for recovering and using clean ultra-fine coal from dryer scrubber effluent. This widely applicable process involves a thickener/belt press system for producing concentrated CWM, then uses the product CWM as the de-dusting agent. A private sector coal producer already has their dryer scrubber effluent segregated in a separate, clean-coal thickener, and is ready to perform a full-scale proof-of-concept (POC) demonstration of this thickener/belt press/CWM de-dusting process at their plant.

Objectives

It is the objective of this program to successfully demonstrate the utilization of thickener underflow-based CWM for dust suppression at the preparation plant, at scales up to 7.3 metric ton/hr (8 ton/hr) of CWM. This scale of testing will provide meaningful results for coal preparation plant operators to evaluate this technology for their particular plant's operation.

Discussion of Existing System

The coal preparation plant at JWRI Mine Number 4 has a design point capacity of 544 metric tons per hour (600 short tons per hour) of clean coal. The coal cleaning circuits are operated continuously on a five day work week. Most in-plant coal transporting is done by 0.91 meter-wide (36 inch-wide) conveyor belts. Raw coal is delivered to the 22,700 metric ton (25,000 short ton) stacker, where it awaits transfer to the preparation plant. In the plant, sizing, cleaning, and drying operations are conducted to produce the required product coal specifications.

The dryer inlet coal moisture content is currently 13%. The fluidized bed dryer evaporation rate is 36.3 metric ton/hr (40 short ton/hr) of water, starting with 680.3 metric ton/hr (750 ton/hr) of wet feed. Dry product coal contains 7% moisture, resulting in the design dryer production rate of 644 metric ton/hr (710 ton/hr) of coal. Design thermal input to the dryer is 44 MW (150 MMBtuh). The dryer is fueled by a portion of the dust collected by the cyclones.

Dry fine product coal collected in the four cyclones is routed to two screw conveyors. A portion of this fine coal passes through other screw conveyors to the burner system of the plant. The balance is de-dusted with a chemical/water solution, then passes through chutes to the dryer product conveyor. Dry product is moved by belt to the 22,700 metric ton (25,000 short ton) dry product stockpile. Ample provisions are made for truck and future rail loadout at the site.

Relative to the preparation plant's dryer subsystem, the static thickener serves as the holding tank for the exhaust gas scrubber liquor, as well as providing clear water for the scrubber system. Figure 3 details the interconnection between the thermal dryer, cyclones, wet scrubber, and thickener. Ultimately, clean coal fines concentrated in the thickener are pumped to one of three clarifying ponds. JWRI currently pays removal costs of $3.31/metric ton ($3/ton) to "muck out" coal fines for ultimate disposal.

Discussion of Proposed System

The proposed change in the process will be to install a belt press after the existing static thickener, as shown in Figure 4. This thickener/belt press combination will recover ultra-fine coal from the thickener underflow, thereby recovering and recycling previously wasted coal fines that would have otherwise been sent to the adjacent pond.

Figure 5 illustrates the arrangement of the additional oversize protection sieve, concentrated CWM holding tank, and pumps required to complete the system. A plant-specific reason necessitates an oversize protection sieve for this process: coarse coal conveyors travel over the thickener, and could possibly deposit oversize material in the system. This sieve will protect the belt press from excessive wear and associated increased maintenance requirements. The CWM holding tank is supplied to even-out the expected process-related variations.

Resultant CWM from the new thickener/belt press system will be used as de-dusting fluid for the two existing 0.51 meter (20-inch) diameter mixing screws, thereby using the coal fines to agglomerate with and de-dust the fine dry coal product. Four to five atomizers will be used in each screw, depending on the final CWM properties.

Program Goals

The goals of this program are to address the problems associated with the integration of fine coal circuits with the balance of the plant, by performing a POC demonstration of the technical advantages and improved economics resulting from using a thickener/belt press ET to dewater and recycle coal fines at a commercial coal preparation facility.

The technical advantages of the thickener/belt press equipment that will be demonstrated include:

- Elimination of 3.6 metric ton/hr (4 ton/hr) of coal fines from JWRI settling ponds, by recovering and recycling fines into the dry product coal.

- Production of pumpable slurry in a simple, once-through process, without a need to bypass and remix raw feedstock with the dewatered product.

- Continuous equipment operation under a variety of operating feed conditions.

- Easily controlled slurry product moisture content, which will be essential for proper control of the de-dusting fluid application process.

- Low plant space requirements, essential for these retrofit installations.

- Low and easily performed maintenance requirements.

The improved economics from commercially recovering coal fines with a thickener/belt press combination are expected to include:

- Maximizing the net production and sales of useful coal from the plant, by recovering and reusing previously wasted low ash, ultra fine coal.

- The use of processed scrubber effluent for de-dusting purposes will reduce the flow rate of scrubber sludge to the settling ponds, which by lowering demands on the plant's clarification system will reduce the associated operating costs.

- A potential reduction in the requirements for de-dusting surfactants and polymers.

In summary, this program will demonstrate the technical and economic benefits that will accrue due to the use of a thickener/belt press system on coal fines. Without increasing the manpower requirements of the plant, addition of the thickener and belt press will increase coal production, thereby increasing coal sales, providing an economic boost to the plant's operation.

System Performance Prediction

The proposed thickener/belt press system will increase the production of JWRI Mine Number 4 by approximately 1%. This production increase will be accomplished by recovering and re-using 3.6 metric ton/hr (4 ton/hr) of coal fines that were previously sent to holding ponds, returning this as a 50% CWM to de-dust the 644 metric ton/hr (710 ton/hr) of existing dryer production.

Mixing of the recovered slurried coal fines with the product coal will occur in an existing mixing screw. This process will use the recovered CWM for providing dust suppression on the over-dry portion of the product coal. This use of CWM will eliminate the existing practice of de-dusting with mixed process water/surfactant solution, reducing the plant's process water usage by 60 lpm (16 gpm).

An improvement in effluent water clarity will result from use of the thickener/belt press system. This improvement will occur due to the elimination of the 3.6 metric ton/hr (4 ton/hr) of thickened solids being sent to the clarification ponds from the thickener, along with the 344 ℓ/min (91 gal/min) of associated wastewater.

System Cost Analysis

Modern day coal mining and preparation facilities are capital-intensive. For example, a $750,000,000 capital investment of the JWRI Mining Division is required for the annual production of 9.1 million metric tons (10 million tons) of clean coal. This investment represents an average of $83 for each metric ton-per-year ($75/short ton-per-year) of annual coal production. The CWM/de-dusting system cost analysis shows that not only is the proposed system much less capital intensive than JWRI's average investment, but simple payback periods range between approximately one-half to one year.

The proposed full-scale fine coal recovery/de-dusting system will require one 18.3 meter (60 foot) diameter thickener and a single one-meter wide belt press to dewater the 3.6 metric tons (four short tons) of dry coal per hour from thermal dryer scrubber effluent. The installed cost of a

typical 3.6 metric ton (four short ton/hour) (solids recovery) commercial thickener/belt press system is estimated to be approximately $550,000. Resultant annual bone-dry fine coal recovery of this system is 16,300 metric ton/yr (18,000 ton/yr), assuming it is installed on a dryer operated on a three-shift/four-day workweek (4500 hours of annual operation) similar to Mine Number 4. Annual revenue derived from using this amount of recovered CWM for de-dusting the over-dry cyclone fines, at current rates of $40.79/metric ton ($37/ton), is $666,000/year. The thickener/belt press system capital investment represents an average of $34 for each metric ton-per-year of annual coal production. These represent conservative annual revenue and capital investment figures; a 78% improvement would occur if a similar unit was operated with four crews or 8000 hours/year.

Currently, JWRI pays $3.31 per metric ton to "muck out" their settling ponds. Consequently, there will be a $54,000/year reduction in this tailings disposal cost due to the 16,300 metric ton/year reduction in fines being sent to the ponds.

The major operating cost for a thickener/belt press involves the polymer flocculants. A maximum of 1.5 kilograms of polymer will be required to flocculate each metric ton of dry coal (3 pounds/short ton). Therefore, at an average cost of $6.61/kilogram, annual polymer costs will total $162,000/yr. As no additional manpower needs to be added to operate the automated thickener/belt press system, and utilities costs are minimal, the additional operating costs are assumed to be zero. Maintenance costs have been taken to equal 4% of the installed plant, or $22,000 per year.

Subtracting the polymer and maintenance costs from the gross annual revenue results in a net revenue of $536,000/year. Dividing the thickener/belt press' $550,000 installed cost by the $536,000 increase in annual revenue yields a simple payback period of 1.03 years. An even more attractive 0.57 year payback period is possible if the proposed system were installed at a four crew plant, rather than at a three crew plant. Clearly, such a capital investment would interest any coal producer.

SUMMARY

The detailed design of the system modifications is complete. Equipment quotations have been recieved. The equipment will be installed during the next six months followed by six months of operation to complete the DOE program.

