ELEVENTH ANNUAL COAL PREPARATION, UTILIZATION, AND ENVIRONMENTAL CONTROL CONTRACTORS CONFERENCE

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PROCEEDINGS

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EVALUATION, ENGINEERING AND DEVELOPMENT OF ADVANCED CYCLONE PROCESSES

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CONTRACT NUMBER: DE-AC22-90PC90177

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INTRODUCTION

This research and development project is one of three seeking to develop advanced, cost-effective, coal cleaning processes to help industry comply with 1990 Clean Air Act Regulations. The specific goal for this project is to develop a cycloning technology that will beneficiate coal to a level approaching 85% pyritic sulfur rejection while retaining 85% of the parent coal's heating value. A clean coal ash content of less than 6% and a moisture content, for both clean coal and reject, of less than 30% are targeted. The process under development is a physical, gravimetric-based cleaning system that removes ash bearing mineral matter and pyritic sulfur. Since a large portion of the Nation's coal reserves contain significant amounts of pyrite, physical beneficiation is viewed as a potential near-term, cost-effective means of producing an environmentally acceptable fuel.

The project agenda consists of three phases. Phase I included a characterization of the four potential feedstock coals, one of which is to be used throughout the project, and a media evaluation consisting of a paper study and laboratory testing. The coal seams selected for use in this project are Upper Freeport, Pittsburgh No. 8, Meigs No. 9, and Illinois No. 6. Phase II involved testing several selected media and separator combinations in a closed loop circuit, conceptual cost estimates, and final medium/process selection. Phase III involves the development of a process Component Test Circuit design, equipment selection, construction and operation of a 1,000 lb/hr closed loop component test system. Prior to medium/separator testing, the selected deliquoring device will be performance tested utilizing conventional froth flotation products to determine applicability to existing coal preparation technology. During the process optimization period, performance tests with one of the four test coals are planned. Finally, generation of a conceptual design and economic analyses for large scale integrated plants is planned.

Nearly 50 media candidates were considered including aqueous suspensions, aqueous solutions, organic solutions and a magnetically enhanced medium (MEM).

Separators that were considered included small diameter, high pressure cyclones, centrifuges, a hybrid separator combining flotation and cycloning in a single device, and a separator designed for MEM. In addition to gathering physical property data via a literature search, laboratory measurements and test separations were completed to eliminate the less desirable media candidates, i.e., viscosity, solubility, pH, and filter cake washing experiments.

COAL TOPSIZE, MEDIUM AND SEPARATOR SELECTION

At the conclusion of Phase I, calcium nitrate/water and the organic mixture of methylene chloride/perchloroethylene (mcl/perc) were selected as the best true heavy liquid candidates for further testing in Phase II. The MEM separator could not be readily scaled up and was dropped. The separators tested in PTI's closed loop facility during Phase II were a Krebs Cyclone, an Alfa-Laval (A-L) Model MOCL hydrocyclone (nested miniclones), and a DOE/PTI vertical centrifuge separator. Testing of decanter centrifuges and Hydro Processing & Mining's Centrifloat separator occurred off-site. All of the indicated separators were tested with both aqueous and organic media except the Centrifloat, which utilizes a water only medium, and the A-L centrifuge, which did not use the organic medium because the lab where it was tested was not equipped to handle organic liquids. HPM's Centrifloat did not achieve the targeted value for pyritic sulfur rejection and was dropped. The vertical centrifuge separator required modification beyond the budget and timing of this project and was eliminated.

Coal characterization and washability of the four test coals showed that grinding the Component Test System feed coal to 100 mesh topsize would theoretically achieve the project goals. Phase II tests were conducted utilizing 100 mesh as the top size; however, 200 mesh and 500 mesh feed coals were also tested to determine separation effectiveness at smaller topsizes.

The separating performances of the calcium nitrate medium in a decanter centrifuge and the organic medium in a two-inch diameter cyclone were nearly identical, making the final separator selection a difficult task. The organic medium circuit is less costly than the salt circuit, both from a capital and operating cost standpoint. However, the calcium nitrate medium has significantly less health, safety and environmental liabilities. If a spill with the organic medium were to occur, it would shut the operation down, cause serious delays, and have the potential for very large clean-up and containment related costs. The difference in cost of the two circuits is viewed as the price paid for safety. Calcium nitrate is more commonly used for its nitrate content as fertilizer and at saturation is able to make a 1.55 sg solution. Viscosity of a 1.35 sg solution is less than 5 centipoise.

Selection of the decanter centrifuge as separator is required to meet the project goals if calcium nitrate medium is used. The high g force generated by the centrifuge is required for the calcium nitrate medium to work as a parting liquid. The Project Team achieved excellent separations with a P-660 Sharples (A-L) decanter. Comparison of the optimum separation results achieved in Phase II to the feed washability (ash vs yield relationship) is shown in Figure 1. Partition curves for the calcium nitrate/decanter centrifuge separation and others are shown in Figure 2. After considering these results and the environmental and safety elements above, the Project Team selected the calcium nitrate medium, decanter centrifuge combination. A decanter centrifuge diagram is shown in Figure 3.

MEDIUM RECOVERY/REGENERATION (FILTERING, WASHING, EVAPORATION)

Development of a medium recovery system is a technical and economic requirement. The filtering system serves two functions: to recover medium and to deliver a relatively salt-free product. This operation is critical to the success of the project. The cost of the medium requires that it be recovered for reuse. Water is used to rinse the filter cake, and the dilute filtrate is recovered for restoration to the original medium gravity. The operating cost of the aqueous process is dominated by the medium regeneration, i.e., water evaporation and calcium nitrate replacement costs.

Test work was performed with two filter vendors to determine filtering efficiencies, capacities, etc. A capillary-effect filtration system utilizing ceramic disks manufactured by Outokumpu of Finland was selected. A diagram of the Outomec filter is shown in Figure 4.

By utilizing a porous sintered alumina material to create a capillary effect, the separation of liquid and solids can take place. Capillary effect causes liquids in small tubes to stand at a greater height than the corresponding static head. Capillary rise is described by:

$$H = \frac{4t \cos \theta}{g\ D\ (\rho_1 - \rho_2)}$$

where H is in cm, t is the surface tension of the liquid in dynes/cm, D is the diameter in cm, θ is the contact angle, $g = 981$ cm/sec^2, and ρ_1 and ρ_2 are the densities of the liquid and surrounding gas in g/cc.

If the height of the column H is multiplied by the difference in density and the acceleration due to gravity, the differential in pressure is obtained.

$$\Delta P = \frac{4t \cos \theta}{D}$$

The above expression indicates that the smaller the pore diameter, the greater the pressure. Capillary force in pores can be greater than that applied by vacuum. Due to the surface tension between the liquid and the hydrophilic porous material, the pores fill with liquid. The pores in the ceramic filter are of such a diameter that they are not emptied of the liquid contained in them and no air is allowed to pass. A small pump is used to convey the collected liquid away. Energy consumption is extremely low.

With the use of ceramic filter media, filter cloths and associated problems are eliminated. The ceramic media is non-blinding, non-tearing and provides an uncontaminated filtrate.

Preliminary process investigation indicates the optimum water evaporation system is a seven-effect evaporator utilizing falling-film and forced circulation units.

The device removes water from the diluted medium, restoring the working medium to its proper density. A diagram of the evaporator components is shown in Figure 5.

Since the medium regeneration system utilizes well understood technologies involving thermal evaporation of water, an evaporator manufacturer will be selected to perform the studies necessary for selection of the optimum medium regeneration evaporator configuration.

DESCRIPTION OF PROCESS CIRCUIT

Detailed engineering design of a Bench Scale Circuit (BSC) capable of processing coals at a feed rate of 1,000 lb/hr was completed in late 1993 but cost constraints and limited available funds dictated a change in project scope. A flowsheet of the BSC is shown in Figure 6. After study of project goals vs. available funds, it was decided that rather than construct the complete integrated circuit, portions of the detailed engineering and the primary circuit components will be utilized to construct a Component Test System (CTS), which will test, in closed loop at 1,000 lb/hr, first the capillary-effect filtration system with a standard preparation plant flotation product and tailings, water slurry, then the separator circuit with aqueous medium and preground project coals, and finally the deliquoring circuit with separator products. In the first phase of testing, the Outomec capillary-effect filter will be tested to determine its potential effectiveness as a dewatering device in existing coal preparation plants. Next, preground samples (100 mesh topsize) of the project coals will be slurried in a sump with calcium nitrate/water medium and beneficiated through the Alfa-Laval P-3000 decanter separator where a gravimetric separation of high and low ash material will be made. The decanter products are either directed back to the feed sump and recirculated or stored/segregated in separate sumps. With the completion of processing/sampling of the project feed coal through the high g separator, the decanter test loop is shut down. The tube and ceramic disc filter circuit, is then started. The stored/segregated low ash (product) solids are pumped to the clean coal side of the deliquoring circuit, comprised of the tube filter and three discs of the ceramic disc filter. Simultaneously, the high ash (refuse) solids are pumped to the refuse side of the filter, comprised of a single disc. The solids are deliquored/sampled and directed either to the feed sumps for recirculation or to final disposal.

CURRENT STATUS AND PLANS

Though this process has great potential, additional work is needed to lower processing costs. This need and cost constraints of the project led to the decision to adopt a simple component test circuit strategy. Construction of the component test facility is expected to be complete in the Spring of 1995, and the test program and final report completed in April, 1996.