Figure 1 Conventional Processing Plant Coal Thickener Arrangement

Figure 2 Proposed Fine Circuit Modification

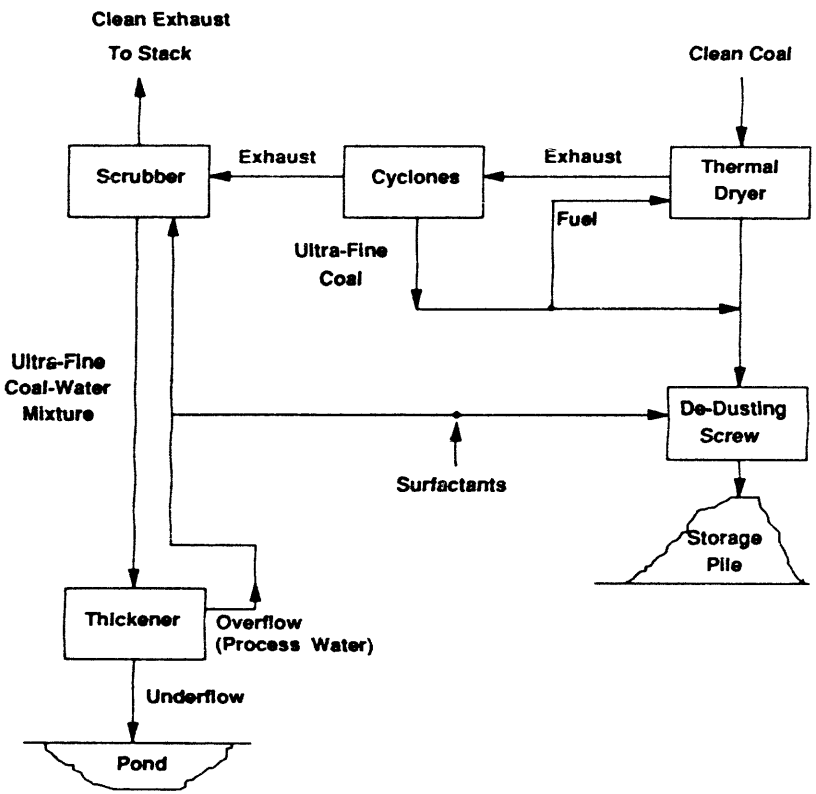

Figure 3 Relation of Thickener to Scrubber

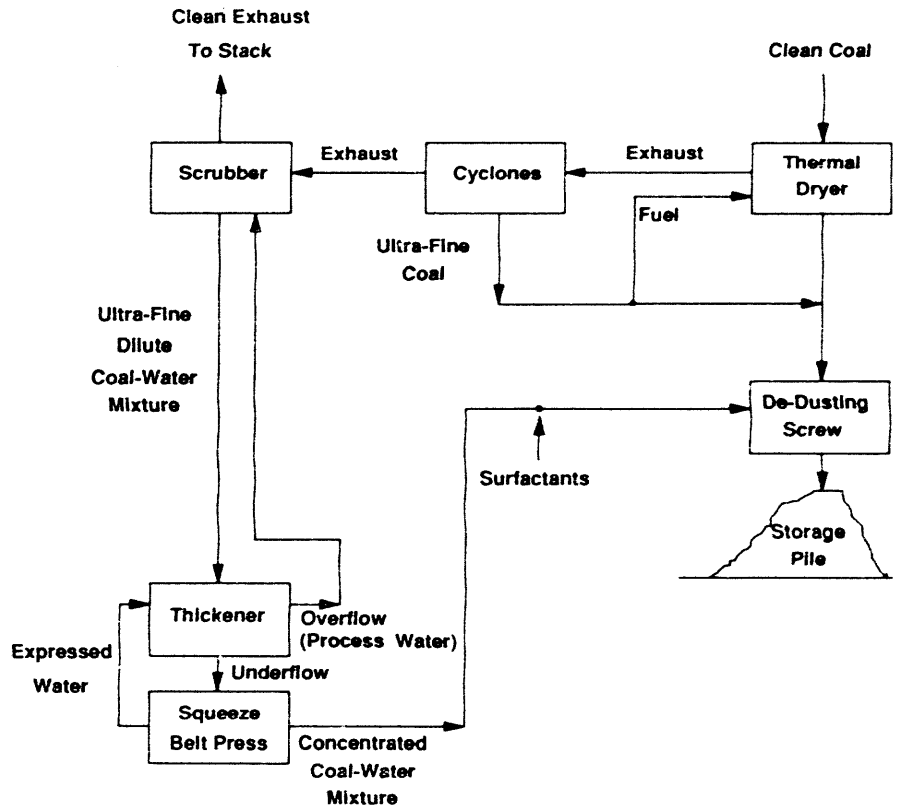

Figure 4 Proposed Belt Press Integrated to Thickener Underflow Stream

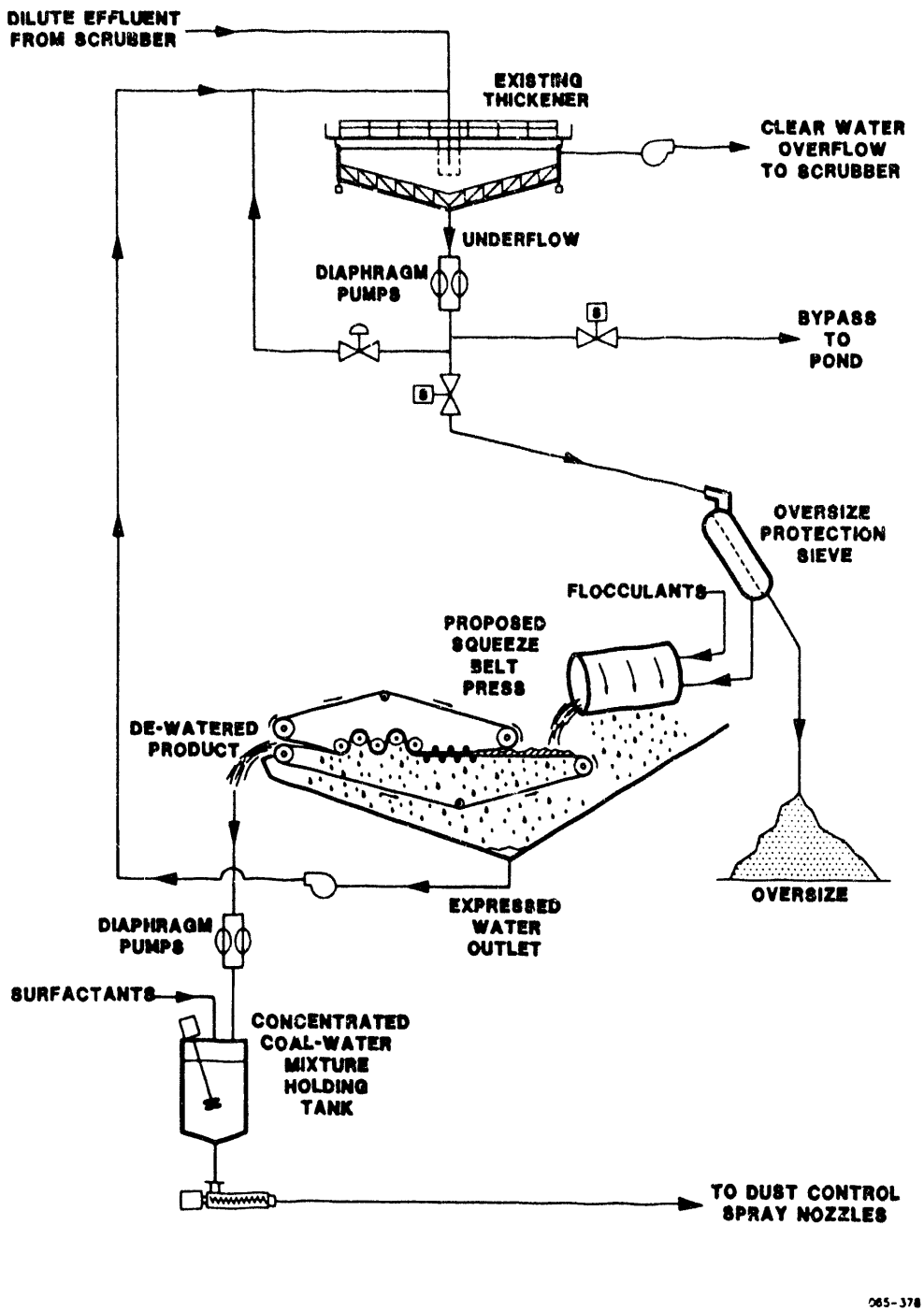

Figure 5 Proposed Modification to Thickener Underflow Stream

EVALUATION OF HYPERBARIC FILTRATION FOR FINE COAL DEWATERING

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Abstract

In this university/industry joint project, application of hyperbaric filtration is being evaluated at the laboratory scale for dewatering fine coal cleaned by froth flotation. The dewatering tests on three different clean coals were conducted to identify optimum dewatering conditions with respect to cake thickness, filtration time, percent solids, pH, viscosity and chemical reagents additions.

Introduction

This project is jointly conducted by the University of Kentucky, Pennsylvania State University and Consol Inc. The Pennsylvania State University is developing a mathematical model for the hyperbaric filter; the University of Kentucky is conducting laboratory hyperbaric filtration tests; and Consol Inc. will conduct pilot plant testing using the Andritz hyperbaric filtration system.

Current fine coal dewatering practice typically utilizes vacuum disc filters which can reduce the moisture content of -28 mesh coal to approximately 25 to 30 percent. A more desirable moisture level range is 15 to 20 percent moisture. At the present time, these low moisture levels are obtained by thermal drying. Hyperbaric filtration has shown potential for lowering product moisture for fine, clean coal to this low moisture level.

The present study is investigating effect of various hyperbaric filter operating parameters such as cake formation/dewatering time, air consumption, etc., on dewatering of ultra-fine clean coal using a laboratory high pressure filter equipment. The overall objective of this study is to determine optimum operating parameters of the hyperbaric filter and to apply the results in evaluating a continuous pilot scale hyperbaric filter for fine coal dewatering at a coal preparation plant site.

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Experimental

Clean coal froth samples were supplied by Consol Inc. from their preparation plants processing Illinois No. 6, Pittsburgh No. 8 and Pocahontas No. 3 coals. Table 1 lists the characterization data for the three slurries. Note, that Illinois No. 6 and Pocahontas No. 3 coal has significant amount of +100 mesh material; whereas Pittsburgh No. 8 sample had about 40 percent passing 500 mesh material.

Laboratory filtration tests were conducted using a 70 mm diameter Mott porous disc pressure filter. Dewatering tests were conducted using Whatman No. 1 filter medium which was supported by a stainless steel disc with 3/16 inch diameter hole pattern drilled to the specifications of the medium support used in commercially available filters. The filter system was modified by installing a stirrer inside the filter to stir the slurry during filtration cycle to avoid segregation of large particles at the bottom of the cake. This setup provided a uniform distribution of particles in the filter cake.

The filtrate was collected in a receiver mounted on a load cell. The signal from the load cell was converted with an A/D converter and recorded with a computer. The load cell signal was recorded at one second intervals so that filtration rate and cake formation time could be accurately determined.

Results and Discussion

Effect of Particle Size: For the coarser size coal feed i.e., Illinois No. 6 and Pocahontas No. 3, Vacuum filtration provided a 25 percent moisture filter cakes, whereas pressure filtration, using 60 psi pressure, Illinois No. 6 coal provided 20 percent moisture and Pocahontas provided 11 percent moisture filter cakes. Pittsburgh Nol 8 coal feed, consisting of finer size particles, provided 70 percent moisture filter cake using vacuum filtration and 22 percent moisture cake using 70 psi pressure. These results indicates that vacuum filtration will be effective for a coarser size particle, whereas pressure filtration will be effective for all size particles in providing low (~20 percent) moisture filter cake.

Effect of Cake Thickness: Increasing cake thickness resulted in higher moisture content as shown in Figure 1. For the Illinois #6 slurry at 40 psi, cake moisture increased from 20.5 to 44 percent moisture as the cake thickness was increased from 0.6 to 2.8 cm. At higher pressure (60 and 80 psi), cake moisture increased from 17 to 24.5 percent over the same cake thickness range. For the Pittsburgh #8 slurry, similar trends were apparent, although the moisture levels were higher. These results show that 60 to 70 psi is sufficient pressure to achieve maximum moisture reduction. No further improvement in cake moisture was observed at higher pressures.

Effect of Applied Pressure: The results presented in Figure 2 show that at a given cake thickness, increasing pressure reduced cake moisture. For the Illinois #6, for a cake thickness of 0.8 cm, increasing the pressure from 40 to 60 psi reduced the moisture from 20.5 to 17 percent. Higher pressure (80 psi) provided no further moisture reduction. At 2.8 cm cake thickness, increasing the pressure from 40 to 60 reduced the moisture

from 44 to 25 percent. Again, no significant improvement was noted at even higher pressure of 80 psi. For the Pittsburgh #8 slurry (Figure 2b), increasing the pressure from 40 to 70 psi reduced moisture from 27 to 21 percent for a 1 cm thick cake. At higher cake thickness (2.0 cm), moisture was reduced from 65 to 44 percent by increasing the pressure from 40 to 70 psi. For this cake thickness range, no further moisture reduction was observed by increasing the pressure to 90 psi.

Effect of pH: A correlation was observed between the pH and electrophoretic mobility of fine solids on cake moisture. The zero-point-of-charge (ZPC) of the coals was near pH 3.0 and at this pH lowest cake moisture was obtained compared to other pH. For Pittsburgh No. 8, 20.5 percent moisture was obtained at pH 3, compared to 24.5 percent obtained at pH ~9.0.

Effect of Slurry Temperature: Effect of changing slurry temperature on final cake moisture of the three coals is shown in Figure 3. Note, that lowering of moisture was substantial in case of Pittsburgh No. 8 coal. Other two coals also indicate lowering of cake moisture with temperature.

Effect of Slurry Concentration: The effect of slurry concentration on filter cake moisture is shown in Figure 4. For Illinois No. 6 coal, varying solids concentration from 5 to 40 weight percent, lowered cake moisture from 32 percent to 20 percent. For the Pittsburgh No. 8 coal slurry, the moisture content of filter cake lowered from 63 percent to 18 percent as the solids were varied from 7 to 40 weight percent.

Effect of Flocculant Addition: For the Illinois No. 6 coal addition of 30 ppm of an anionic flocculant lowered filter cake moisture from 25 percent to 17 percent. Similarly, for the Pittsburgh No. 8 coal, addition of 5 ppm of the anionic flocculant lowered the filter cake moisture from 22 percent to 16.5 percent. The Pocahontas No. 3 coal, being very hydrophobic, did not show very significant reduction of filter cake moisture, viz, a high dosage of 40 ppm of a nonionic flocculant was required to lower the filter cake moisture from 11 to 9.5 percent.

Future Plans

Additional laboratory hyperbaric filtration studies will be conducted with the Pocahontas No. 3 coal. Pilot scale hyperbaric filtration tests will be conducted at one of the Consol Inc. preparation plants using the optimum filtration\dewatering conditions found in the laboratory studies.

Conclusions

Based on the results obtained so far, it can be concluded that

- A novel process was developed for forming uniform filter cake from coarse coal slurry.
- For the Illinois No. 6 coal, at 40 psig, the moisture increased from 22 to 45 percent as the cake thickness increased from 0.8 to 2.8 cm. For Pittsburgh No. 8 slurry, the

filter cake moisture increased from 27 to 65 percent as the cake thickness increased from 1 to 2 cm.

- Increasing slurry temperature lowered moisture content of filter cake. Lowest filter cake moisture was obtained at 90 C.
- Increasing slurry concentration for Illinois No. 6 slurry to 40 weight percent lowered moisture content of filter cake to 20 percent. Similarly, for the Pittsburgh coal 18 percent moisture filter was obtained using 40 percent solid slurry.
- The optimum dewatering conditions for the Illinois No. 6 were 2 cm cake thickness, 60 psi pressure and 2 minute filtration time. For the Pittsburgh No. 8 slurry, 70 psi pressure, 1.5 cm thick cake and 3 min. filtration/dewatering time were optimum.
- Tests are in progress using the Pocahontas No. 3 coal.

Acknowledgements

Financial support provided by the United States Department of Energy Contract No. DE-PS22-92PC92520. Coal slurry samples were provided by Consol Inc.

TABLE 1. CHARACTERIZATION DATA FOR ILLINOIS NO. 6, PITTSBURGH NO. 8 AND POCAHONTAS NO. 3 COALS.