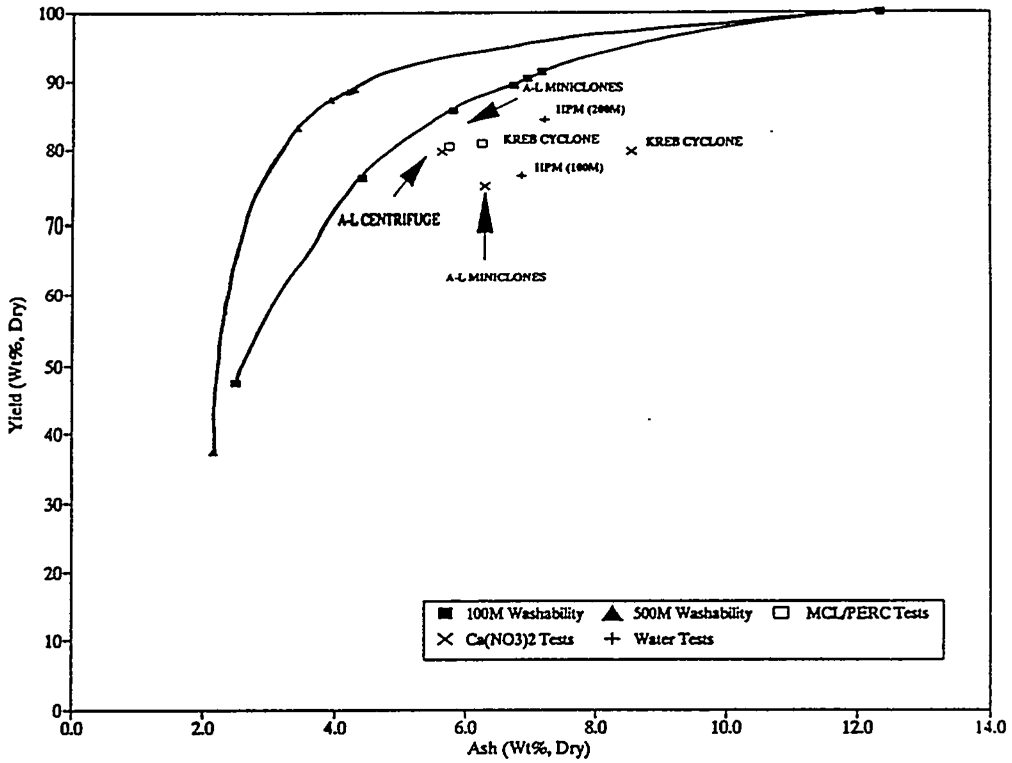
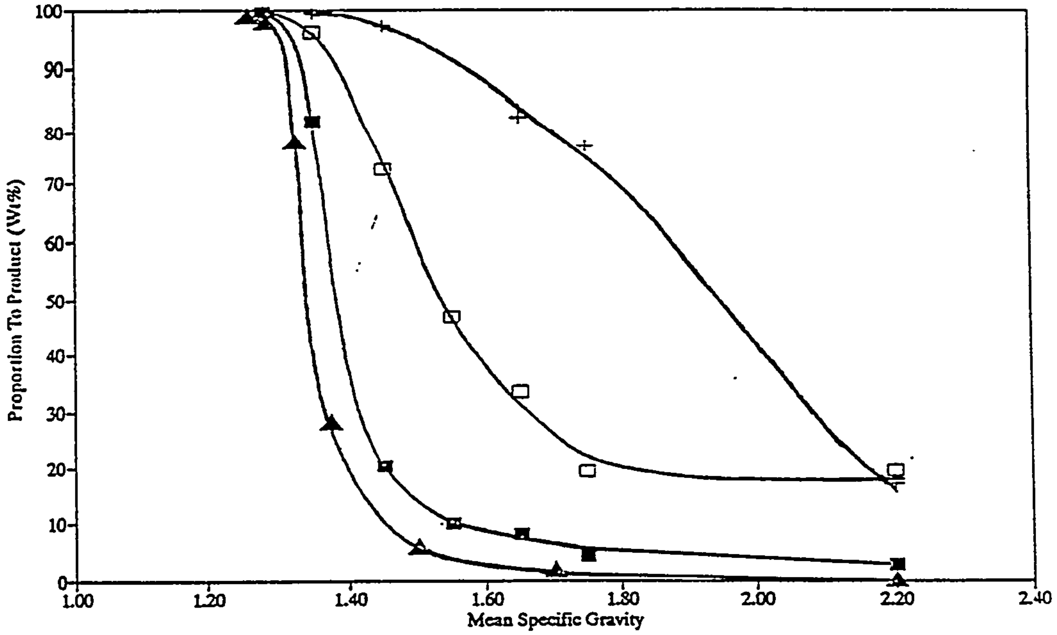


Figure 1. Comparison of Optimum Separation Results



Legend	Media	Separator	Size	Media SG	Sep'n SG	Ep
▲	Micromag	3" Cyclone	28M x 400M	1.30	1.35	0.025
☐	MCL/PERC	2" Cyclone	100M x 0	1.45	1.53	0.135
■	Ca(NO3)2	A-L Centrifuge	100M x 0	1.35	1.39	0.035
+	Ca(NO3)2	A-L Centrifuge	500M x 0	1.40	1.92	0.195

Figure 2. Partition Curves of Media/Separators

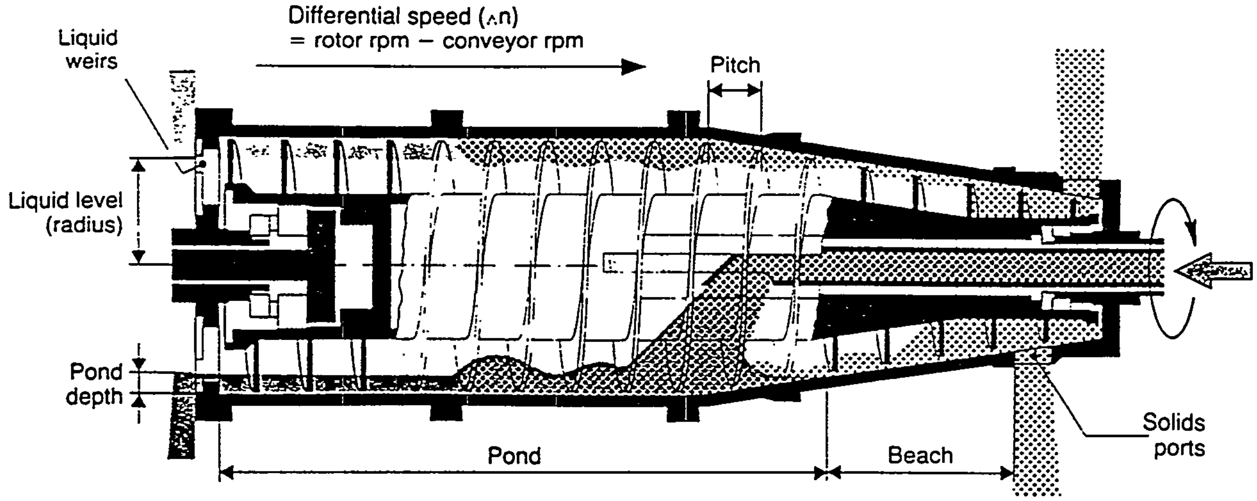


Figure 3. Decanter Centrifuge

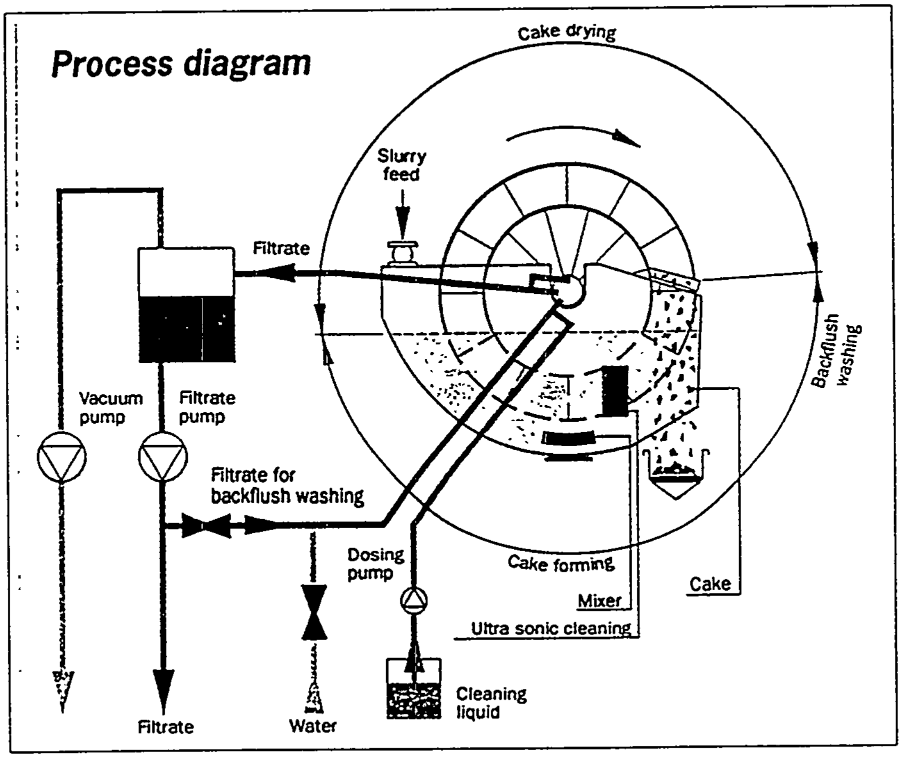
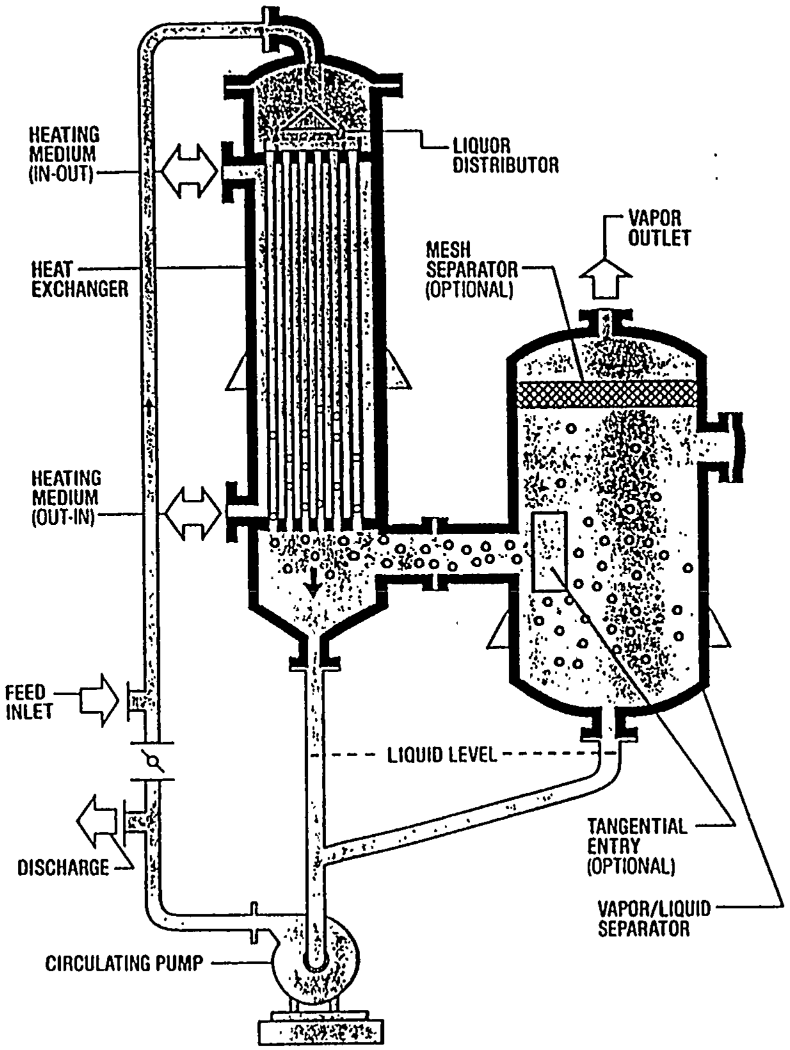
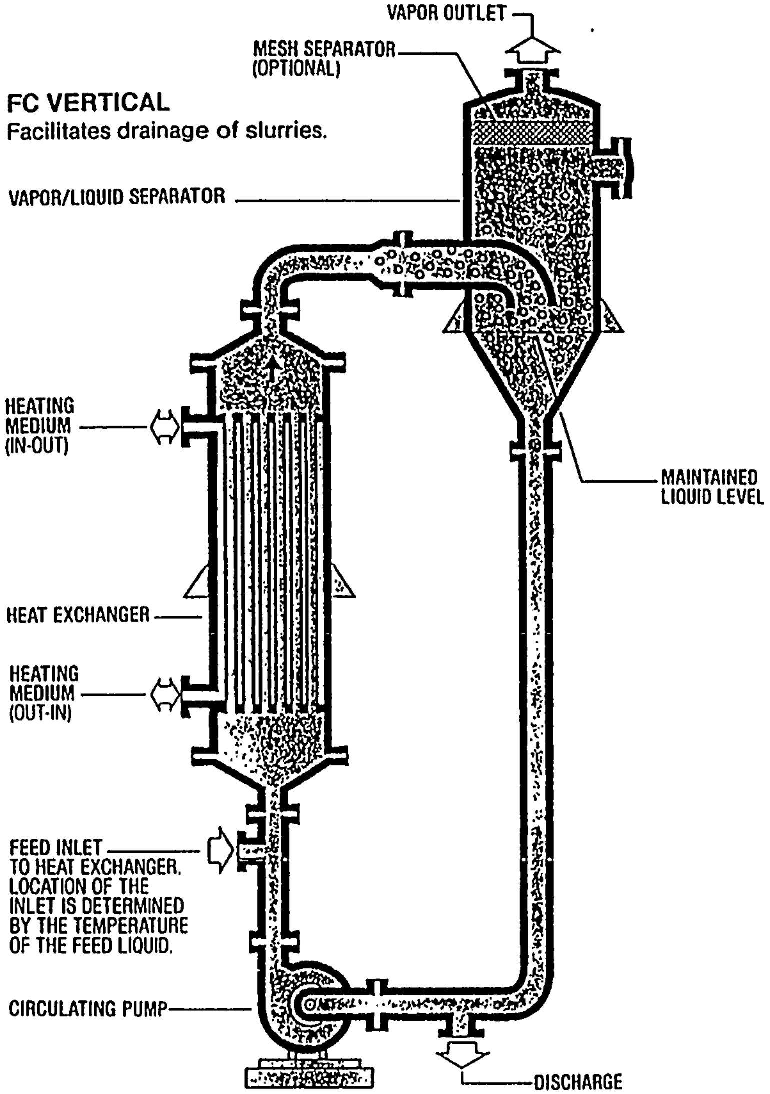