Characterization Data for Illinois No. 6 Seam Coal Froth

Size (Mesh)	Weight Percent	Ash Percent	Percent Ash Distribution
+100	45.84	4.28	34.2
100x200	14.72	4.77	12.2
200x325	7.72	5.43	7.3
325x500	11.85	3.71	7.7
-500	19.87	11.14	38.6
Feed (Calc)	100.00	5.73	100.0
(Actual)		6.43	

Percent Solids in Froth = 26

Characterization Data for Pittsburgh No. 8 Seam Coal Froth

Size (Mesh)	Weight Percent	Ash Percent	Percent Ash Distribution
+100	2.77	2.43	0.8
100x200	19.14	2.52	5.6
200x325	13.59	3.34	5.2
325x500	22.23	3.98	10.2
-500	42.27	15.92	78.2
Feed (Calc)	100.00	8.60	100.0
(Actual)		8.78	

Percent Solids in Froth = 11

Characterization Data for Pocahontas Coal Froth

Size (Mesh)	Weight Percent	Ash Percent	Percent Ash Distribution
+28	4.5	3.02	2.4
28x48	24.8	3.61	16.1
48x100	20.5	4.20	15.5
100x200	17.3	4.44	13.8
200x325	6.9	3.95	4.9
-325	26.0	10.00	47.3
Feed (Calc.)	100.0	5.53	
(Actual)		5.55	

% Solids in Froth = 25

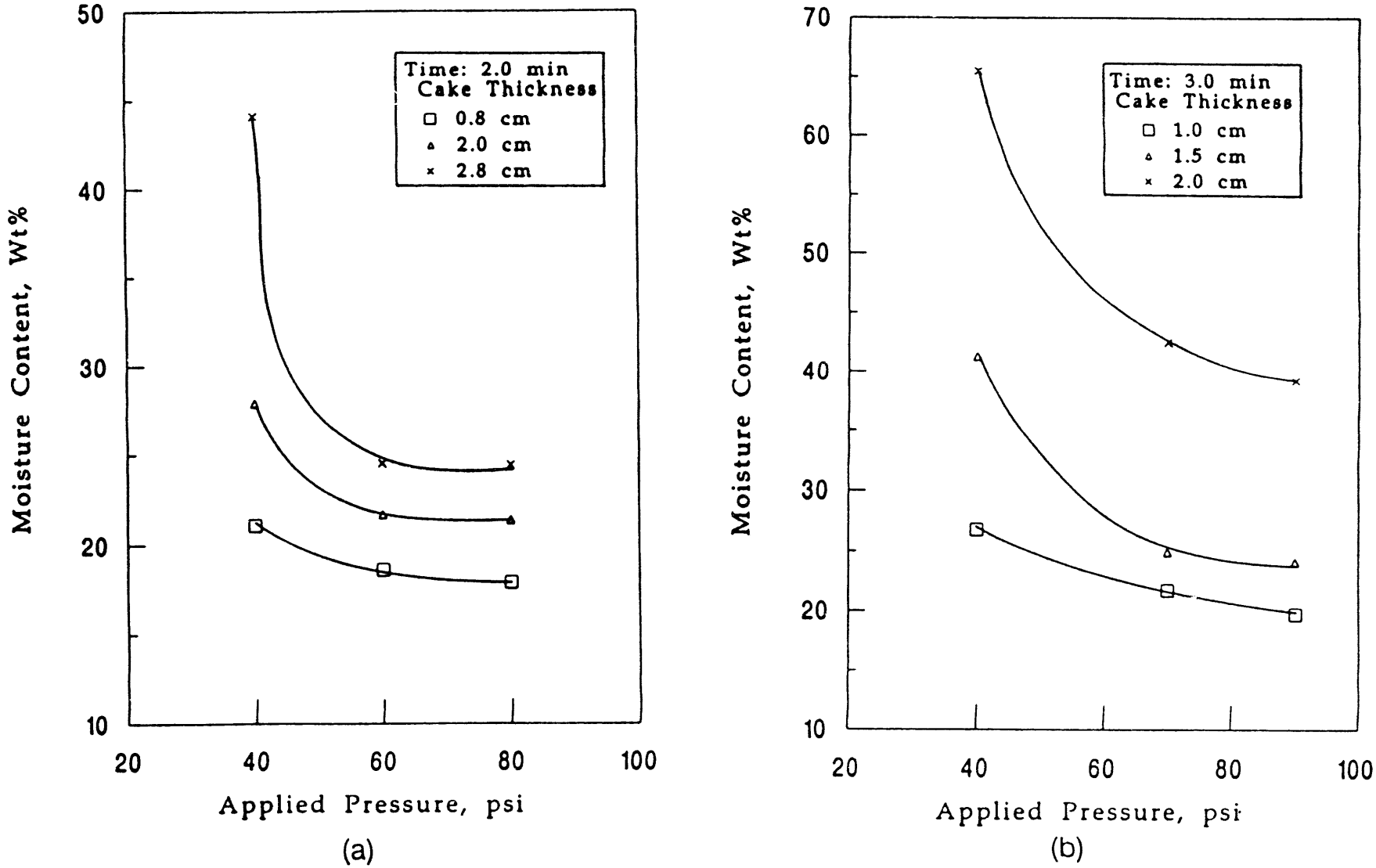

Fig. 1. Determination of optimum process condition for applied pressure.
(a) Illinois No. 6 coal; (b) Pittsburgh No. 8 coal

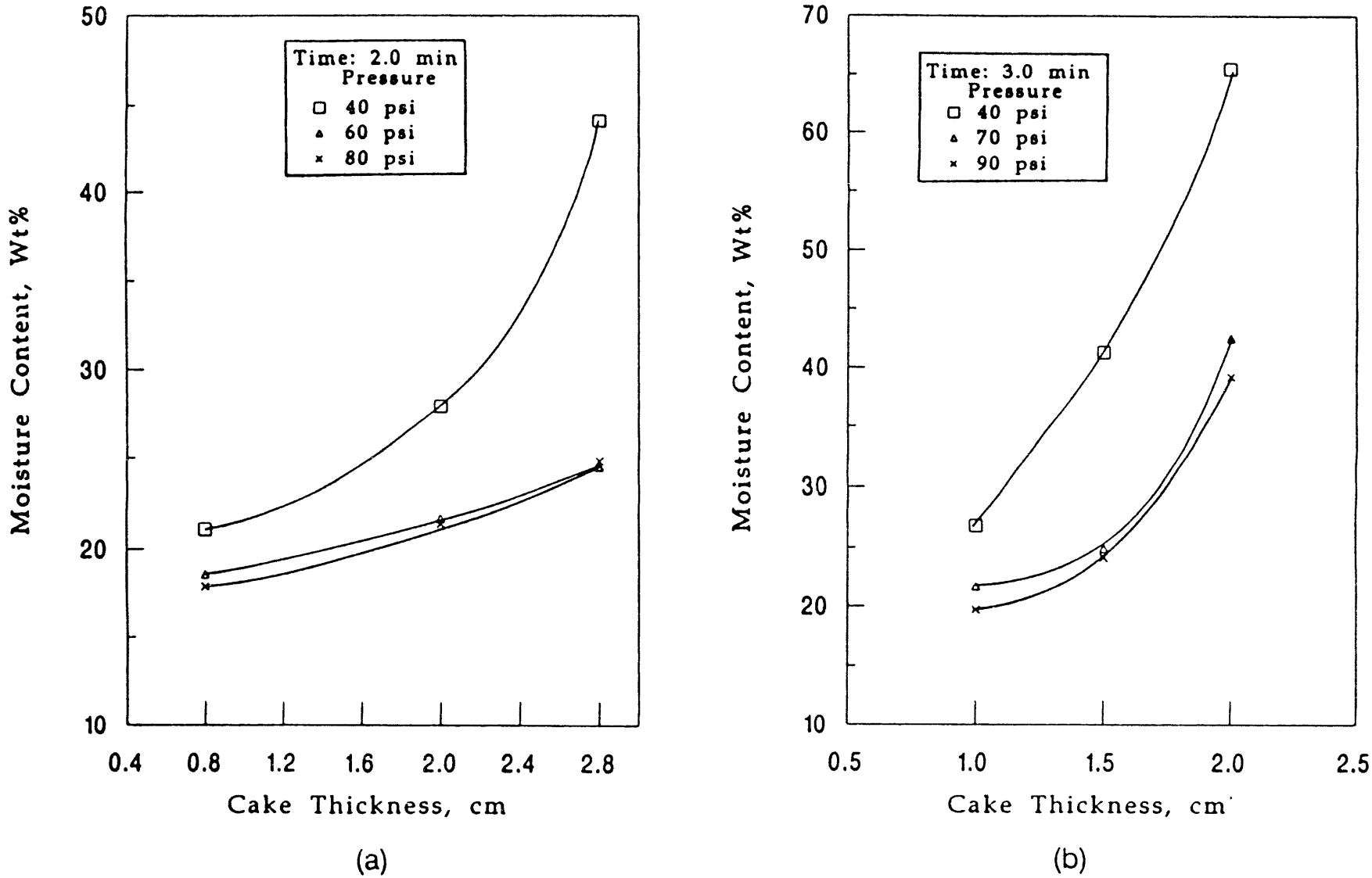

Fig. 2. Determination of optimum process condition for cake thickness.
(a) Illinois No. 6 coal; (b) Pittsburgh No. 8 coal

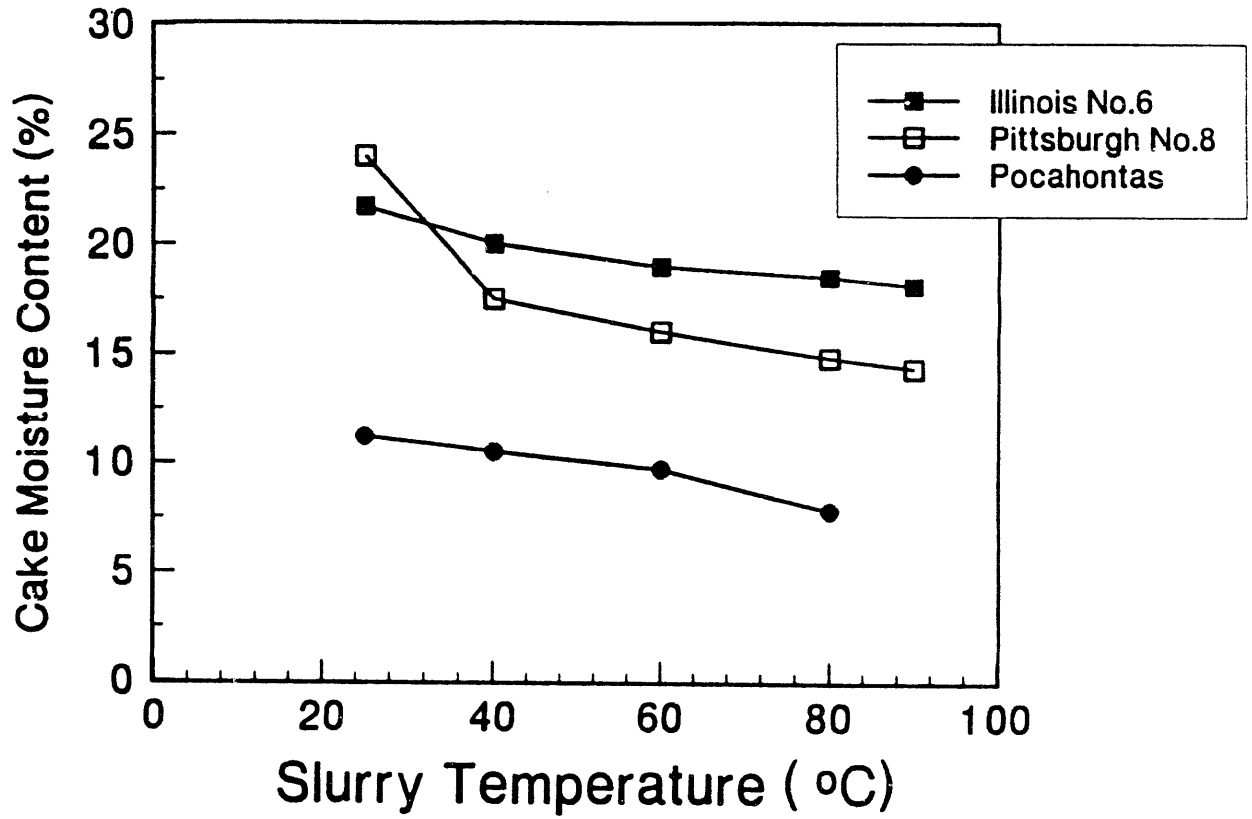

Fig. 3. Effect of temperature on cake moisture (pressure: 70 psi; cake thickness 1.5 cm)

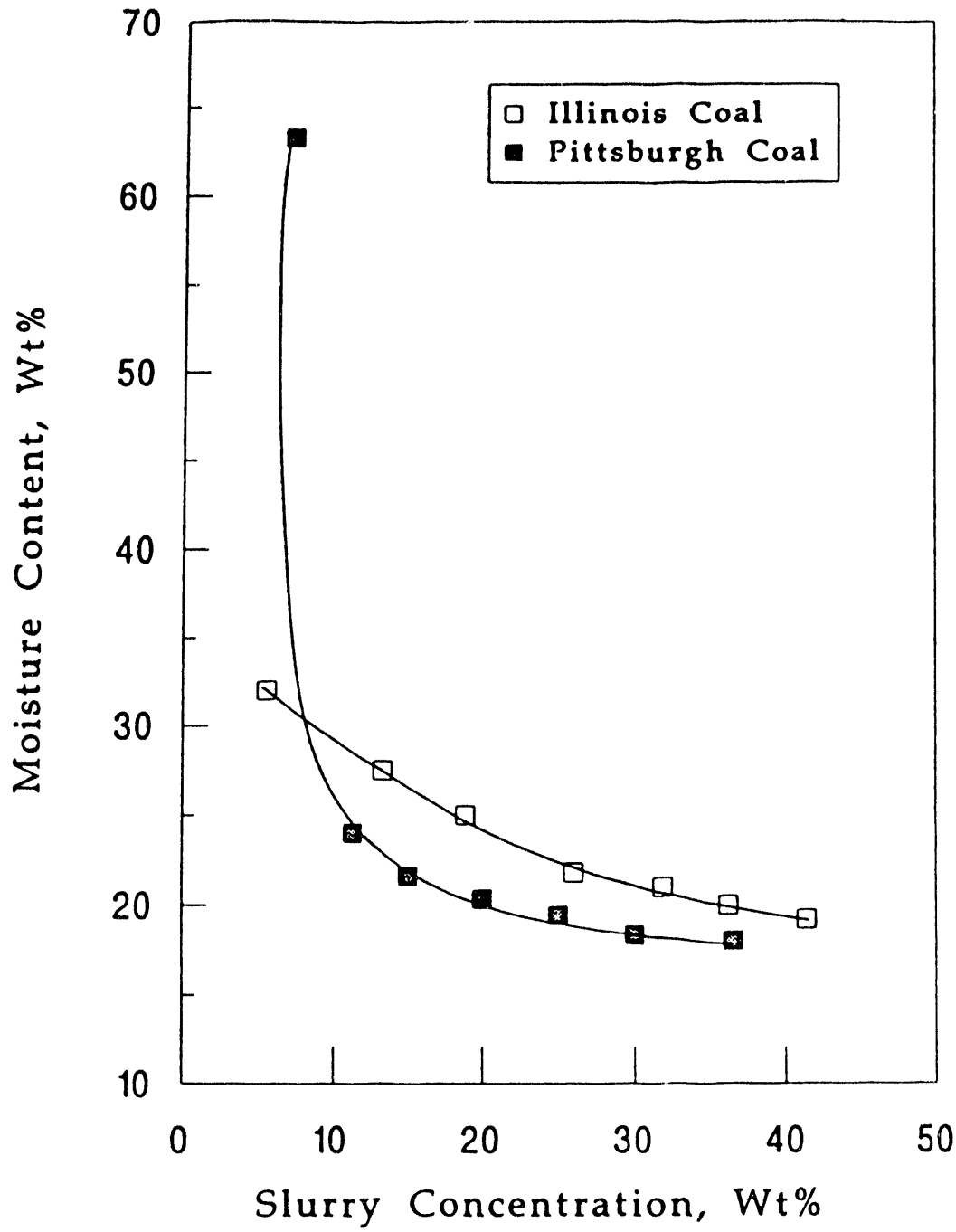

Fig. 4. Effect of slurry concentration on cake moisture.

Reconstitution of Beneficiated Coal Using the BioBinder Process

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1. Introduction

Coal fines can be beneficiated by froth flotation to produce a clean fuel. However, the resulting product consists of a wet cake material that is difficult to handle, store and transport [1]. If the material dries, it will become dusty and can be an explosion hazard. If it remains wet, it can be subject to freezing. As an alternative to this form, the cake could be processed into pellets, that have much improved handling, storage and transport characteristics [2].

Patents relating to the pelletizing of coal fines can be found as far back as the 1920's [3]. In these early efforts, the purpose of pelletizing the coal was to reclaim waste coal fines produced as part of the coal washing process. More recently, it has been of interest to develop an inexpensive coal based fuel that has lower sulfur emissions and ash levels. This beneficiated fuel could be used in a range of combustion equipment to meet evolving regulatory constraints. Further, if the coal was sufficiently cleaned, it could even be used as a substitute for oil or gas in existing combustors.

In the past, corn starch has been proposed as a binder to reconstitute coal fines. This works relatively well and produces a pellet that has adequate durability. Recent tests showed the ability of two percent corn starch binder to pelletize beneficiated coal[2]. Similar results were achieved for Shur Bond [2]. Pellets using these binders even survived two weeks exposure of hard winter conditions (rain, snow, freezing temperatures). These results suggest that durable pellets can be made from beneficiated coal fines. However, binders are expensive, costing $200 to $700 per ton. This increases the cost of the fuel by greater than 15 percent, and makes the pellets uncompetitive with the parent coal.

New, low-cost, or even negative cost, binders are needed. Negative cost binders would be wastes that have a significant disposal cost. An example of a waste binder would be lignocellulosic waste and sawdust [4]. These materials have been used to successfully pelletize cleaned coal fines. The pellets met strength and durability goals. However, lignocellulosic and sawdust binders have limitations related to cost and availability.

A more available and less expensive waste binder is municipal sewage sludge. Over 30 million tons of sewage sludge are produced every year [5]. Sludge is composed of sticky hydrocarbons and some fibrous material. In the past, sewage sludge has been dumped in the ocean, applied to the land or incinerated, with the resulting ash landfilled. Regulations have now banned ocean dumping and require significant processing prior to land application or landfilling [6]. This processing is required to reduce the biological activity of the sludge. Through processing and ultimate landfilling, the cost to dispose of sludge is over of $40/ton [7]. Thus there is a significant incentive to reuse this material.

2. BioBinder Process

In the BioBinder process, sludge is used to bind the coal particles together. Steps are included in the BioBinder process to biologically deactivate the sludge, mix the sludge and coal together, and finally form dry pellets of a specific diameter and length. In addition, a proprietary and low cost agent is also introduced into the mixture to enhance the forming and weatherability of the pellets. Based on Altex and equipment supplier tests, the BioBinder process steps can be implemented on commercially available equipment. Therefore, special process equipment development efforts are not required.

Preliminary bench-scale tests by Altex showed that the BioBinder process has potential to reconstitute finely ground coal to a durable pellet. These efforts helped promote the award of a Phase I SBIR feasibility program under DOE sponsorship. The objectives and results of the Phase I program are briefly highlighted below.

3. Phase I BioBinder Program Technical Objective

The objective of the Phase I effort was to show the feasibility of the BioBinder process to create beneficiated coal pellets that have low-cost and good handling, transport, and storage, characteristics.

4. Program Results

To show the feasibility of the BioBinder process, tests were performed to form the pellets and then subject them to a suite of tests that show their handling, transport, storage and combustion characteristics. Based on the pellet forming test results, a full-scale 1000 tons-per-day BioBinder system was designed that incorporates commercial equipment. System capital and operating costs were then estimated to determine the cost per ton of pellets produced. Test and economic results showed the performance and economic feasibility of the BioBinder process.

4.1 Pellet Formation and Performance Evaluation

In the bench-scale tests that preceded the Phase I program, 10 lb/hr capacity mixer and pellet forming equipment were utilized. For the Phase I tests, a 500 lbs/hr pelletizer was utilized in tests at Altex. In addition, pelletizing equipment suppliers tested the BioBinder material for forming and handling characteristics, at similar scales. According to the equipment suppliers, the test equipment used has processing characteristics that are scaleable to the 1000 tons/day capacity of the projected full-scale system. Therefore, Phase I pellet formation test results should be a good indicator of how full-scale equipment should perform.

Pellet characteristics will depend on mating the proper formation equipment to the feed material. The equipment imposes pressure that shears and compacts the material as it is formed . Higher compaction levels reduce voidage and usually produce a harder pellet. In addition to the pellet forming equipment, the composition of the mixture and the level of homogeneity plays an important role in determining pellet characteristics. For the Phase I tests, 5 types of sludges , 5 coals and one additive were used to prepare pellets. These raw materials are listed in Table 1. The sludges tested covered major classes of municipal sludges available, as well as one industrial sludge. Undigested or raw sludge consists of primary and secondary sludges. These bio-solids receive no treatment for deactivation prior to dewatering, and are typically incinerated at the water treatment plant Digested

sludge is processed anaerobically and aerobically in large tanks for several weeks, and is then land applied. Sludge qualities and heat content vary substantially between digested and undigested sludges. In addition to common sludges, the novel Zimpro process sludge was also tested. In the Zimpro process, the sludge is brought to high temperature and pressure and then allowed to expand to atmospheric pressure. This process causes the water filled fibers and micro-organisms to burst, which makes it easier to dewater the sludge. Also, by processing the sludge at high temperature, the sludge is biologically deactivated. Both of these characteristics were of interest to examine under the BioBinder program.

An industrial sludge obtained from a pulp and paper mill was also investigated for its possible binding characteristics. There are approximately 45,000 tons of paper sludge produced at pulp mills every year. Only a small fraction is recycled back into the process. This sludge is typically comprised of primary and secondary solids and has a high percentage of fiber. It is usually not treated, other than for odor, since it contains no coliforms or pathogens.

Finely ground coals tested under the BioBinder program are listed in Table 1. These coals span a range of typical coals. Two western pulverized coals were initially tested for there ease in handling. The Black Thunder and Utah P&L samples were typically 62% minus 200 mesh. The cleaned coal samples were provided by Consol and Kerr-McGee. These froth flotation fines were received in a slurry form then decanted before use. Consol's Pennsylvania seam coal was 90% minus 100 mesh, and 60% minus 325 mesh. The Kerr-McGee Illinois No.5 sample was classified as 100% minus 100 mesh. In addition, a sample of dried eastern coal baghouse fines was supplied by Consol. This material is typically 85% minus 325 mesh and low moisture.

Table 1 -- Pellet Formation

- **Sludge Samples**
  - **San Leandro, digested**
  - **Union City, digested**
  - **San Mateo, Zimpro Process**
  - **Palo Alto, undigested**
  - **Weyerhaeuser, primary and secondary paper sludge**
- **Coal Samples**
  - **Dry, pulverized or bag house fines**
    - **Utah P & L- Blind Canyon, Utah**
    - **Black Thunder - Powder River, Montana**
    - **Consol, dried Eastern fines**
  - **Froth Flotation Fines**
    - **Consol, Pennsylvania**
    - **Kerr-McGee, Illinois #5**

Moisture is a significant parameter in pellet formation. Moisture levels were varied to define ranges where good pellets were formed. In all, over 30 mixtures were formed into pellets using the 500 lbs/hr processing equipment. As needed, the formed pellets were dried and subjected to several tests to determine their handling, transport, storage and grinding characteristics. Preliminary combustion tests showed that dried sludge pellets have excellent combustion characteristics. This is due to the higher volatiles content of the sludge compared to the coals tested in this study. A proximate and ultimate analysis of a typical BioBinder mix is given in Table 2. As of the date of this report, coal and sludge mixture combustion tests have not been carried out. Given the good results for sludge, it is expected that the coal/sludge mixture combustion tests will also show good combustion characteristics.

Table 2 -- BioBinder Mix Analysis

Analysis Report No. 71-70976

Proximate Analysis			Ultimate Analysis		
	As Received	Dry Basis		As Received	Dry Basis
% Moisture	32.65	xxxxx	% Moisture	32.65	xxxxx
% Ash	5.98	8.88	% Carbon	50.35	74.76
% Volatile	25.94	38.51	% Hydrogen	3.4	5.05
% Fixed Carbon	35.43	52.61	% Nitrogen	1.05	1.56
	100	100	% Sulfur	0.87	1.29
			% Ash	5.98	8.88
Btu/lb	8974	13325	% Oxygen (diff)	5.70	8.46
% Sulfur	0.87	1.29		100	100
MAF Btu		14624			

There is currently no universally recognized standard tests for pellet quality. Most studies utilize similar tests, but the conditions and procedures often vary widely. In Battelle's study of coal reconstitution(8), several tests were outlined that measured pellet quality. Altex incorporated into the BioBinder program five tests related to strength, abrasion resistance, and long term storage from the Battelle study. The pellets were subjected to the tests listed in Table 3. Note that durability tests were performed on the pellets after each process.

Table 3. BioBinder Process Pellet Tests

Test Category	Test Purpose	Approach	Parameters
Pellet Properties	Heat Content & Ignitability	Proximate analysis	Heat Content Volatile Content
	Emissions Potential	Proximate and ultimate analysis	Sulfur, Nitrogen and Ash Contents
Durability	Fracture Load	Impose increasing load with flat bar across pellet	Load which achieves severe deformation and surface cracking Pellets tested: wet, dried, frozen, thawed
	Axial Compression	Axial load applied until failure on 1" specimen	Load volume which achieves pellet failure Pellets tested: wet, dried, frozen, thawed
	Storage Pile Crushing Resistance	Load applied to column of pellets	% volume reduction, % fines created, agglomerated? Pellets tested: wet, dried, thawed
	Transport Abrasion Resistance	Fixed weight of pellets is tumbled in 8-mesh screen drum.	% fines created, drum rpm, run time Pellets tested: dry
	Drop Resistance	Pellets dropped variable heights onto steel plate	Number of drops until remaining pellet L/D is equal to 1 Pellets tested: dry, thawed
Weatherability	Rain Absorption Resistance	Pellets submerged under water, then re-dried	Time submerged, % H_2O increase, % weight increase, durability tests
	Freeze/Thaw Resistance	Freeze and thaw pellets in repeated cycles	Freeze thaw cycles, % residual H_2O, durability tests
Grindability	Pulverization Potential	Hardgrove Test	HGI
Combustion Characteristics	Fixed Bed Burning	Burn pellets in a small fixed bed under controlled air flow and heat loss	Burn rate, emissions of NO, NO_2, CO, CO_2, O_2 and unburned hydrocarbons, bed temperatures
Drying Characteristics	Estimate Commercial Dryer Performance	Dry pellets in a small fixed bed under controlled conditions	% weight loss, time elapsed, pellet configuration, gas temperature, gas velocity

Test Results

In Table 4. the test results of four BioBinder samples , three produced at Altex and one at a commercial pellet former manufacturer, are shown. All of these BioBinder mixes used the Consol froth flotation fines. The Altex samples vary only in content of the proprietary WeatherGuard agent used to enhance forming and improve weatherability. Note the high axial compression strength of 300 to 425 lbf/in^3. This compares to maximum loads of only 200 lbf/in^3 in the Battelle study for a cured extruded pellet using Shurbond and cornstarch as binders.(8) As of the date of this publication, Altex has produced pellets, of similar formula to those in Table 4, that withstand axial compression loadings of 550 lbf/ in^3.