Figure 4. Capillary Effect Filter



Falling Film Evaporator



Forced Circulation Evaporator

Figure 5

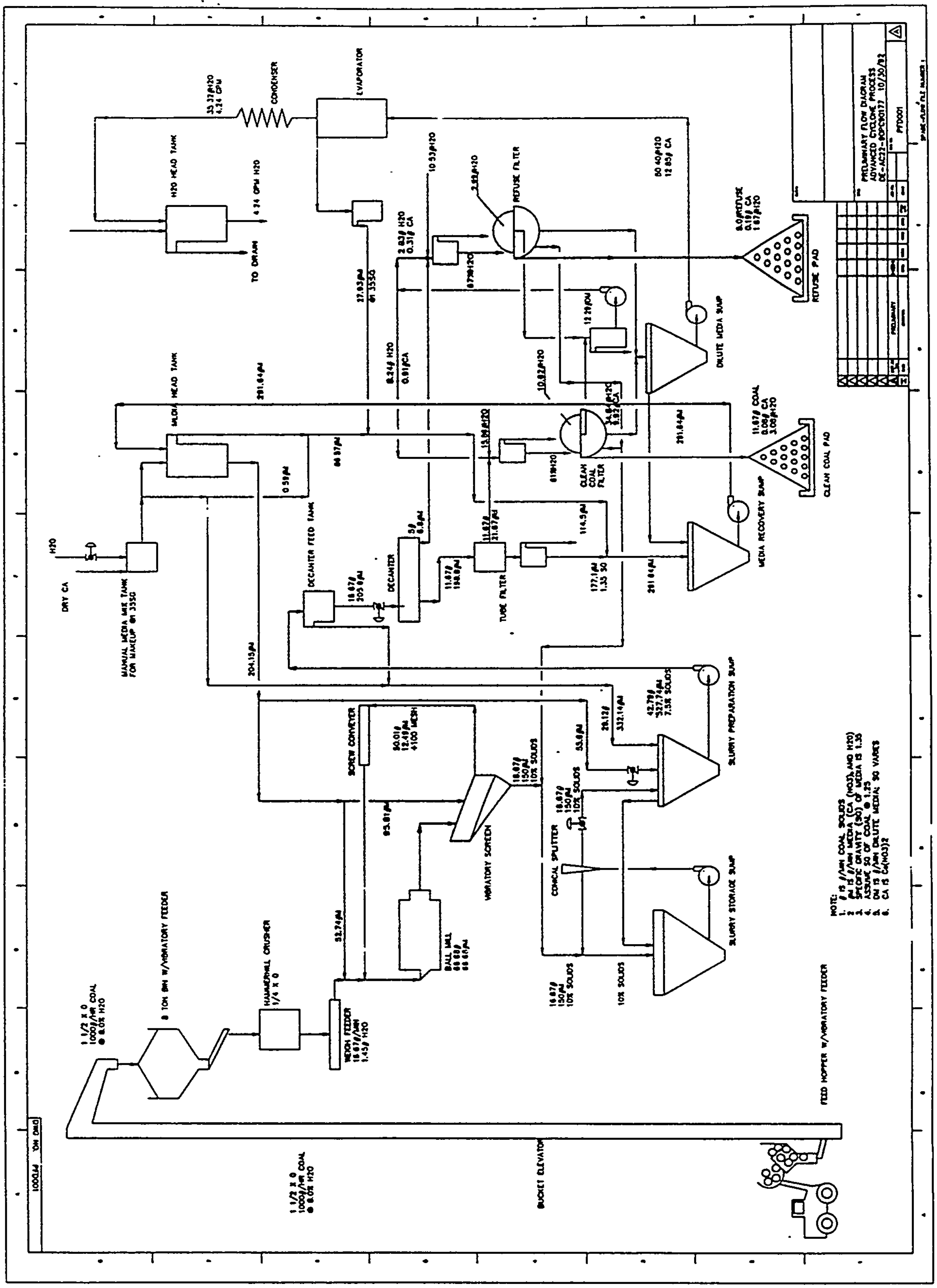


Figure 6. Bench Scale Circuit Flowsheet

ENGINEERING DEVELOPMENT OF ADVANCED PHYSICAL FINE COAL

CLEANING TECHNOLOGIES - FROTH FLOTATION

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In 1988, ICF Kaiser Engineers was awarded DOE Contract No. DE-AC22-88PC88881 to research, develop, engineer and design a commercially acceptable advanced froth flotation coal cleaning technology. The DOE initiative is in support of the continued utilization of our most abundant energy resource. Besides the goal of commercialability, coal cleaning performance and product quality goals were established by the DOE for this and similar projects. Primary among these were the goals of 85 percent energy recovery and 85 percent pyrite rejection. Three nationally important coal resources were used for this project: the Pittsburgh No. 8 coal, the Upper Freeport coal, and the Illinois No. 6 coal. Following is a summary of the key findings of this project.

Proof-of-Concept (POC) Scale Test Results

The POC testing circuit was constructed as a addition to the John P. Apel Ohio Coal Testing and Development Center (OCTAD) located between Beverly and McConnellsville, Ohio. OCTAD is a state-of-the-art coal cleaning facility that contained much of the equipment needed to pre-clean the coal for the POC test program. A separate bay was added to the OCTAD facility to house the additional process equipment needed for the DOE POC test program. Many modifications were needed to the piping, electrical and process control systems to properly integrate the POC process into the OCTAD operation.

Figure 1 represents a simplified block flow diagram of the POC flowsheet. In the process, 1/4 inch x 48 mesh was cleaned using a heavy-media cyclone with the objective of removing coarse refuse from the coal matrix while at the same time maximizing the recovery of energy values. Clean coal from the heavy-media-cyclone overflow stream was crushed to minus 48 mesh using a cagepactor crusher and combined with the raw 48 mesh by zero and fed to a water-only cyclone. The water-only cyclone was used to remove liberated mineral matter and pyrite from the coal prior to grinding to finer sizes for additional liberation. Clean coal from the water-only cyclone reported to the ball-mill where it was ground to nominal 200 mesh by zero and fed to the column flotation cell for final cleaning. The microcel™ column flotation technology, developed at Virginia Polytechnic Institute and State University, was used in the POC flowsheet. Primary performance goals of the POC flowsheet were to achieve 85 percent energy recovery with 85 percent pyritic sulfur removal when compared to the raw coal feed of the plant.

Prior to the 24-hour demonstration runs for the three coal seams tested, each coal was subjected to a series of optimization runs to determine the effect of changing process variables and set-points on cleaning performance. Tests were completed to determine the optimum conditions to run the heavy-media cyclone and water-only cyclone operations. In addition, important variables were examined for their impact on the ability of the grinding mill to achieve fine grinding performance. To determine the best conditions for microcel™

column flotation cell, a fractional factorial experiment was completed to determine the best operating levels for frother type and amount, wash-water rate and aeration rate. Details of these results can be obtained in the reports filed for the project with DOE.

With the heavy-media cyclone and water-only cyclone maximized, and with the best conditions for aeration rate, wash-water rate, and frother type determined for the column flotation cell, investigators conducted a Box-Behnken test matrix on each of the three coals. Profiles were developed showing the energy recovery as a function of ash rejection, pyritic sulfur rejection, and total sulfur rejection for the overall POC circuit based upon Box-Behnken test matrices for the Pittsburgh No. 8, Upper Freeport, and Illinois No. 6 coal seams. As before, the results of these studies are available from DOE under separate cover.

To conduct the 24-hour demonstration runs, all process variables in the POC were pre-set at their optimum values as determined in the previous statistical studies. During the 24-hour tests, a complete set of samples were collected every eight hours for all of the pertinent circuits in the POC operation. Statistically adjusted material balances were constructed for each eight hour period using the Bilmat computer software. All of the eight hour periods were then averaged to obtain the condensed results for each of the three coals studied.

Results of the 24-hour demonstration runs for the Pittsburgh No. 8 seam are shown in Figure 2, which gives the material and attribute balance around the major unit operations within the POC process. Overall results indicated 48.2 percent yield, 89.5 percent energy recovery, and 73.6 percent pyrite rejection. Results for the Upper Freeport seam and Illinois No. 6 seam are summarized in Figures 3 and 4, respectively. Performance evaluations for the Upper Freeport seam indicated an overall energy recovery of 87.5 percent, while 76.2 percent of the raw pyritic sulfur was removed. For the Illinois No. 6 seam, the POC recovered 85.8 percent of the energy and removed 79.4 percent of the pyritic sulfur in the raw coal.

Table 1 presents the contributions of each coal-cleaning circuit to total energy loss and pyritic-sulfur removal for each of the three coals. These values are based on the average results obtained during the 24-hour demonstration runs. The heavy-media cyclone circuit for each coal removed the bulk of the pyritic sulfur while losing relatively low amounts of energy. Depending on the coal, the heavy-media cyclone circuit removed 60 to 70 percent of the total amount of pyrite that was removed. Meanwhile, the energy content in the heavy-media cyclone circuit refuse was only 32 to 36 percent of the total energy losses.

Since large amounts of pyrite have been removed prior to the water-only cyclone and the advanced froth flotation circuit, the ratio of pyrite rejection to energy loss is significantly lower for these circuits. For the Upper Freeport Seam, for example, the advanced froth flotation circuit removed 25 percent of the total amount of pyrite that was rejected, and the energy content in the flotation tailings accounted for 71 percent of the total energy losses. Contributing to the lower ratios is the more-difficult-to-clean nature of fine- and ultrafine-particle streams.

Cumulative plant availability during 24-hour demonstration runs for the three coals was 94.9 percent. Individually, plant availabilities for the Pittsburgh No. 8, the Upper Freeport and the Illinois No. 6 coals were 98.0, 97.4, and 89.9 percent, respectively. No downtime encountered during the demonstration runs was attributed to the advanced process equipment.

20 TPH Semi-Works and 200 TPH Commercial Plant Economic Analysis

As part of the DOE project, a 20 TPH Semi-Works facility was designed as a starting point to determine the economics of the advanced flotation process. Generally, the Semi-Works process flowsheet is identical to the block flow diagram as previously shown in Figure 1. To construct this facility, approximately $10.9 million will be required. Another $750K will be needed for the purchase of land and working capital.