To simulate conditions for storage in piles, as well as transport and handling, the crush test and tumbling abrasion tests are used. Although the losses due to abrasion are only 1 to 2 percent, these can be reduced further with more refined post forming techniques. The dried pellets do well in crush tests, which simulate piles that are 110 feet high. Although there is a substantial loss in strength when the pellets have been submerged under water for three days, there is no disintegration of the pellets. When redried, over 90% of the original strength is recovered. Under the less severe weather conditions of the 11 week long-term exposure tests, pellets with the Altex proprietary WeatherGuard agent typically recovered over 92% of the original strength within two days after a heavy rainfall. Freezing and thawing repeatedly had little affect on strength. In some cases "aging" improved performance. Coal type seemed to have little impact on either formation or durability. All five of the coals tested were of minus 100 mesh or smaller, Coarser coals may not fair as well, as the binder may not be as finely dispersed. This would reduce the binder's effectiveness and the material's forming characteristics.

In summary, the pellet forming and characterization tests showed that robust BioBinder pellets could be formed. Based on the test results, the pellets should be able to withstand needed handling, transport and storage. These results clearly show the feasibility of the BioBinder process to reconstitute coal fines. Economic analyses results, briefly highlighted below, show the cost benefits of the BioBinder process.

4.2 Economic Evaluation

A key factor in promoting the use of the BioBinder process is the cost per ton of the pellets relative to the baseline coal. An economic analyses was performed for the BioBinder process and the results compared to baseline coal costs. Both capital and operating costs were considered.

To establish the costs of the major equipment elements, vendor quotes were solicited from the equipment manufacturers. Table 5 gives a listing of purchased equipment. Installation costs were based on a percentage of equipment costs. The ratio was estimated from equipment supplier experience. A contingency factor of 10 percent was also used in this estimate. A 12 year depreciation schedule was used in the costs.

To define operating, labor and maintenance costs, equipment supplier inputs were utilized. Expendable material costs were based on coal fines costs of $30/ton and sludge disposal costs of $30 ton. Coal fines costs are variable, depending on the specific case. At a minimum, the coal fines cost will be equal to the coal cleaning cost of approximately $6/ton. At a maximum, it could

Table 4 Altex-Lab and Commercial Pellet Tests

Commerical
****Test*****

Sample No.	Altex 01	Altex 02	Altex 03	CS01B
Binder Type	Sludge	Sludge	Sludge	Sludge
MIX DATA				
Coal Type	Consol-Pitt	Consol-Pitt	Consol-Pitt	Consol-Pitt
Other Additives	none	1% WeatherGuard	2% WeatherGuard	1% WeatherGuard
** Cured Pellet Data**				
% Shrinkage	0.80	-----	0.80	---
Bulk Density (lbm/cuft)	27.40	28.47	27.30	28.80
Drop Test 5'	10+	10+	10+	10+
Drop Test 15	8/pass	3/Pass	4/pass	5/Pass
Fracture Test (lbs)	30.00	31.00	26.00	----
Comprssion Test (lb/cuin)	425.00	335.00	305.00	407.20
Crush Test (psi)	24.50	24.50	24.50	24.50
% Vol. Diff	4.34	5.74	7.60	5.20
% Fines	0.52	1.60	0.34	3.50
Abrsn Test (% Loss)	0.76	0.95	1.67	1.68
** Weatherability**				
Absorbsion Test(%wt. incr)	39.00	30.60	25.25	28.40
Time Submergd (hrs)	80.00	74.00	76.00	75.00
** Wet Tests**				
Facture Test (lbs.)	7.00	6.00	7.00	11.00
Crush Test (psi)	24.50	24.50	24.50	24.50
% Vol. Diff.	36.80	40.50	32.50	45.00
% Fines	-----	59.20	24.70	50.00
Sticky?	-----	yes	yes	yes
Re-Dry Tests				
Drop Test 60"	9.00	10+	10+	10+
Drop Test 15'	8/pass	4/pass	3/pass	2/pass
Fract. Test (lbs)	38.00	32.00	21.00	55.00
Crush Test (psi)	24.50	24.50	24.50	24.50
% Vol. Diff.	8.70	17.50	12.50	13.00
% Fines	2.40	5.50	5.00	16.80
Freeze/Thaw				
Thawed Moist. (%)	0.00	0.00	0.00	1.00
#Freez/Thaw Cycle	3.00	3.00	3.00	3.00
Froz Fract Test (lbs)	35.00	38.00	29.00	65.00
Froz Comp (lb/cuin)	----	----	----	407.20
Thaw Fract (lbs)	35.00	34.00	28.00	47.00
Drop Test 60"	10+	10+	10+	10+
Drop Test 15'	9/pass	8/pass	7/pass	5/pass
Crush Test (psi)	24.50	24.50	24.50	24.50
% Vol. Diff.	5.10	5.50	12.50	20.00
% Fines	1.00	0.20	2.10	12.20
Sticky?	no	no	no	no
** Longterm Storage**				
No. Weeks Exposure	11	----	11	----
% Strength-Uncovered	89.00	----	93	----
% Strength-Covered	92.00	----	96	----

be equal to the base coal cost. Given the form and usability of the fines, the $30/ton is a high estimate. To be conservative, a coal fines cost of $30/ton was used in the baseline analyses.

The sludge negative cost for disposal is site specific. According to our survey, digested sludge, which is costly to produce, has a disposal cost of from $16/ton to over $50/ton. This type of material must be biologically deactivated before it can be applied to the land. The disposal price depends greatly on local restrictions, and proximity to either large agricultural or urban areas. Undigested sludge typically costs $50 to $60/ton to incinerate including disposal cost for the resulting ash. For the purpose of the analyses, a conservative sludge disposal cost was of interest. Therefore, a disposal cost of $30/ton was assumed for the analyses.

Sludge transportation impacts pellet costs. Investigation of coal preparation and sludge production sites in the eastern United States indicated that there are some large capacity sludge sources within 20 miles of coal preparation sites. Based on vendor input, truck transport of sludge to the coal preparation plant site was determined to be around $3.12/ton. Barge transport is an option for plants located on rivers. Costs for barge transport are less than for truck transport per mile, but loading and unloading costs may increase total costs. To be conservative, truck transport costs were used in the baseline analyses.

Using these inputs, an economic analyses was performed to determine the cost-per-ton of pellets for baseline conditions. In addition, cost elements were individually varied to determine the sensitivity of pellet costs to various elements that influence cost. The sensitivity analyses showed that the primary elements controlling pellet cost are coal and sludge disposal costs and coal to sludge ratio. Other elements that are secondary but still significant are sludge moisture content and sludge transportation costs. Sludge disposal costs are significant in that they can totally offset other pellet production costs. This is also illustrated by the baseline results.

Table 5 presents the baseline case capital cost estimates. As shown, the Purchased Equipment (PE) for a 1000TPD plant is nearly $4.6 million. To the PE is added 40, 24 and 10 percent to cover direct, indirect and contingency costs. These amounts, plus the working capital (one-twelfth of annual labor and overhead), then defines the total investment capital of $8.4 million. Assuming an 8 percent capital charge then gives the annual cost of capital.

Table 5 Capital Costs Estimate 1000 ton/day Plant				
Purchase Equipment	**Quantity**	**Total Cost**		
			Total PE	**$4,545,000**
Bucket Elevator	**2**	**$ 17,000**		
Feed Bin	**3**	**$ 27,000**	**Direct Cost**	
Screw Conveyor	**21**	**$ 34,000**	**40% of PE**	**$1,818,000**
Sprayer/Heater	**1**	**$ 20,000**		
Mixer	**1**	**$ 150,000**	**Indirect Cost**	
Former	**1**	**$ 450,000**	**24% of PE**	**$ 1,090,800**
Dryer/cooler	**3**	**$2,200,000**		
Belt Conveyor	**4**	**$ 102,000**	**Contingency & Fees**	
Cyclone	**2**	**$ 30,000**	**10% of PE**	**$ 454,500**
Screener	**1**	**$ 65,000**	**Total Fixed Capital**	**$7,908,300**
Scrubber	**1**	**$ 250,000**		
Sludge Dryer	**1**	**$ 1,100,000**	**Working Capital**	
600 hp Boiler	**1**	**$ 100,000**	**(1/12 Annual Operating Costs)**	**$ 512,200**
Total		**$ 4,545,000**	**Total Investment Capital**	**$8,420,500**
			Annual Capital Charge	
			8% of Fixed Capital	**$632,664**

Operating cost estimates for the process are given in Table 6. As shown, operating costs include raw material, transport, utilities, labor, operating supplies, plant overhead, taxes and insurance and annual capital charges. Adding these costs together provides the yearly cost from which cost-per-ton of product and cost-per-million Btu in the product can be determined. These are listed in the last two columns. As shown, the cost-per-ton of product is $21.26. This compares with the assumed baseline coal cost of $30/ton. Therefore, even for the conservatively high baseline coal fines cost of $30/ton that goes into the pellets, the product cost is 28 percent less than the parent coal cost ($30/ton). Furthermore, if, as expected, coal fines costs are less than the parent coal cost of $30/ton, then additional reductions are possible. Cost estimates were prepared for coal fines costs for zero to the maximum $30/ton baseline case. As shown in Table 7, compared to the baseline parent coal, cost-per-million Btu of the BioBinder fuel can be from 28 to 100 plus percent lower in cost per million Btu, depending on coal fines cost. This shows the substantial cost savings that can be achieved using this reconstitution process.

Table 6 BIOBINDER COSTS WORKSHEET 4/26/94					
	$/TON	TON/YR	$/YR	@ 4% H2O $ TON PROD	@ 4% H2O $/MMBtu PROD
RAW MATERIAL:					
COAL FINES(DRY)	30	212500	$6,375,000	$24.48	$0.95
SLUDGE	-30	163,044	-$4,891,320	-$18.78	-$0.73
SLUDGE TRANSPORTATION	3.12	93750	$292,500	$1.12	$0.04
UTILITIES			$1,939,340	$7.45	$0.29
TOTAL LABOR			$828,000	$3.18	$0.12
MAINTENANCE			$115,080	$0.44	$0.02
PLANT OVERHEAD			$165,600	$0.64	$0.02
PROPERTY TAXES AND INSURANCE			$79,083	$0.30	$0.01
DEPRECIATION/CAPITAL CHARGE			$632,664	$2.43	$0.09
TOTAL ANNUAL OPERATING COSTS			$5,535,947.00	$21.26	$0.81

Table 7 COAL COST FACTOR				
	ANNUAL OPERATING COSTS			PERCENT COST SAVINGS vs
COAL FINES		@4%H2O	@4%H2O	PARENT COAL COST (@ $1.15/MMBtu)
$/TON	$/YR	$/TON PROD	$/MMBtu PROD	%
$0	($839,053)	($3.22)	($0.13)	111
$5	$223,447	$0.86	$0.03	97
$10	$1,285,947	$4.94	$0.19	83
$15	$2,348,447	$9.02	$0.35	70
$20	$3,410,947	$13.10	$0.51	56
$25	$4,473,447	$17.18	$0 67	42
$30	$5,535,947	$21.26	$0.83	28

5.0 Conclusions and Recommendations

Using commercially available equipment, of 500 lbs/hr capacity, tests showed that the BioBinder process can produce robust coal/sludge pellets. These pellets are projected to be able to withstand the handling, transport and storage they would be subjected to as a stoker fuel source. The material produces little dusting upon handling. Even when exposed to weather conditions, including freeze/thaw cycles, the material has good strength. Dried sludge, by itself, has very good combustion characteristics. Coal/sludge pellets are also expected to have good combustion characteristics. This will be shown in upcoming small-scale combustion tests.

Besides good pellet characteristics, the BioBinder process produces low cost pellets. As in any coal fuel based pellet, coal fines cost drives the pellet cost. The other important driver is the sludge disposal cost. Even assuming a conservative disposal cost, the sludge disposal cost can totally offset the pelletization cost, yielding a low cost pellet. Assuming conservatively that the coal fines costs are the same as the $30/ton parent coal cost, the BioBinder pellet cost is 28 percent lower in cost then the parent coal. Furthermore, if the coal fines cost is $20/ton, then the BioBinder cost reduction is 56 percent. On a Btu basis, BioBinder pellets can be 28 to 100 plus percent lower in cost than the parent coal. This savings are a function of the coal fines cost. These results show the substantial economic benefits of the BioBinder process.

Given the clear feasibility of forming BioBinder pellets and the substantial economic benefits of the process, it is recommended that larger scale production and combustion tests be performed in a Phase II effort. Also a more extensive economic analysis for a site specific case should be carried out under a Phase II effort. The results from these efforts will then form the foundation for a subsequent commercialization effort, involving a coal preparation facility operator and a municipal sludge producer.

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IMPROVEMENT OF STORAGE, HANDLING, AND TRANSPORTABILITY OF FINE COAL

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PROJECT SUMMARY

Background and Objectives

Fine coal production is on the rise in the U.S., and it will continue to increase as underground mining companies invest in more productive equipment. Fine coal cleaning technologies have been developed which can efficiently and economically separate coal from clay and other mineral matter in the fine size fractions, but they have not gained universal acceptance in the coal industry because they produce a product which is too wet for acceptance in the market place.