Based on the capital cost of the Semi-Works plant, the cost for 200 and 500 TPH facilities were extrapolated. In addition, fixed and variable, first-year operating-and-maintenance costs were estimated for these commercial plants. Table 2 presents a summary of these costs for a 200 TPH facility operating 7,560 hours per year.

In order to compare these values to those of existing facilities and to evaluate them on an investment basis, these values were input into an economic model developed by EoS Technologies, Inc. for the U.S. Department of Energy. Based on the above costs and a set of economic assumptions, the model generates the clean coal price required for the investment to generate a user-established, after-tax rate of return. Also, the cost to remove sulfur, on a dollars per ton of SO_2 equivalent, is determined. Table 3 presents these values for 200 and 500 TPH commercial facilities as well as the economic assumptions on which the estimates are based.

As the table indicates, plant size and operating schedule both significantly affect the costs of coal production and thus, the desulfurization costs. For the Pittsburgh No. 8 coal, for example, desulfurization costs for a 200 TPH (product) plant operating 7,560 hours per year are estimated at $327 per ton of SO_2 equivalent. For a 500 TPH plant with the same operating schedule, desulfurization costs drop to $275 per ton. On an average, desulfurization costs decrease by 15 percent for the 500 TPH plants.

Due to the large capital requirements for these advanced plants, operating schedule affects costs to a greater extent than plant size. A typical coal preparation facility operates five days per week, two shifts per day. Maintenance is conducted on the third shift. This operating schedule results in 3,456 hours of operation per year and, as the cost estimates reveal, a gross under utilization of facilities. For the Illinois No. 6 coal, a 200 TPH plant operating 7,560 hours per year can remove sulfur for about $305 per ton of SO_2 equivalent. When the same size plant operates only 3,456 hours per year, the desulfurization cost climbs to $435 per ton.

Conclusions

(1) The proposed process technology is ready for commercialization. However, the product from the process may require reconstitution in order to be marketable.
(2) The proposed technology can recover 85 percent of the energy in each of the raw coals while removing 75 to 80 percent of the pyritic sulfur.
(3) Clean coal ash values of 7.2 to 10.3 percent were achieved with in the POC.
(4) Grinding to levels finer than 85 percent passing 200 mesh will be required to remove 85 percent of the pyrite while maintaining 85 percent energy recovery.
(5) Based on raw coal costs of $0.80 per million Btu, clean coal prices of $1.50 per million Btu will be needed to earn a 10 percent after-tax rate of return.
(6) Desulfurization costs, on a dollars per ton of SO_2 equivalent, of $255 to $275 are estimated for a 500 TPH commercial facility incorporating the proposed process.

Table 1
Contributions to Energy Loss and Pyritic Sulfur Removal*
During 24-Hour Demonstration Runs

Circuit (Size Cleaned)	Pittsburgh No. 8 Coal		Upper Freeport Coal		Illinois No. 6 Coal	
	Energy Loss	Pyrite Removal	Energy Loss	Pyrite Removal	Energy Loss	Pyrite Removal
Heavy-Media Cyclone (+48 mesh)	3.8	44.3	2.7	49.4	4.5	55.4
Water-Only Cyclone (48mesh x 0)	4.0	16.6	0.9	7.9	2.2	10.4
Adv. Froth Flotation (200 mesh x 0)	2.7	12.7	8.9	18.9	7.5	13.6

* Values represent the percentages of the raw coal energy content and pyritic sulfur content found in the refuse stream of each circuit.

Table 2
Capital Requirements and 1st Year Operating and Maintenance Costs
for a 200 TPH Advanced Froth Flotation Plant (7560 Hours)

Construction Capital Requirement	$59,624,735	$59,547,893	$59,669,206
Working Capital Requirement	$467,723	$449,603	$478,500
Fixed Operating and Maintenance Costs	$5,892,451	$5,892,451	$5,892,451
Variable Operating and Maintenance Costs	$12,080,238	$11,158,134	$12,613,895

Table 3
Advanced Froth Flotation Economics (Annualized Basis)

200 TPH Commercial Plant Operating 7,560 Hours per Year			
Cost Item	Pittsburgh No. 8	Upper Freeport	Illinois No. 6
Clean Coal Price ($/MMBtu)	1.49	1.50	1.55
Clean Coal Price ($/Ton)	40.65	40.27	41.96
Annualized Desulfurization Cost*	$326.82	$305.42	$304.60
200 TPH Commercial Plant Operating 3,456 Hours per Year			
Clean Coal Price ($/MMBtu)	1.81	1.81	1.87
Clean Coal Price ($/Ton)	49.32	48.68	50.63
Annualized Desulfurization Cost*	$477.08	$442.49	$435.09
500 TPH Commercial Plant Operating 7,560 Hours per Year			
Clean Coal Price ($/MMBtu)	1.38	1.39	1.44
Clean Coal Price ($/Ton)	37.68	37.30	38.99
Annualized Desulfurization Cost*	$275.27	$256.95	$259.84

ECONOMIC ASSUMPTIONS

Plant Life	20 Years	Inflation Rate	4.0 %
Royalties	None	Corporate Tax Rate	38.0 %
Financing	100% Equity	Rate of Return	10.0 %

* Annual Desulfurization Costs are per ton of SO_2 equivalent.

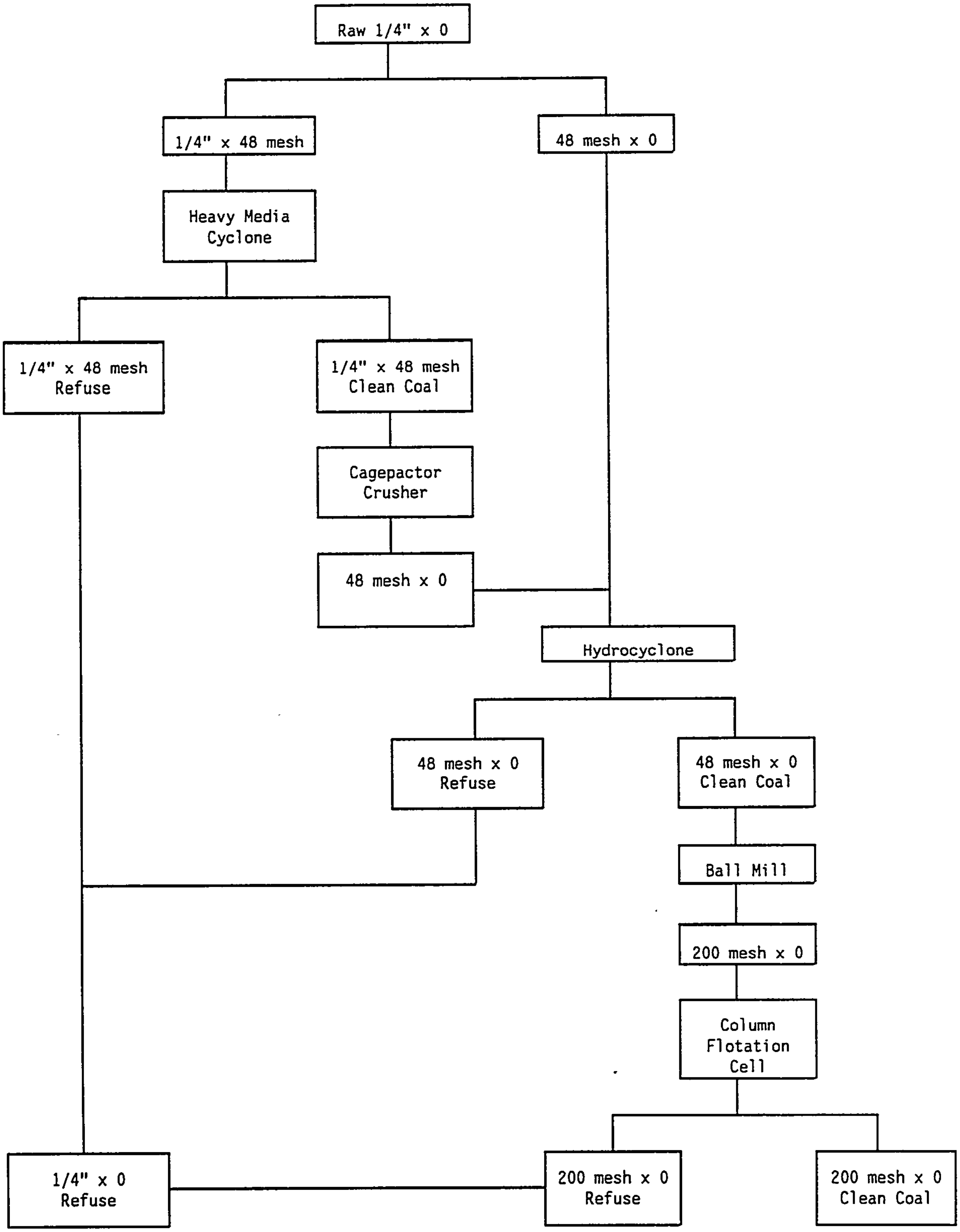

Figure 1. Simplified Block Diagram of the POC Process

Figure 2
Pittsburgh No. 8 Seam Coal
Average Performance
During 24-Hour Demonstration Run

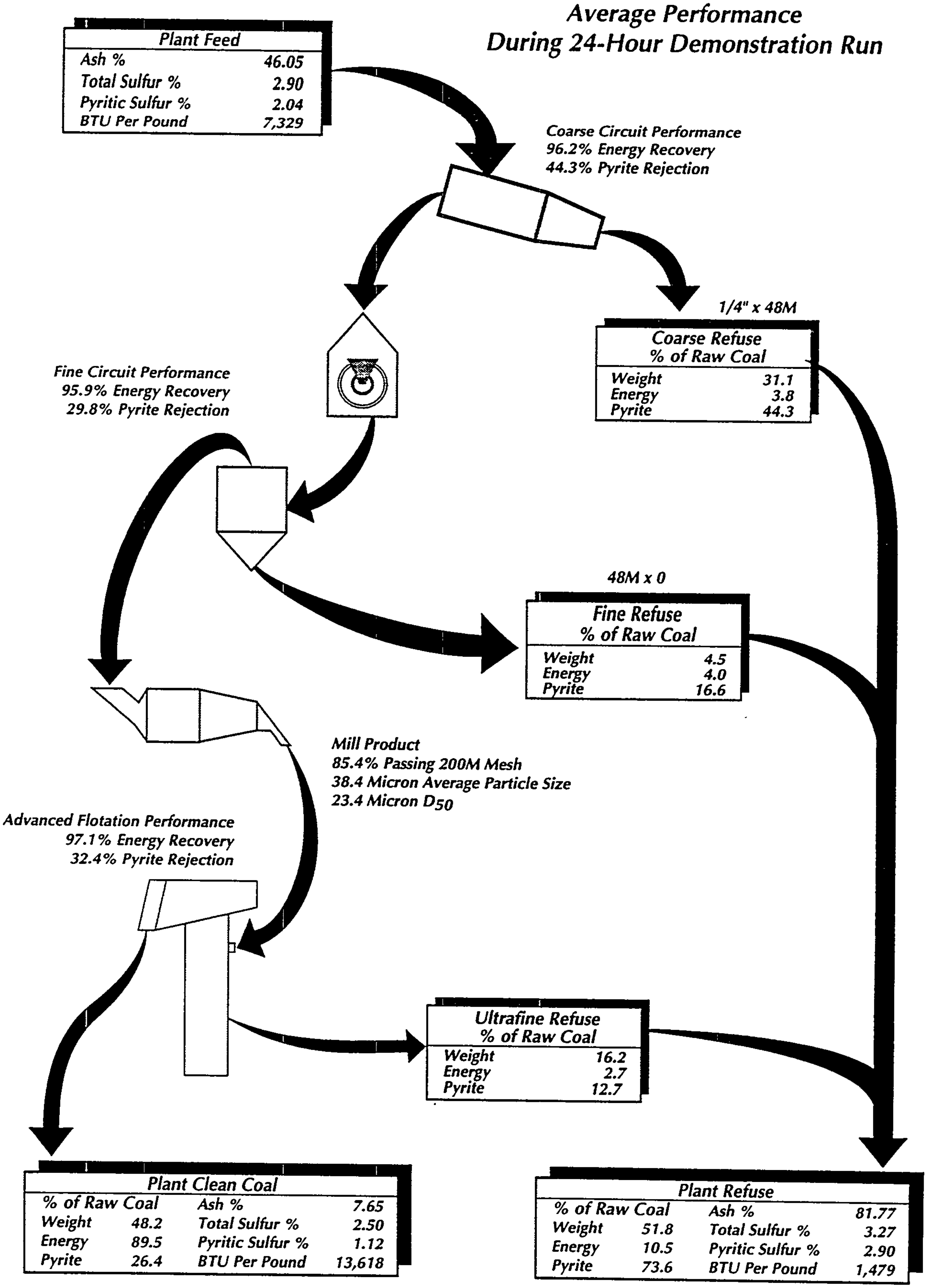

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Figure 3
Upper Freeport Seam Coal Average Performance During 24-Hour Demonstration Run

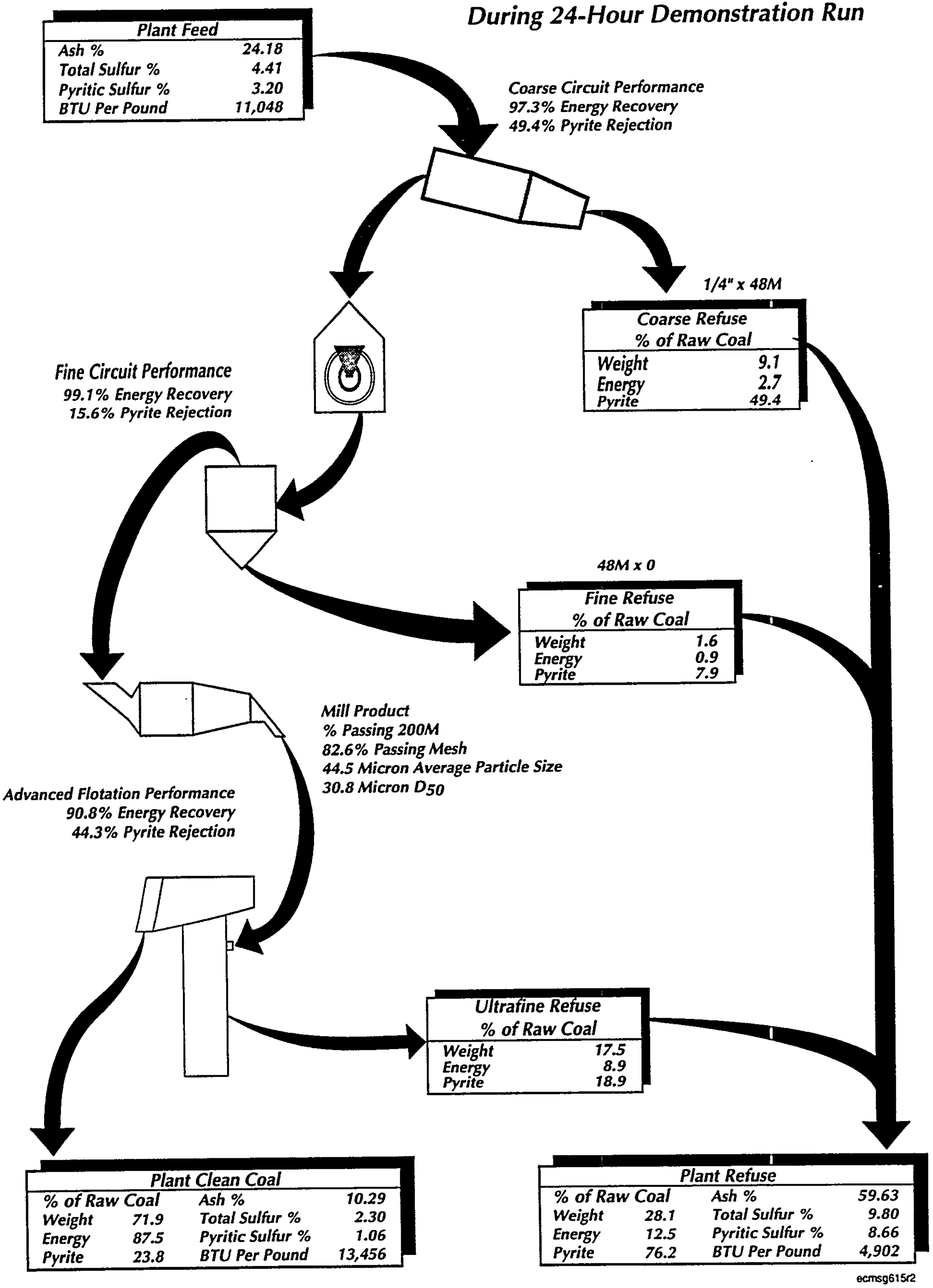
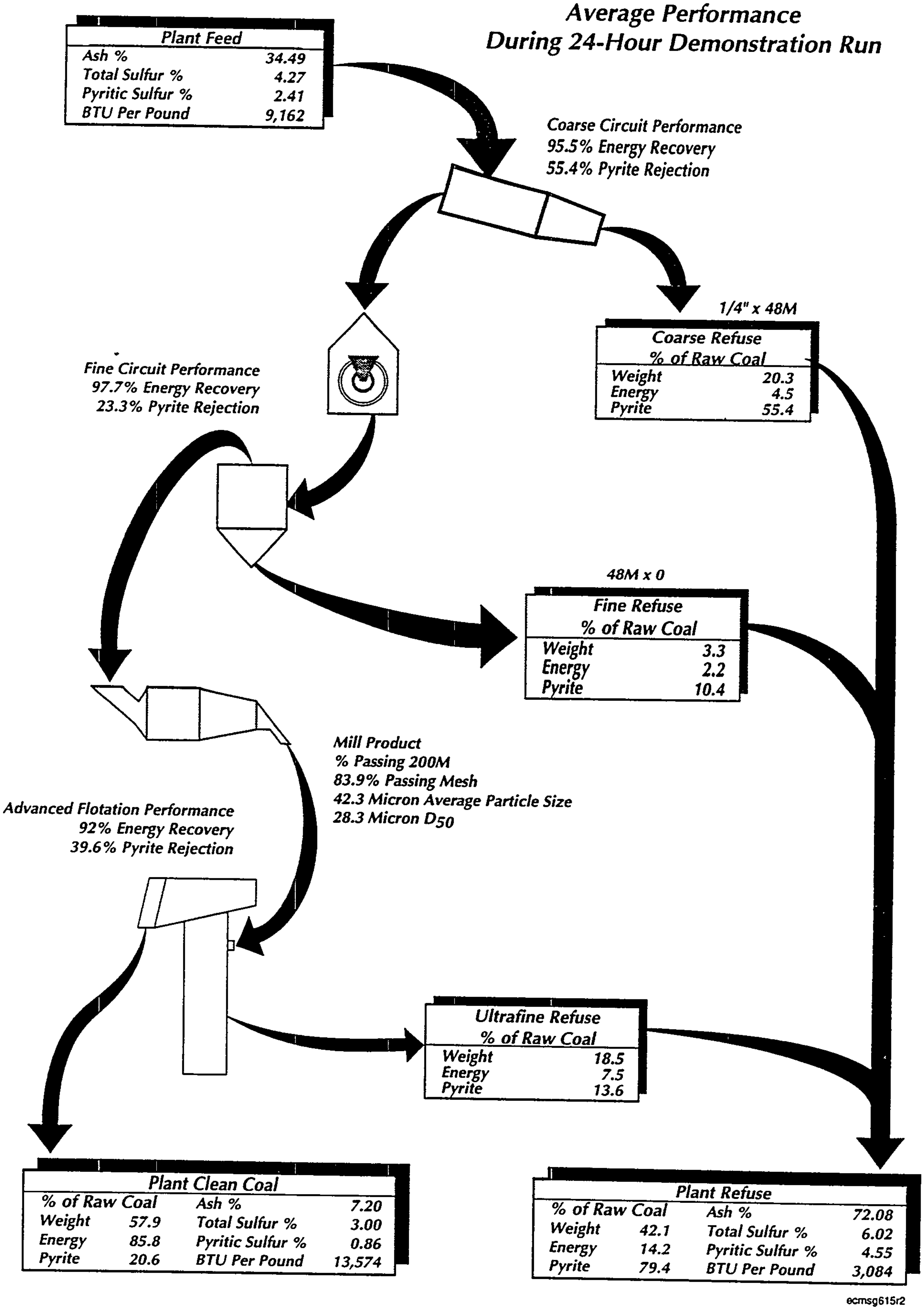


Figure 4
Illinois No. 6 Seam Coal
Average Performance
During 24-Hour Demonstration Run



ENGINEERING DEVELOPMENT OF ADVANCED PHYSICAL FINE COAL CLEANING FOR PREMIUM FUEL APPLICATIONS

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INTRODUCTION

The objective of this project is to develop the engineering design base for prototype fine coal cleaning plants based on Advanced Column Flotation and Selective Agglomeration processes for premium fuel and near-term applications. Removal of toxic trace elements is also being investigated. The scope of the project includes laboratory research and bench-scale testing of each process on six coals followed by design, construction, and operation of a 2 tons/hour process development unit (PDU). Three coals will be cleaned in tonnage quantity and provided to DOE and its contractors for combustion evaluation.

Amax R&D (now a subsidiary of Cyprus Amax Mineral Company) is the prime contractor. Entech Global is managing the project and performing most of the research and development work as an on-site subcontractor. Other participants in the project are Cyprus Amax Coal Company, Arcanum, Bechtel, TIC, University of Kentucky and Virginia Tech. Drs. Keller of Syracuse and Dooher of Adelphi University are consultants.

Additional details on the project can be found in two papers presented at the last two contractors conferences [1,. 2]. The selection of six feed coals (Taggart, Elkhorn No. 3, Winifrede, Indiana VII, Sunnyside and Dietz) is covered in a paper presented at the High Efficiency Coal Preparation Symposium [3]. The goal of this paper is to report on the progress made during the last twelve months in performing various tasks.

TASK 1. PROJECT PLAN REVISIONS

A revised Project Management Plan was prepared to reflect the organizational changes resulting from the Cyprus Amax merger. The project is now scheduled for completion by June 1997. The budget estimate remains the same. The Industrial Company of Steamboat Springs, Colorado, (TIC) was awarded a subcontract for the construction of the PDU. Dr. John Dooher was added as a consultant to assist in the coal water slurry fuel (CWF) formulation studies.

TASK 3. DEVELOPMENT OF NEAR-TERM APPLICATIONS

Cyprus Amax Coal Company, through its Cannelton division, has selected the Lady Dunn Coal Preparation Plant in West Virginia for evaluation of the advanced column flotation technology to process minus 100 mesh coal fines. The plant is undergoing expansion which will increase the tonnage of fines.

Figure 1 shows typical results obtained in a laboratory column. It appears that a clean coal with 8 to 10 percent ash can be produced at about 80 to 90 percent energy recovery. These results are significantly better than those obtained in the past by conventional flotation process.