Historically coal producers take one of two approaches in dealing with fine coal production. On the one hand, they may wash it and recover it as a wet cake which must be thermally dried prior to shipment. On the other hand, many operators make no attempt to recover fine coal, and dispose of it as a wet cake or slurry in refuse piles, slurry impoundments and abandoned deep mines. There are environmental problems related to both of these practices.

The Mulled Coal Process was developed as a means of overcoming the adverse handling characteristics of wet fine coal without thermal drying. The process involves the addition of a low cost, harmless reagent to wet fine coal using off the shelf mixing equipment. Based on laboratory- and bench-scale testing, Mulled Coal can be stored, shipped and burned without causing any of the plugging, pasting, carryback and freezing problems normally associated with wet coal. On the other hand, Mulled Coal does not cause the fugitive and airborne dust problems normally associated with thermally dried coal.

The objectives of this project are to demonstrate that:

- The Mulled Coal Process, which has been proven to work on a wide range of wet fine coals at bench scale, will work equally well on a continuous basis, producing consistent quality, and at a convincing rate of production at a commercial coal preparation plant.

- The wet product from a fine coal cleaning circuit can be converted to a solid fuel form for ease of handling and cost savings in storage and rail car transportation.

- A wet fine coal product thus converted to a solid fuel form, can be stored, shipped, and burned with conventional fuel handling, transportation, and combustions systems.

Project Overview

It is useful to describe the project in groups of activities in order to fully understand the interactions between activities and to better understand the information flow and decisions of the project. The project is organized around two major demonstrations: (1) the production of Mulled Coal in a commercial operating setting, and (2) the delivery of the Mulled Coal product through existing commercial storage, transport, and handling systems. An Information Flow Diagram is presented in Figure 1.

The initial project activities are performed largely at the EI facilities and will produce the formulations, test procedures, and design packages required to procure and install the Mulled Coal circuit at the Drummond Company, Inc. Chetopa Preparation Plant in Graysville, Alabama. The installed circuit will be used for the demonstration of Mulled Coal production. The second set of demonstrations will be the shipment and handling of Mulled Coal in existing coal transportation systems available to Drummond and ER&L, a subsidiary of CSX Transportation. Data collected from all phases of production and delivery will then be analyzed, evaluated and reported.

The Mulled Coal circuit will be installed in the operating preparation plant located at the Chetopa Mine site. The Chetopa plant processes 360 to 450 tonnes/hr (400 to 500 tons/hr) of raw coal to produce 250 to 320 tonnes/hr (275 to 350 tons/hr) of clean coal for shipment to the steam coal market. Approximately 45 to 55 tonnes/hr (50 to 60 tons/hr) of fine coal is cleaned in froth cells to produce 40 to 45 tonnes/hr (45 to 50 tons/hr) of a fine clean coal that is 10-14 percent ash. Froth concentrate reports to a vacuum filter where a 24-27 percent moisture filter cake is discharged to a collecting belt. In current operations, the wet filter cake is combined with the coarser size fractions of clean coal for storage and delivery to market. The wet filter cake comprises about 15 to 18 percent of the total clean coal product from the plant.

The proof-of-concept (or POC) circuit will process a 2.7 tonnes/hr (3 tons/hr) slipstream of wet filter cake into a free-flowing granular material and direct it to a 450 tonne (500 ton) open storage pile . The POC unit will be of a design that can be scaled up to 135 tonnes/hr (150 tons/hr). Figure 2 shows the key components of the Chetopa Plant cleaning circuit and the Mulled Coal circuit that will be installed.

The Mulled Coal circuit will be installed in an empty bay at the Chetopa Plant. This area is immediately adjacent to the vacuum filter and at a lower elevation. The use of gravity feeds will minimize field fabrication. Equipment will be installed to divert a 2.7 tonnes/hr (3 tons/hr) slipstream of the wet cake from the filter directly to a short transfer conveyor and then on to the Mulled Coal circuit. The Mulled Coal product will be gravity discharged from the circuit to a truck which will

haul the product to a stockpile which will be located at the edge of the active clean coal stockpile area.

During the 3-month operating period, the facility should produce 910 tonnes (1000 tons) of the Mulled Coal for evaluation in various rail transport configurations. Figure 3 provides a Project Flow Diagram for the Off Site Facilities. Bottom dump and bathtub type open top hopper cars will be loaded with straight Mulled Coal and blends of Mulled Coal with washed Chetopa coarse coal. The loaded cars will be shipped to various locations representing:

Rail/river barge terminals
Transshipment terminals
Industrial users
Utility users

The cars will be monitored and sampled during transit and dumping. Videotapes will be made during unloading and transfer operations to observe any differences between the test shipments and other coals being handled at the same facility.

PROJECT TECHNICAL WORK PLANS

Technical Approach and Work Plan Overview

This project focuses on achieving two demonstrations of the Mulled Coal technology: (1) Production in a commercial operating environment, and (2) Delivery of product through a representative cross-section of existing storage, transportation, and handling systems. To successfully complete these demonstrations, the project has been organized into a series of task activities which lead to the demonstrations, support the engineering and management needs of the project, and assess and report the activities and results. Further, the technical approach to structuring and accomplishing these work activities enables the key information and data base to be generated and used in support of the overall project work plans. The development of the design basis and assessment of Mulled Coal technology application are direct parallels to activities that would be needed in any specific individual commercial application.

The technical approach as depicted in the Information Flow Diagram (Figure 1) is comprised of the following:

1. Prepare work plans at the beginning of the project with mechanisms for adding detail and updating the plans as new information is generated.

2. Collect and evaluate information specific to the coal and plant operations at the host site that is needed to complete the circuit design, equipment selections, installation plans, and production scheduling and plans.

3. Use the evaluation results to complete the design, equipment selection, and production planning.

4. Procure, install, and start-up the Mulled Coal circuit at the host site.

5. Conduct the demonstration of production operations.

6. Select delivery destinations and develop specific plans for monitoring dumping, fuel handling, etc. at each unique destination. Final decisions and detailed plans will be made when coal deliveries are ready to be scheduled which in commercial practice is several months from the expected availability of product for shipment.

7. Conduct the demonstration of Mulled Coal technology in existing storage, transportation, and handling operations.

8. Prepare technical and economic assessment of the technology based on the data generated in the demonstration operations.

The key features to this approach include defined work plans, generation of information that enables specific decisions and contingencies to be addressed, and the utilization of experience to adjust the operations and data collection processes.

The work plans are consistent with the detail needed to direct and monitor the activities. This includes tasks in which the information that is specific to the coal and operations of the Drummond Chetopa facility will be used to update the work elements, make key decisions, or make revisions to the work plans as appropriate.

The evaluations and tests conducted in the early bench scale engineering activities will provide information needed to make key decisions. If we find that results fall outside the expected range, then the plans and the capabilities of the facilities and personnel are sufficiently versatile to revise the work plans.

Work Plan Assumptions

Developing the work plans has required making key assumptions which are:

1. The fine wet clean coal produced at the Chetopa Plant can be mulled using the experience base of reagent formulations and dosage rates.

2. The dilute froth concentrate will be a suitable alternative feedstock should the vacuum filter cake not be suitable.

3. The slipstream from the vacuum filter can be taken without disruption of the existing plant operations.

4. The storage, transportation, handling, and stability characteristics of Mulled Coal will be similar to the those properties as evaluated in the bench-scale engineering evaluations and testing.

CURRENT STATUS

The project work plans have all been developed and work on the engineering activities is currently continuing. The bench scale engineering has been analyzing the potential feed for the circuit, characterizing the mulling to affirm the reagent selection and dosage, and to determine the design criteria for the circuit. That task is well underway and should be completed by August. It is anticipated that the Mulled Coal proof-of-concept circuit will be designed, procured, installed and ready for production by December 1994.

MULTI-PARAMETER ON-LINE COAL BULK ANALYSIS

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ABSTRACT

Prompt Gamma Neutron Activation Analysis (PGNAA), has already been proven to be a viable technique for the on-line analysis of coal parameters such as the S, Cl, Fe etc. content of coal. The above technique utilizes very low energy (thermal) neutrons, usually emitted by a radioactive source. Two major drawbacks of the method as currently applied are a) only those elements that can be excited by thermal neutron capture can be measured, and b) ^{252}Cf radioactive sources are utilized which require a substantial shielding whether they are used or not. The utilization of pulsed fast-thermal neutrons will augment the PGNAA technique, and will allow the simultaneous on-line determination of the calorific value, % of volatile matter, and moisture of coal, along with all the parameters determined by PGNAA. Preliminary data indicate the feasibility of such a technique, at a cost comparable to the cost of a conventional PGNAA analyzer.

INTRODUCTION

Chemical and atomic methods have been and continue to be the traditional means for the measurement of various bulk parameters of industrial materials. Parameters such as the sulfur content of coal, its ash content, density, etc., are measured in an analytical laboratory environment with accuracy and precision. Most of the methods require an elaborate sampling procedure in order to produce a representative sample that has been removed from the bulk of the material, i.e. they are methods for off-line analysis. Because of the heterogeneity of coal and the dependence of its composition on the depositional environment, it would be more beneficial to monitor these parameters on a continuous basis. Such monitoring would allow the simultaneous optimization of the efficient burning of coal as well as the control of the emitted S.

These requirements cannot be met by developing a more accurate laboratory method. What is needed is the development of on-line methodology that would allow the continuous monitoring of the parameters deemed important to the coal industry. Any method developed for on-line analysis must be able to produce results which are (a) precise, (b) accurate, (c) continuous, and (d) rapid. Furthermore, the method must be able to operate in an instrumentally hostile environment (e.g, a coal conveyor belt or hopper).

Nuclear methods have been shown to be capable of allowing continuous monitoring of a number of parameters of importance to the coal industry. X-rays, gamma rays and neutrons have been successfully utilized for the on-line analysis of the ash content, sulfur content and density of coal.[1,2] A number of such units are commercially available and are already installed in power plants, coal preparation plants and mines.

While x-rays and gamma rays are capable of measuring bulk properties of coal, neutrons can give quantitative information on specific elemental content. For example, neutrons produced from a ^{252}Cf source have been used for the on-line determination of S.[3,4] The ^{252}Cf produced neutrons are thermalized mainly by the H content in coal. They are subsequently captured by

the S nucleus through the $^{32}S(n,\gamma)^{33}S$ nuclear reaction. The excited ^{33}S nucleus decays to the ground state, emitting a 4.52 MeV γ-ray. A number of other elements can simultaneously be excited along with S such as H, N, Si, Cl, Ca, and Fe. Whether a particular element can be quantitatively measured via the (n,γ) reaction depends on the thermal neutron capture cross section for each isotope of the particular element, and the amount of the element contained in a sample. For example, Cl has a very large (43 b) neutron capture cross section. Although the Cl content of coal is in the few hundreds of ppm range, it is possible to acquire an accurate quantitative measure of it.

There are other elements however, such as C and O, which cannot be determined with thermal neutrons. It has been shown[5] that there is an interrelationship between various coal properties, and these properties include C and O. For example, the following elemental relationships have been established:

Calorific value (BTU/lb) = a_1[%C] + b_1[%H] + c_1[%N] - d_1[%S] + e_1[%O] +f_1[%ash]
% volatile matter = a_2[%C] + b_2[%H] + d_2[%S]
density (g/ml) = a_3[%C] + b_3[%H] + d_3[%S] + e_3[%O] + f_3[%ash]

where the coefficients a_1, a_2, a_3 etc. have been experimentally determined by a number of authors.[5,6,7]

Based on the above, Table I shows the various elements that can be simultaneously measured through neutron induced reactions, and the various coal parameters that can be determined through them. The table includes only some of the elements that can be determined through thermal neutron capture, since this technique has already been fully developed.