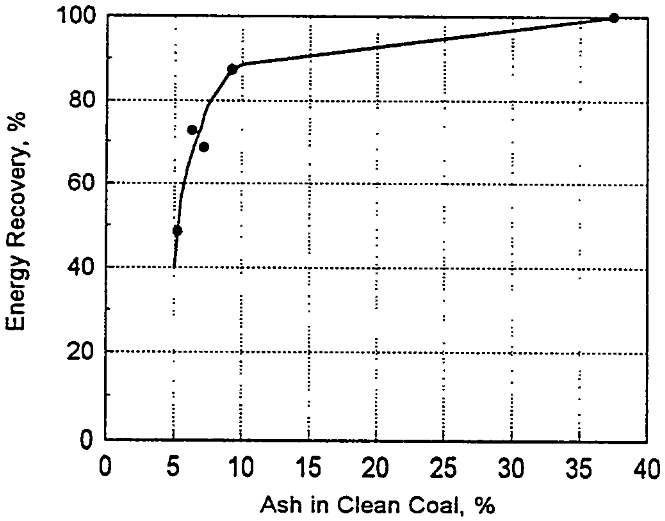

Figure 1. Laboratory Test Results on Lady Dunn Plant Coal Fines.

Based on the encouraging results and continued interest of the company in this technology, a 30-inch Microcel™ column is being installed at the Lady Dunn plant with help from Virginia Tech which will also assist in the operation of the column and evaluation of the results. This information will provide the design basis for the commercial plant - several columns of 10 or 12-ft diameter.

The clean coal slurry will be processed in a centrifuge or a filter press under a variety of conditions to determine the quality and quantity of product recovered and the anticipated cost for a commercial plant. Drying and briquetting of the clean coal fines will be investigated at vendor facilities to determine whether the product will be suitable for two niche markets - injection of dry coal fines in a blast furnace or use of briquettes as a stoker fuel.

In view of the importance of the dewatering of coal fines, a new subtask was added to the project for dewatering studies. Under Subtask 3.3, Virginia Tech will research and develop a hydrophobic dewatering process.

TASK 4. ADVANCED FLOTATION R & D FOR PREMIUM FUELS

This task consists of five subtasks which have all been completed. Results from Subtasks 4.1, Grinding, and 4.2, Process Optimization Research, were presented last year [2].

Subtask 4.3 covered coal water slurry fuel (CWF) formulation studies. Samples of clean coal produced in Subtasks 4.2 and 4.4 were used. It was found that the solids loading varied significantly based on the particle size of the coal and its inherent moisture content. Table 1 presents a summary of the results. The highest loading was attained with Taggart coal (about 70 percent). Elkhorn No. 3 and Sunnyside coals could be loaded to about 65 percent solids. The Indiana VII could be loaded to just above 50 percent solids. Attainment of a bimodal distribution by selective grinding of part of the cleaned coal was found to be the most important parameter. A small amount of A23 was used as dispersant. While the use of a stabilizer improved the stability of the slurries, it also increased the viscosity. A topical report presenting the detailed results has been submitted to DOE.

Table 1. CWF Slurry Comparison For Project Coals

	Taggart		Sunnyside	Elkhorn No. 3		Indiana VII	
	Regrind Blends	Selective Regrind	Regrind Blends	Regrind Blends	Coarsened Feedstock	Regrind Blends	Coarsened Feedstock
PSD MMD, microns	44	55	30	28	70	10	65
Coal Loading, wt%	64-68	68-70	63-65	61-64	64-66	47-51	50-53
Without Stabilizer							
Viscosity, cp	400-1400	800-1400	500-1200	500-1000	600-800	900-1600	600-1000
Stability Rating	1-3	1-3	1-3	1-3	1	1	1
With Stabilizer							
Viscosity, cp	800-2000	NA	NA	1600-3500	3500	NA	NA
Stability Rating	7-10	7-10	NA	7-10	3	NA	NA

Under Subtask 4.4, bench-scale (30-cm diameter) Ken-Flote™ and Microcel™ columns were installed and operated on the five bituminous coals. It was found that Taggart, Sunnyside, and Elkhorn No. 3 coals could be cleaned to meet the project specifications at design capacity (100 lb/hour or more). Indiana VII coal could be cleaned to the specifications but at a somewhat lower rate. Specifications could not be met with Winifrede coal. Better performance was obtained with the Microcel™ column. Based on the test results, greater confidence in scale up of the design, and commercial considerations, the Microcel™ column was selected for the PDU design. Samples of feed and product were analyzed to determine the removal of toxic trace elements. Samples were also provided to Combustion Engineering and Penn State for combustion evaluation. The results from this subtask are currently being compiled in the form of a topical report. Figure 2 shows a summary of results for Taggart, Sunnyside, and Indiana VII coals cleaned by the Microcel™ column under a variety of conditions.

The results from the above subtasks were used by Bechtel to prepare the conceptual design of the 2 tons/hour process development unit (PDU) and the advanced flotation module under Subtask 4.5. Taggart, Sunnyside, and Indiana VII coals were selected for the PDU design and operation. The plant will be able to process Sunnyside coal at design capacity, Taggart coal at a higher capacity, and Indiana VII coal at a lower capacity.

TASK 5. PDU AND FLOTATION MODULE DETAILED DESIGN

The detailed engineering design of the PDU and advanced flotation module was completed during the reporting period. The plant consists of four areas:

1. Area 100 Grinding
2. Area 200 Column Flotation
3. Area 300 Selective Agglomeration
4. Area 400 Dewatering

Detailed design of Area 300 will be performed under Task 7. Area 100 and 400 will serve both the column flotation and selective agglomeration modules as well as the utilities. A key feature of the design was the utilization of existing Amax R&D/DOE equipment and minimum alterations to the existing building structures.

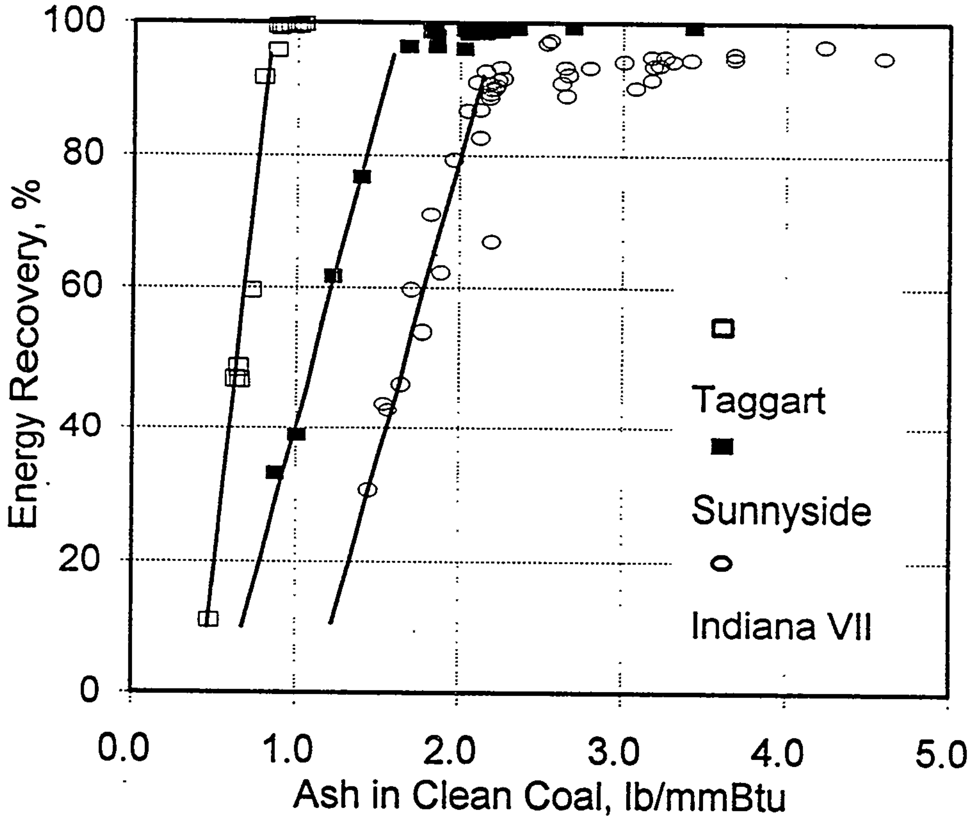

Figure 2. Performance of Taggart, Sunnyside, and Indiana VII coals in bench-scale column.

A 2-volume detailed engineering design package was prepared and submitted to DOE. The first volume contains Process Flow Diagrams (PFDs), Piping and Instrument Diagrams (P&IDs), Equipment Arrangement Drawings, Civil and Structural Design Drawings, Electrical Design Drawings, Equipment, Motor and Instrument List and Construction Specifications. The second volume contains Material Requisitions for various pieces of equipment and instruments.

Figure 3 shows a block flow diagram of the PDU and advanced flotation module. A closed grinding circuit will be used to assure efficient grinding of coals to required size. A Netzsch stirred ball mill will be used for fine grinding. Cyclones and Sizetec screens will be used for size classification. A 6-ft diameter Microcel™ column will be used for flotation. The concentrates will be dewatered by vacuum filtration and stored as filter cake. The tailings will be thickened, filtered, and disposed in a land fill. Normally, the PDU will be operated on a long day shift for parametric evaluation on each of the three coals. Once the optimum conditions have been established, 100-hour runs will be made to produce 200-ton lots of clean coal.

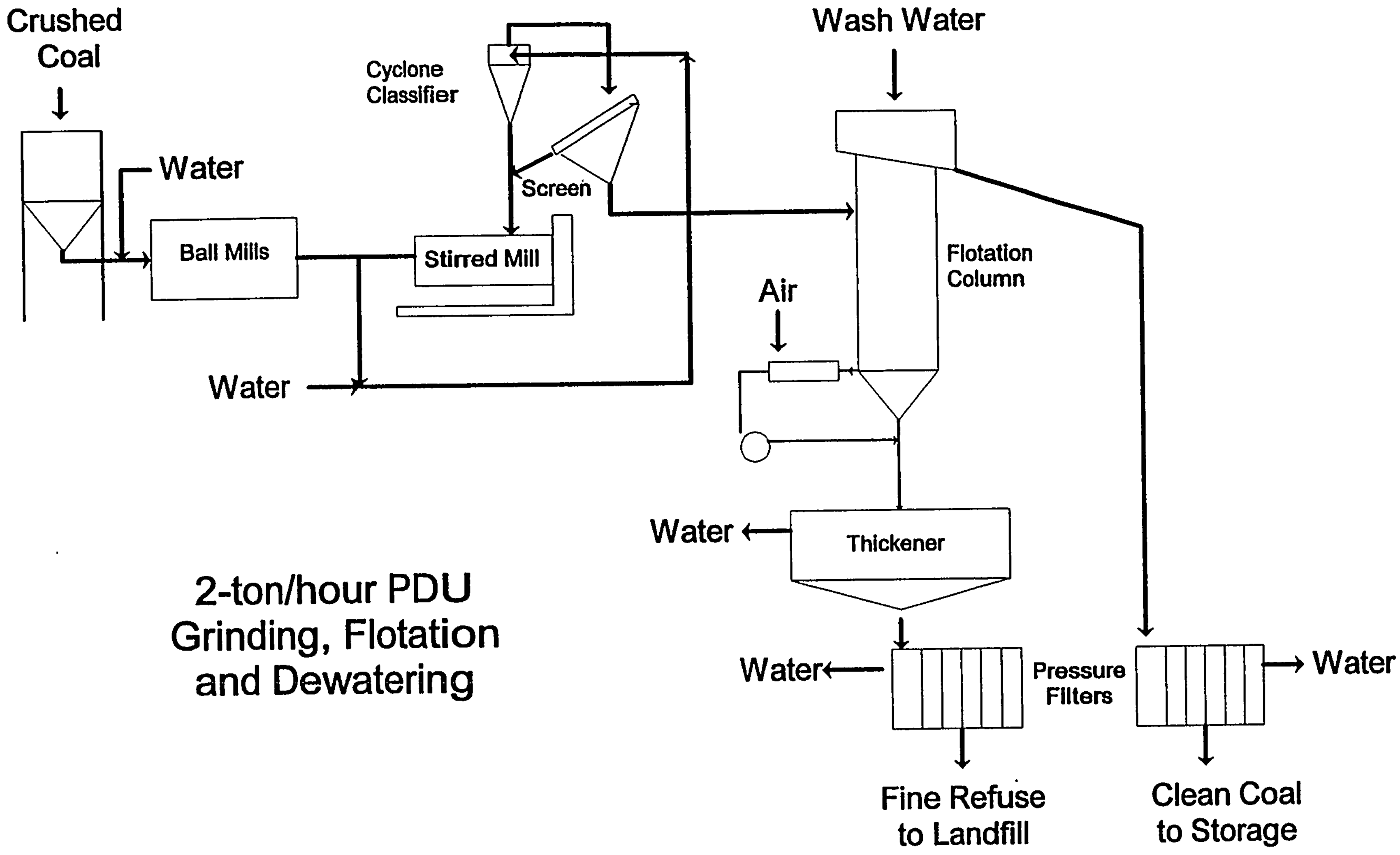

Figure 3. Block Flow Diagram for the PDU and Advanced Flotation Module.

TASK 6. SELECTIVE AGGLOMERATION R&D

This task is parallel to Task 4 for flotation. It is divided into six subtasks. Subtask 6.1, Agglomerating Agent Selection, and Subtask 6.2, Grinding, have been completed and the results have been presented in topical reports. They were covered in last year's presentation [2].

Subtask 6.3, Process Optimization Research, was completed during the reporting period and the topical report has been drafted. Two agglomerating agents (pentane and heptane) and two reactor design concepts (unitized reactor of a novel design and staged high and low shear reactors of a conventional design) were investigated in the test work performed at the Amax R&D Center and at Arcanum Corporation with Dr. Keller of Syracuse University providing guidance as a consultant.

Table 2 presents a summary of the results obtained with the unitized reactor using heptane as the agglomerating agent. It can be seen that ash specifications were met for all bituminous coals and the energy recoveries were significantly higher in comparison to the column flotation process. The low-rank coal could be cleaned, but it required acid pretreatment which will be costly. Similar results were obtained with pentane, but the feed slurry had to be cooled for steady operation. Arcanum evaluated the conventional two stage reactor design with heptane as the bridging liquid. Both ash specifications and energy recovery goals were met.

Table 2. Selective Agglomeration Process Optimization Results for Six Feed Coals

	Taggart	Indiana VII	Sunnyside	Elkhorn No. 3	Winifrede	Dietz
Clean Coal:						
Residual Ash, lb/mmBtu	0.99	1.62	1.80	1.83	1.75	1.9-2.5
Energy Recovery, %	99.8	98.6	99.4	99.4	94.2	93-98
Feed Slurry:						
Solids, %	10-13	10	7-10	7-13	7	7
Heptane/coal Ratio, g/g	0.21	0.28	0.22	0.25	0.35	0.5
Asphalt, lb/st	none	16	none	none	none	25-80
Acid	none	none	none	none	none	pH 3-4
High-Shear Mixing:						
Tip Speed, m/s	15.5	15.5	15.5	17.5	15.5	15.5
Retention, minutes	0.10	0.16	0.10	0.10	0.11	0.19
Low-Shear Mixing:						
Tip Speed, m/s	11.8	11.8	11.8	13.3	11.8	11.8
Retention, minutes	0.34	0.58	0.29	0.34	0.39	0.56
Estimated Mixing Energy:						
Unitized Reactor, kwhr/st	14-18	30	16-23	19-35	30	45

Subtask 6.4, CWF Formulation Studies, has been initiated. The test plan has been approved. For the most part, it will follow the approach used in Subtask 4.3. There will be greater emphasis on slurry stability and viscosity as they relate to atomization and combustion in commercial systems. Dr. John Dooher of Adelphi University has been retained as a consultant for this study.

Subtask 6.5, Bench-scale Testing and Process Scale-Up, is currently in progress. Based on the scale up considerations it was decided to use the conventional two stage design in the PDU and therefore in the 25 lb/hour bench-scale unit. Figure 4 shows a block flow diagram of the system. Besides the high and low-shear reactors and a vibrating screen for separation of clean coal agglomerates from ash laden tailings-slurry, the system includes a partially packed column for steam stripping of heptane, and a condenser for the recovery and recycling of heptane. Shake down testing has been completed and parametric testing is currently in progress.

A conceptual design of the selective agglomeration module was performed by Bechtel under Subtask 6.6. Based on the potential cost savings, the unitized reactor with pentane as the bridging liquid was selected for the preliminary design. However, a more thorough evaluation based on some of the measurements made in the laboratory reactor (Subtask 6.3 above), indicated that the cost savings may not be realized because of the expensive cooling (refrigeration) required for the pentane system. There was also concern for the scale up of the unitized reactor of the novel design. Thus, the detailed design to be performed under Task 7 will be based on the conventional two-stage system with heptane.

TASK 8. PDU CONSTRUCTION AND OPERATION

The Industrial Company (TIC) of Steamboat Springs, Colorado, has been selected as the construction subcontractor. The construction of the PDU started in March and is scheduled for completion by July with shakedown testing of equipment in August. Start up testing of the process will be performed in the September-October time frame followed by parametric testing on each of the three coals. It is planned to complete the testing of the advanced flotation module by August 1996.

ACKNOWLEDGMENTS

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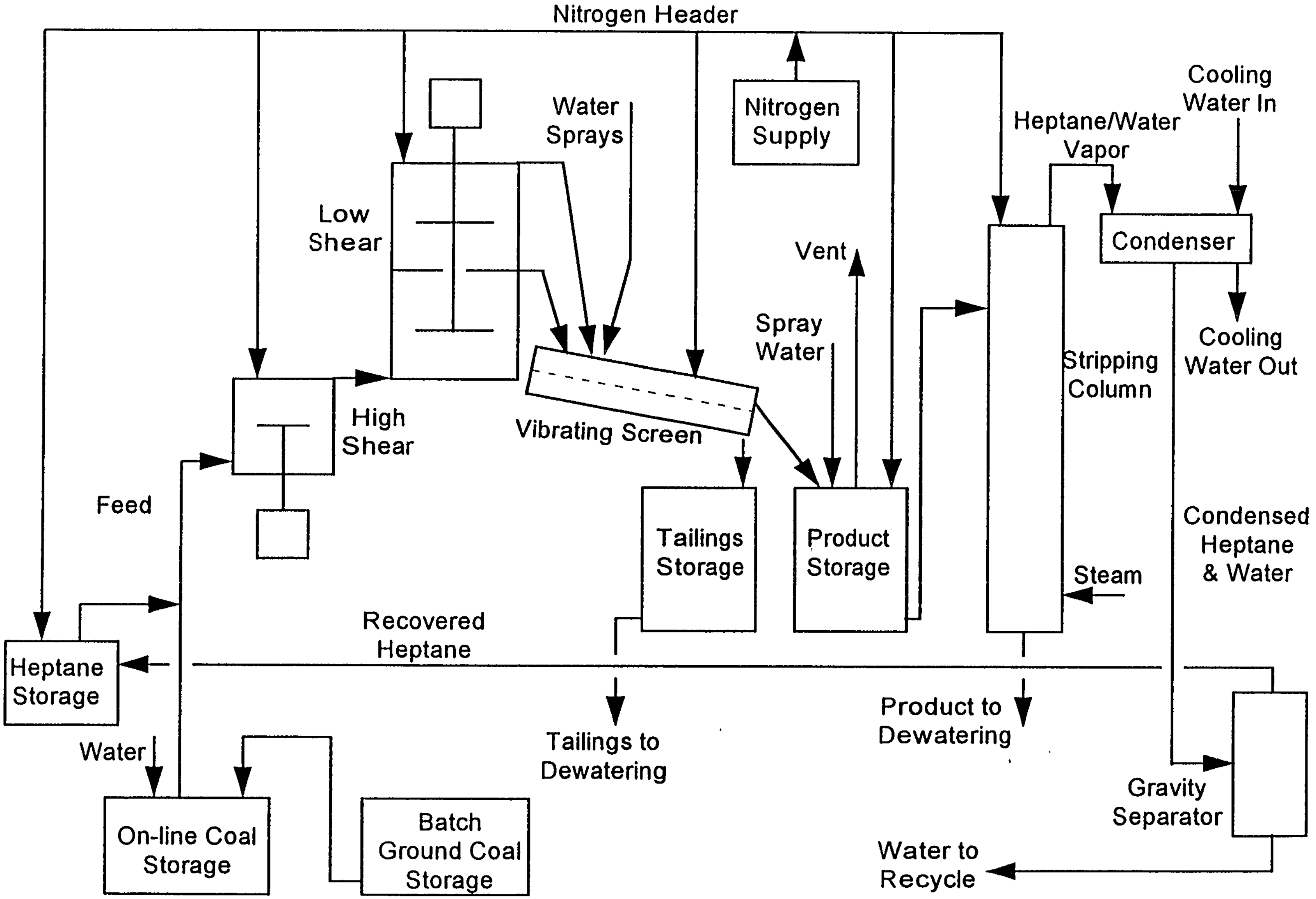

Figure 4. Schematic Block Flow Diagram of the Bench-scale Selective Agglomeration Unit.

CONTROLLING AIR TOXICS THROUGH ADVANCED COAL PREPARATION

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ABSTRACT

This project involves the assessment of advanced coal preparation methods for removing trace elements from coal to reduce the potential for air toxic emissions upon combustion. Scanning electron microscopy-based automated image analysis (SEM-AIA) and advanced washability analyses are being applied with state-of-the-art analytical procedures to predict the removal of elements of concern by advanced column flotation and to confirm the effectiveness of preparation on the quality and quantity of clean coal produced. Specific objectives are to maintain an acceptable recovery of combustible product, while improving the rejection of mineral-associated trace elements.

Current work has focused on determining conditions for controlling the column flotation system across its operating range and on selection and analysis of samples for determining trace element cleanability.

The column flotation system has traditionally yielded high recovery of coal, but with less than optimum rejection of ash and especially pyrite. Multiple factors were changed in order to gain greater mineral rejection while sacrificing a minimal amount of coal recovery. This was accomplished by raising the feed rate thus reducing the residence time, reducing the air addition rate thus reducing the air-to-coal ratio, and increasing the froth depth thus reducing entrained minerals. We were able to collect samples which well-defined the operating curve of this column for Pittsburgh No. 8 coal.

Samples of several column flotation runs, float-sink washability separations, and the head sample were analyzed for trace elements using state-of-the-art techniques. Those early results indicate that nearly all elements of concern show some affinity with mineral constituents. The elements arsenic, barium, cadmium, manganese, and lead show a strong affinity for the mineral constituents and can thus be greatly reduced through coal preparation. The other elements show more affinity for the organic components and so cannot be reduced as effectively; nevertheless, some reduction as a result of cleaning has been observed.