Table I. Selected elements and relevant nuclear information

Element	Reaction	E_n^{thresh} (MeV)	Sigma (mb)	Eγ (MeV)	Coal Parameter
H	n,γ	-	332	2.23	% volatile matter
H	measure fast neutron absorption				Moisture
C	n,n'	4.8	200*	4.43	Calorific value, density % volatile matter,
N	n,n'	4.7	70*	2.31	Calorific value
N	n,γ	--	75	10.83	Calorific value
O	n,n'	6.4	96*	6.13	Calorific value, density
S	n,γ	--	520	5.42	Emission control % volatile matter

*Measured at E_n=14 MeV

The value of the coal parameters, other than moisture, listed in Table I cannot be currently determined on-line. Their measurement requires elaborate, time-consuming chemical methods. Capacitance measurement and microwave transmission have been commercially used for the on-line determination of moisture.[8*] These methods, however, are not suitable

when the conveyor belt contains metallic parts or the coal has high electrical conductivity, as is the case of coke. In these cases, fast neutron transmission would produce better results.[9]

EXPERIMENTAL PROCEDURE AND RESULTS

We are a developing a nuclear system that would simultaneously determine on-line the calorific value, % volatile matter, density, S content, and moisture of coal. The determination of the above parameters will depend on the quantitative measurement of a number of elements. To measure these elements, it is necessary to utilize the thermal neutron capture (n,γ) reaction for H, N, and S, and the fast neutron inelastic reaction (n,n') for C and O. Such a task requiring both fast and slow neutrons can be accomplished with a pulsed neutron generator. Fig. 1 shows a diagram of the pulsing of the neutron generator and the measurements

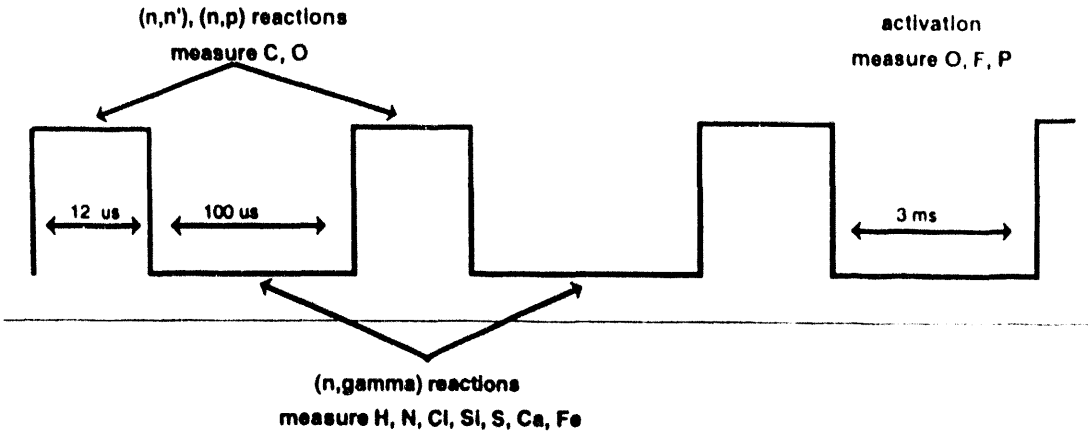

Figure 1. Schematic diagram of the neutron generator pulsed output.

performed. Neutrons are produced in short pulses. During this time interval, a data acquisition system is gated and γ-rays produced from (n,n') reactions are accumulated. At the end of the neutron pulse, the fast neutrons that were produced during the pulse pass through a neutron moderator and a percentage of them becomes thermalized. The pulse remains off for a longer period. During this time a different data acquisition system is gated and γ-rays from (n,γ) reactions are collected. In this manner, the two data acquisition regions are triggered separately, eliminating appreciably the background.

Proof-of-principle of this technique has already been shown.[10] Previous experiments have demonstrated the ability to identify C and O contained in a coal sample, along with N, S, and Cl. Since the algorithms that relate elemental content with calorific value, density, etc. depend not only on the presence of elements such as C and O but also on the amount contained in the sample, a series of measurements was taken with various chemical compounds that have different H, C, N, and O content (C/O ratios varying between 0.3 and 1, and O/N ratios between 0.5 and 2.5). The quantities of the materials used varied between 0.5 kg and 3.5 kg. Fig. 2 shows a schematic diagram of the neutron interrogation system. A sealed tube neutron generator produces pulses of 14.7 MeV neutrons through the d-T nuclear reaction. The pulse

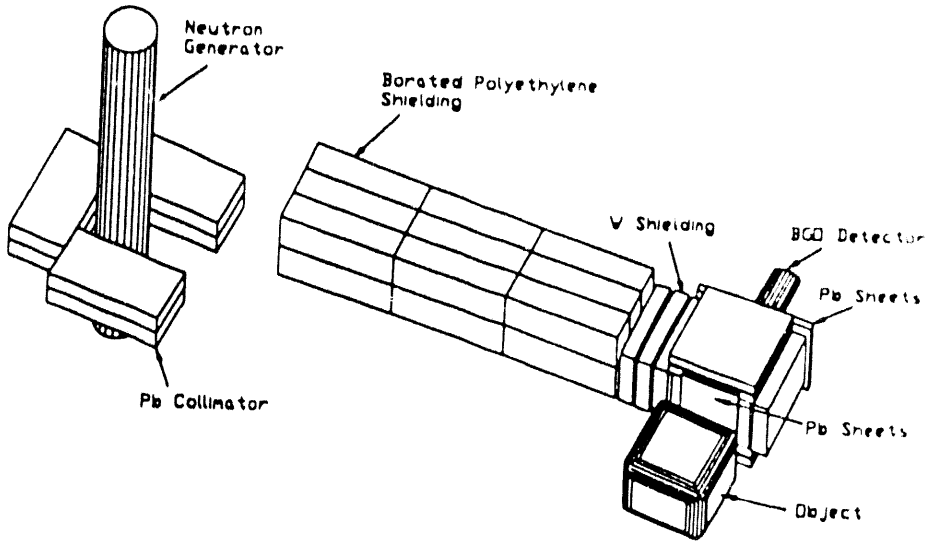

Figure 2. Schematic presentation of the measurements layout.

duration is approximately 10 μs and the pulse repetition rate varies between 8 and 10 kHz. The neutron generator is computer controlled and all essential parameters are remotely monitored through a fiber optics cable connecting the generator console with the computer. The emitted neutrons impinge onto the interrogated object, and the γ-rays that result from the nuclear reactions are detected in BGO detectors. The thermalization of the fast neutrons occurs during the time interval between the neutron pulses, via inelastic collisions that the fast neutrons suffer with the light elements contained in the interrogated object. The Pb collimator around the neutron generator is also part of the system that aids the thermalization of neutrons. The BGO detector is shielded from the neutrons coming from the neutron generator with a substantial borated polyethylene shield. W and Pb shielding around the detector minimize the γ-ray background at the detector position. Figures 3 and 4 show some of the results obtained with urea $\{(NH_2)_2CO\}$.

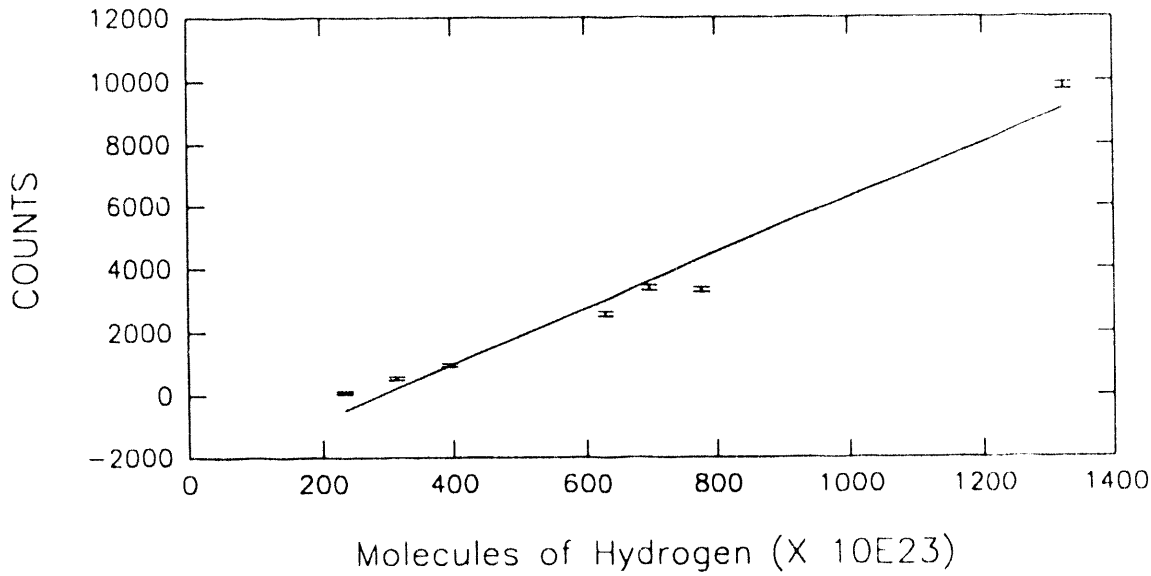

Figure 3. Hydrogen γ-ray yield for various quantities of urea.

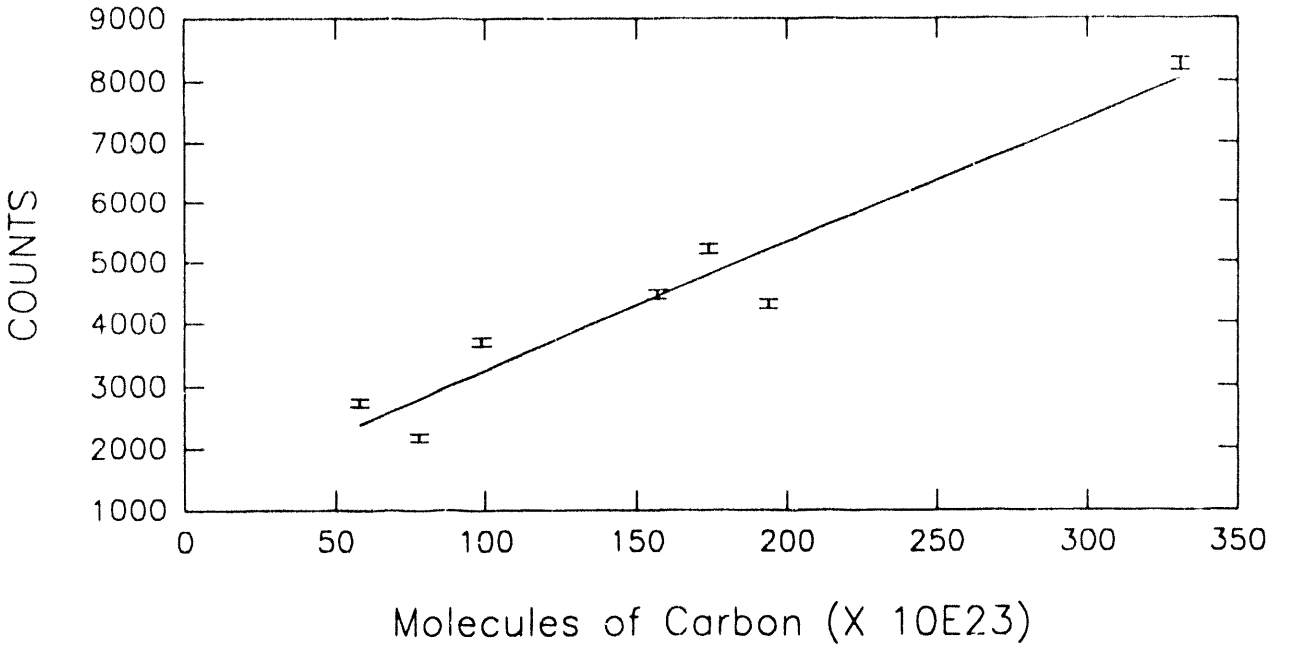

Figure 4. Carbon γ-ray yield for various quantities of urea.

In these figures it can be seen that the amount of H and C in urea can be determined with better than 90 % precision over a sevenfold increase of the size of the sample. Similar results were obtained with other chemical compounds.

CONCLUSIONS

The previous results indicate that the pulsed fast-thermal neutron interrogation technique has the potential to quantitatively measure on-line the elemental content of coal, and thus afford, through the appropriate algorithms, the determination of quantities such as calorific value, % volatile matter, S content etc. The measurements are reliable over a wide range of the quantity of the material involved, although self-shielding and increased neutron thermalization as a function of the volume of the interrogated object should be taken into consideration. The above measurements were obtained with pure chemical compounds. In the actual case of coal, matrix effects need to be taken into consideration. Elements such as Fe, Cl, and Ca emit γ-rays that can interfere with those of the elements of interest. Spectral unfolding techniques, and expert systems are under development for the reduction and meaningful evaluation of the coal data.

ACKNOWLEDGEMENT

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BENCH SCALE TESTING OF MICRONIZED MAGNETITE BENEFICIATION

DOE CONTRACT NO. DE-AC22-93PC92206

AMAX R&D Center
Kurt Anast

INTRODUCTION

Amax Research & Development Inc. (Amax R&D) was awarded a cost-shared contract to design, fabricate, install and operate a 500 lb/hour fine coal cleaning circuit in the Emerging Technologies (ET) area at PETC's Coal Preparation Process Research Facility in Pittsburgh, Pennsylvania. This project is one of three awarded under the "High Efficiency Separation" subprogram to be conducted at PETC's CPPRF. The project, "Bench Scale Testing of Micronized Magnetite Beneficiation", No. DE-AC22-93PC92206, addresses the cleaning efficiency program area. Similar processes based on this concept have been successfully researched at PETC and elsewhere but have not been studied in a continuous bench-scale unit.

The project is aimed at development of a process that, by using an ultra fine magnetite suspension, would expand the application of dense medium separation technology to include processing of fine, -28 mesh coals. These coal fines, produced during coal mining and crushing, are separated in the conventional coal preparation plant and generally impounded in a tailings pond. Development of an economic process for processing these fines into marketable product will expand the utilization of coal for power production in an environmentally acceptable and economically viable way.

The proposed circuit will utilize ultrafine magnetite and a heavy media cyclone to impart a gravity separation on fine coal and use a variety of magnetic recovery devices. The proposed circuit is composed of three unit operations which can be evaluated separately and then combined in an integrated fashion. The Test Plan will focus on magnetite recovery, as this portion of the process is critical to economic viability and has not been adequately evaluated in the laboratory. Specifically, fine magnetite loss and magnetite contamination will be closely examined. High efficiency screens, conventional drum magnets, rare earth , and high gradient magnets will be evaluated separately and together in the magnetite recovery circuit.

PROJECT DESCRIPTION

PROGRAM OBJECTIVES

The proposed project will provide valuable operating information regarding the use of micronized magnetite media for fine coal cleaning. The overall objective of the proposed project is to determine if this technology should be considered for commercial installation at an operating coal mine. This project will allow high-quality bench scale data to be collected in a timely and cost-effective manner. Three primary objectives have been identified to achieve the overall project objective:

1. **Verify the effectiveness of the Micro Mag process at rejecting ash and sulfur from the fine fraction of various coals on a continuous bench scale basis.**

2. **Collect operating data which will determine the economic viability of the Micro Mag process in order to determine its commercial potential.**

3. **Collect engineering data for scale-up of the process for commercial operations.**

The proposed concept as tested in the CPPRF will provide operating and economic information relevant to commercial operations. The three feed stocks prepared from each coal are similar to the potential feed sources available commercially. The most prevalent slip stream that could be tested in the near term is fines screened from the run-of-mine coal.

APPROACH

The team of Amax R&D, CLI, and Amax Coal Industries was formed to accomplish the objectives of the project. Amax R&D will be the prime contractor and manage the project, operate the ET unit, perform sample analyses, technical evaluation, and economic evaluation. To enhance the possibility of successfully accomplishing the project goals within the schedule constraint and in a cost effective way, Amax R&D has teamed up with CLI Corporation, Pittsburgh. CLI is an engineering and construction company specializing in design and construction of coal preparation plants. CLI will perform the engineering design and assist in technical and economic evaluation. A company experienced with process fabrication will be selected for constructing and dismantling the plant. Amax Coal will also assist in selection and procurement of feed coal samples and evaluation of results to determine commercialization potential.

The primary goal of the proposed program is to investigate the technology in a continuous circuit at a reasonable scale to provide a design basis for larger plants and a commercial feasibility study. To accomplish this goal, the project is divided into the following eight tasks which will be completed over a 24 month period:

Task 1. Project and Test Planning
Task 2. Engineering and Design
Task 3. Procurement and Fabrication
Task 4. Installation and Shakedown
Task 5. Sample Analysis and Characterization
Task 6. Operation/Testing
Task 7. Technical and Economic Evaluation
Task 8. Decommissioning and Removal

The test work will be conducted at PETC's CPPRF located in Pittsburgh, Pennsylvania. The circuit will be installed in the High Bay area of the CPPRF referred to as the Emerging Technology area. The required environmental, health, and safety aspects for pilot plant type operations have been implemented for the CPPRF. This project will utilize the necessary faciiities such as solids handling, feed preparation, and waste handling, which are in place. Specially designed platforms and equipment will be installed according to the engineering drawings provided by CLI.

The installation phase is scheduled to begin on July 1, 1994. The schedule of activities at the CPPRF are :

- Installation & Shakedown (July - September)
- Component Testing (October& November)
- Integrated Testing (December)
- Extended Runs (January)
- Decommissioning (February)
- Final Report (March)

Total on-site activity will last seven months from July 1994 through February of 1995.

SYSTEM DESCRIPTION

The circuit design reflects a balance between cost and project objectives. The final circuit design includes suggestions and comments from PETC and other interested parties.

As shown in Figure 1 the proposed flow sheet employs:

- A multi-stage classification circuit to properly size the feed coal to the heavy media cyclone.
- A correct medium sump-wing tank arrangement to control the density of the medium and to deliver feed coal and medium to the cyclone.
- Drain screen and rinse screen of Sizetech design and of different sizes to recover heavy media and rinse cyclone products of adhering medium.
- Combined drum, rare earth, and high-gradient magnetic separators for multi-stage magnetic recovery circuits or for individual evaluation.

The preliminary design of the proposed circuit is composed of two major subcircuits working together, the heavy media cyclone circuit and the magnetite recovery circuit.

Heavy Media Cyclone Circuit - Feed coal in the form of a coal-water slurry is prepared and then delivered to the classifying cyclone sump of the Micronized Magnetite ET circuit. The coal slurry is fed to the slimes cyclone with a centrifugal pump where minus 30 micron fines are removed and sent to the high rate thickener of the CPPRF. The cyclone underflow is repulped and screened across a vibrating Sizetech screen to remove all potential slimes. Slimes from the screen are recirculated to the classifying cyclone sump.

The coarse cyclone underflow is sent to the fine heavy-media cyclone feed sump. Magnetite from the magnetite recovery circuit and deslimed coal are mixed and pumped to the heavy-media cyclone for cleaning. Cyclone underflow and overflow is passed across two dual media recovery drain screens, one set for refuse and one set for product. The drain screens recover most of the magnetite along with some fine coal. The drain screen undersize is recycled to the heavy-media cyclone feed sump. Oversize from the drain screens reports to the rinse screens. The rinse screens further separate magnetite and coal before the undersize streams are sent to the magnetic separator circuit. Underflow from these screens is fed to the magnetite recovery circuit. Oversize from the drain screen constitutes the final cleaned product and refuse.

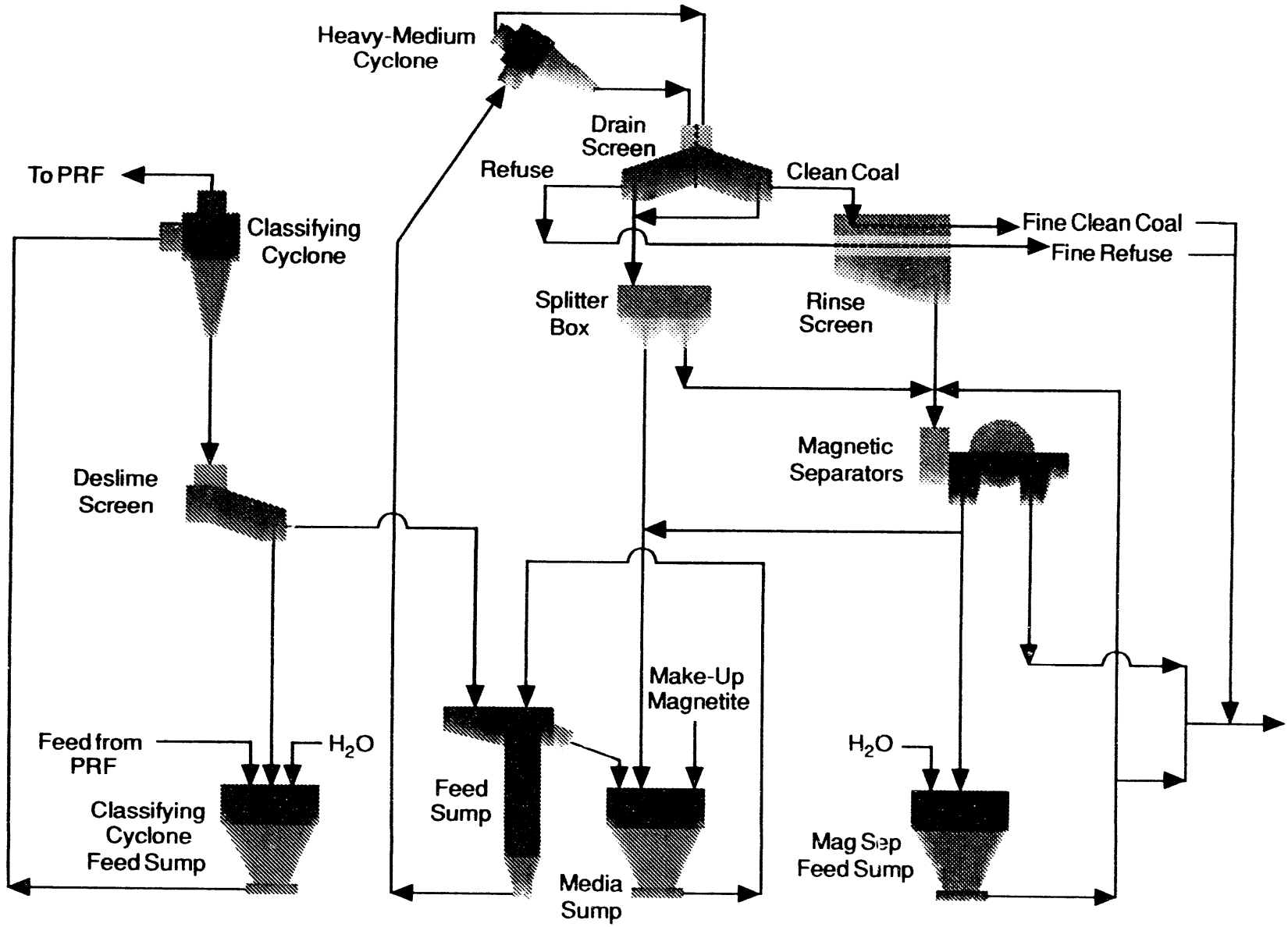

Figure 1 - Proposed Micronized Magnetite Process Flowsheet

Magnetite Recovery Circuit - Five drum magnetic separators and a high gradient magnetic separator (HGMS) will be employed in the circuit. Four conventional wet drum separators and a rare earth drum magnetic separator will be configured as in a rougher, cleaner, scavenger circuit. The HGMS will also be evaluated separately and in conjunction with the drum separators. The rinse screen undersize streams are sent to a rougher recovery circuit consisting of two rotating drum separators. The magnetite stream is then cleaned in a single stage rare earth drum before being returned to the cyclone feed sump. The rougher reject is sent to a two stage circuit consisting of conventional drum magnetic separators for scavenging any remaining magnetite. The magnetics recovered in the scavenger circuit is returned to the cyclone feed sump. Cyclone underflow or overflow streams could be directed to the magnetic separation circuit without prescreening to evaluate the importance of screens on magnetite recovery.

The HGMS will be used as a separate magnetite recovery. The unit can be tested (1) as a stand alone magnetite recovery unit, (2) substituted for the rare earth drum as a scavenger, and (3) as the cleaner.

Magnetite from the first rinse screens and from the magnetic separators is repulped in the heavy-media sump and then pumped to the heavy-media cyclone feed sump. A level control and a nuclear density gauge will control the addition of water and magnetite to the heavy-media sump.

Fines from the classifying cyclone and from the magnetic recovery circuit are sent to the static thickener for recovery. Water from the thickener is recycled for use in the ET circuit.

The proposed circuit will fit into the provided ET staging area at the CPPRF. The equipment will be installed onto a support structure which will be installed prior to equipment connection. The equipment will be stacked four levels high utilizing gravity as much as possible. The cyclone will be located at the top of the unit and the drain screens below the cyclone. The magnetic separators will be located below the screens along with the magnetite feed bin. The sumps and feed pumps will be located on the floor along with the product and refuse collection bins.

FEED PREPARATION

Coal will be processed for the micronized magnetite circuit using the existing coal preparation equipment in the CPPRF. Feed coals to the CPPRF will be delivered off site to a local holding facility where the coal will be blended and stored. Delivery in increments of 5 tons will be arranged to minimize storage of coal at the CPPRF.

Component and process variable testing will be performed using ROM Pittsburgh coal crushed to 28 mesh top size, minimizing the amount of coal required for the project. Raw coal is reduced to a top size of 1/2 inch through a two-stage double roll crusher and diverted to two (2) five ton weigh bins equipped with a vibrating bin bottom. Each weigh bin discharges onto weigh feeder which meters coal onto a flight conveyor. The coal can be stored in two 2 1/2 ton storage bins for feeding to the dry hammermill where the coal is reduced to minus 28 mesh. The coal will then be fed directly to the micronized heavy media circuit.

PROCESS CONTROL

The circuit will be controlled through the use of a control panel located on the top floor of the ET area. The control panel will include the circuit electrical and pneumatic instrumentation, start/stop buttons, instrument power supply, relays, and computer interface. The equipment will be started from the panel and can be stopped in the field with a local interrupt or at the panel. A computer interface will be set up to collect operational data. The computer will not start or stop equipment nor will it control any parameters such as pump speed or magnetite addition. Control of these parameters will be handled with simple digital instruments and controls. All of the sumps in the circuit will be controlled with multiple point capacitance level indicators that control the addition of material into the sumps. All of the sumps operate in a water addition mode to maintain a level. In the event that a sump overfills, the sump will discharge into the available floor sumps for collection.

PROJECT STATUS

The merger of Cyprus Mineral Company and AMAX Inc. to form Cyprus Amax Minerals Company caused the present delays to the project. The merger which was approved by both companies on November 14th, 1993 significantly affected Amax R&D, Inc.. Cyprus Amax Minerals Company decided to close Amax R&D Center as of December 29, 1993. All activities will cease at the Center except those associated with DOE contracts. Cyprus Amax will determine their interest in the contracts and take appropriate action to ensure the contract obligations are met as well as the company's interests. Since it was known the Center would be closing upon shareholder approval of the merger, project activities were minimized in order to save funding and a decision was made to complete work on Tasks 1 and 2 only. The remaining tasks as shown in the Work Breakdown Structure will be scheduled after contract issues are resolved.

TASK 1. PROJECT PLANNING

The Preliminary Project Work Plan was prepared and submitted. The Project Work Plan includes all the details relating to responsibilities, timing, scheduling, costs, objectives, as well as preliminary drafts of Task plans. Initial environmental, safety, and health planning has also been included.

TASK 2. ENGINEERING AND DESIGN

CLI Inc. prepared the final design of the bench scale test circuit. The final design was reviewed at the Final Design Meeting held in December. Two complete sets of drawing were left with the DOE for comments. The circuit design was reviewed and a proposed start date is presently not defined. The final design report is pending due to the contract novation process.

TASK 4. INSTALLATION AND SHAKEDOWN

As part of this task an Environmental, Safety, and Health (ESH) Plan was prepared for review by the COR and interested parties. The ESH plan was provided to the DOE because it is a function of the circuit design and intended operating philosophy. It can be used in conjunction with the final design for necessary permitting.