Seven pairs of froth and tails samples were selected from flotation runs along with three head samples representing a sampling of points over a wide range of ash and sulfur contents in the froth and tailings streams. Those samples have been submitted for trace element analysis to fill gaps along the operating curve and to verify the preliminary findings above. Those samples will also be examined by SEM-AIA for characterization of mineral components. Trace element and SEM-AIA mineral results will be combined to determine the association of trace elements with particular mineral phases. That, in turn, will be used with predictions of mineral removability to predict trace element cleanability.

PURPOSE

The purpose of this project is to determine the potential for reducing air toxics emissions from the conventional combustion of coal through the application of advanced coal preparation technologies. The major goal is to remove trace elements of concern such as arsenic, beryllium, mercury, lead, cadmium, chromium, manganese, cobalt, selenium, and zinc to the greatest extent possible while maintaining an acceptable recovery of heating value from the raw coal.

BACKGROUND

Increasing concern over potential environmental damage and risk to human health has led to efforts to minimize potential sources of air toxics. The sources of many air toxics resulting from the combustion of coal are trace elements that occur in coal at concentrations of less than one thousand parts per million (one-tenth of one percent). Because of the magnitude of coal use in this country, even low concentrations of toxic materials can lead to significant emissions when coal is burned and thus, may pose a threat to health and the environment. Potentially harmful trace elements may be associated with the mineral matter or organic (carbonaceous) portion of coal. Fortunately, most trace elements of environmental concern are associated with pyrite and accessory minerals in coal. For example, arsenic is commonly found in association with pyrite, as are cobalt, manganese, and selenium; lead is often found as galena or lead selenide, and zinc and cadmium are found with the mineral sphaelerite. The removal of minerals from coal through physical cleaning offers a simple and economical means for reducing air toxics resulting from the conventional combustion of coal.

APPROACH

The approach of this project is to predict the theoretical washability of trace elements in an economically significant coal, to perform advanced column flotation tests designed to remove trace elements from the froth product, and to compare the actual and predicted results for trace element partitioning.

PROGRESS

In early stages of this project, several items of fundamental significance were addressed. A quantity of Pittsburgh No. 8 coal was collected, divided, and stored under argon. A grinding protocol was established for repeatably producing a feed coal which approximates a typical power plant grind (i.e., 75% passing a 200-mesh sieve, herein referred to as "200-mesh"). The column flotation circuit was upgraded with several improvements made to the controlling algorithm to better handle fluctuation in conditions. Float-sink washability was determined for this coal by separating the feed coal in cesium chloride at densities of 1.3 g/cc and 1.6 g/cc. Samples of the feed coal were characterized by scanning electron microscopy-based automated image analysis (SEM-AIA) to determine the association of coal with mineral matter and to predict the cleanability of this sample of Pittsburgh No. 8 coal. The predicted cleanability of 200-mesh Pittsburgh No. 8 coal is shown in Figure 1 as determined from float-sink separation, as predicted by SEM-AIA, and as demonstrated by a column flotation run. The SEM-AIA predictions indicate that cleanability is greater for density-based (i.e., bulk) processes as compared to surface based processes. It indicates that better than 85% of the pyrite and other minerals should be removable while recovering more than 85% of the heating value. Both washability and column flotation data points fall below the cleanability curves indicated by

SEM-AIA. This is as expected since SEM-AIA tends to overestimate ideal cleanability by nature of its measurements while both actual separations are limited to less than ideal performance. The ideal cleanability curve would lie somewhere in the narrow band between these measurements.

The data points for column flotation plotted in Figure 1 are typical of many of our column flotation tests in that a high recovery of coal was achieved, but with less than optimum rejection of ash and especially pyrite. Therefore, multiple factors were changed in order to gain greater rejection while sacrificing a small amount of coal recovery. This was accomplished by raising the feed rate by a factor of two thus reducing the residence time, reducing the air addition rate thus reducing the air-to-coal ratio, and increasing the froth depth thus reducing entrained minerals. We were able to collect samples which well-defined the operating curve for this column for Pittsburgh No. 8 coal. That curve is shown in Figure 2 for a selection of runs to date. The conditions for these runs are shown in Table 1. Thus, the new runs have served to define the operating curve over a broad range. The most recent runs have been conducted in the region around the knee of the curve representing the greatest selectivity of separation.

Samples of several column flotation runs, density separations, and the head sample were analyzed for trace elements using state-of-the-art techniques. Samples selected for trace analysis are shown in Table 2. Data were checked for mass balance and generally good closure was obtained for all elements. Problems were noted for Cr and Ni results for two pairs of samples from flotation runs that had been performed with 5 µm coal according to earlier convention. The elevated levels of Cr and Ni in those samples was determined to be from the stainless steel grinding media and the extra grinding necessary to reduce the 200-mesh coal to 5 µm. Also, results for Sb and Hg showed levels of these elements in solution of less than the detection limit of 1 ppb which corresponds to about 0.1 ppm in the coal. Therefore, no conclusions can be reached regarding these elements for this coal.

A linear regression of trace element concentration versus ash content was performed to determine the association of trace elements with the coal and mineral components. Results are shown in Table 3 and in Figure 3 for the extreme cases of lead and beryllium. The concentrations of the elements in the organic fractions were determined assuming that an ideal, ash-free sample could be obtained. Good correlations of trace element concentration with ash content were generally found. This was mildly surprising in view of the fact that the composition of minerals which gave rise to the ash was not consistent across the range of ash contents. For example, pyrite generally accounted for large fraction of the ash in the low-ash float samples, while clays accounted for the bulk of the minerals in the high-ash samples. The correlations are summarized in Figure 4 showing the distribution of the elements between coal and mineral components in the feed sample. Since a large fraction of most elements are associated with the ash, substantial reductions should be achievable and were attained through column flotation.

The results were used to plot trace element rejection versus coal recovery for the actual data points in Figure 5. Similar curves can be prepared using the derived association of elements with the coal and ash. The elements arsenic, barium, cadmium, manganese, and lead show a strong affinity for the mineral constituents and can thus be greatly reduced through coal preparation. The elements beryllium, cobalt, and selenium show more affinity for the organic components and so cannot be reduced as effectively; nevertheless, some reduction as a result of cleaning has been observed.

Although the results shown above indicate a good correlation between most elements and ash content, there are several large gaps in the data. Therefore, additional samples have been selected for trace element analyses from recent column flotation runs. Seven pairs of froth and tails samples were selected from flotation runs along with three head samples representing a sampling of points over a wide range of ash and sulfur contents in both the froth and tailings streams. Those samples have been submitted for trace element analysis to verify the preliminary findings noted above.

Those samples will also be examined by SEM-AIA for characterization of mineral components. It is hoped that the composition of the mineral matter in these samples will vary from sample to sample so that it will be possible to determine the association of the trace elements with the various minerals. Assuming that will be the case, it should be possible to use the cleanability information from SEM-AIA for each of the minerals to predict the cleanability of the trace elements. Likewise, this information may help to focus the cleaning process for removal of the elements of most concern.

SUMMARY

Column flotation tests have been performed to define the grade-recovery curve for 200-mesh Pittsburgh No. 8 coal processed in our column flotation system. We found it necessary to increase feed rate (decrease residence time), to lower the air-to-coal ratio, and to increase the froth depth layer from our traditional settings in order to improve ash and pyrite rejection while sacrificing a minimal amount of coal recovery. Recovery of 80% of the heating value with rejection of nearly 80% the pyrite and mineral matter was attained for this sample of coal.

Trace element results were obtained for samples from float-sink washability and selected column flotation tests. Regression analyses of trace element concentration and ash contents of those samples showed a strong correlation between trace element concentration and ash content for many elements. Many of the trace elements showed a strong affinity for the ash-forming minerals. For others, there was only a slight dependence of elemental content on ash content. No cases were found where the trace element was associated only with the organic fraction. Therefore, significant reduction of the emission potential should be possible for most if not all elements.

Additional samples are currently undergoing trace element analysis to confirm the above findings. Samples are also being characterized by SEM-AIA in an effort to determine the association of trace elements with particular minerals.

ACKNOWLEDGEMENT

Ames Laboratory is operated for the U.S. Department of Energy by Iowa State University under Contract No. W-7405-ENG-82. This work was supported by the Assistant Secretary for Fossil Energy through the Pittsburgh Energy Technology Center.

Table 1. Conditions and results for selected column flotation tests for Pittsburgh No. 8 coal.

Conditions-feed rates (ml/min) Slurry/Reagent/Wash	MAF Rec. %	Froth T.S. %	Froth Ash %	Tails T.S. %	Tails Ash %	Mass Balance	Pyr Rej (Fd) %
400/40/400 bad MIBC	11.0	3.4	14.8	4.6	27.6	94	95
400/40/400	95.0	4.2	7.3	3.2	83.5	84	58
800/80/400 100% air	96.0	4.8	9.4	4.5	86.3	97	28
800/80/400 3/4 air	89.0	4.2	7.3	5.7	81.3	86	55
800/80/400 1/2 air	46.0	3.9	6.5	6.3	50.2	76	80
800/80/400 5/8 air	46.0	4.1	6.4	4.8	41.9	84	77
800/80/400 5/8 air	49.0	4.0	6.3	4.7	38.9	93	76
800/80/400 5/8 air Fuel oil 1 lb/ton	94.0	5.4	11.4	4.3	81.2	91	8
800/80/400 5/8 air Fuel oil .5 lb/ton	94.7	5.5	11.3	5.3	82.3	91	9
800/80/400 5/8 air Fuel oil .5 lb/ton (deep froth)	74.8	4.2	6.7	6.5	54.0	95	64
800/80/400 5/8 air Fuel oil .5 lb/ton (deep froth)	86.8	4.3	6.9	5.0	65.4	97	54

Table 2. Samples of Pittsburgh No. 8 coal selected for trace element analysis.

Sample	Wt. Frac.	Tot. S.	Ash%
Pitt #8 head sample	**100.00**	**4.60**	**27.30**
Pitt #8 1.30 float	**40.20**	**2.85**	**2.83**
Pitt #8 1.30-1.60	**31.10**	**3.05**	**12.23**
Pitt #8 1.60 sink	**28.60**	**7.67**	**75.64**
Pitt #8 04-14 F-1	**62.70**	**3.29**	**3.51**
Pitt #8 04-14 T-1	**37.30**	**5.55**	**70.49**
Pitt #8 05-03 F-2	**71.90**	**3.99**	**6.01**
Pitt #8 05-03 T-2	**28.10**	**5.53**	**90.83**
Pitt #8 06-23 F-4	**81.30**	**4.35**	**7.76**
Pitt #8 06-23 T-4	**18.70**	**4.07**	**85.05**

Table 3. Regression analysis of the association of trace elements with coal and ash-forming fractions of Pittsburgh No. 8 coal.

(conc. in ppm)	Ba	Mn	Zn	Cr	As	Pb	Ni	Co	Se	Be	Cd
Conc - org.	**1.18**	**1.44**	**9.35**	**7.80**	**1.71**	**1.37**	**1.85**	**2.11**	**1.22**	**1.02**	**0.08**
Conc - ash	**352.60**	**267.30**	**100.53**	**134.00**	**43.45**	**27.77**	**6.35**	**9.60**	**3.92**	**2.21**	**0.25**
R-squared	**0.99**	**0.99**	**0.98**	**0.48**	**0.95**	**0.98**	**0.47**	**0.81**	**0.71**	**0.96**	**0.27**
Feed - meas.	**92.50**	**76.17**	**30.80**	**30.30**	**18.00**	**8.60**	**3.74**	**2.81**	**2.50**	**1.34**	**0.40**
Feed - calc.	**97.12**	**74.02**	**34.24**	**42.25**	**13.11**	**8.58**	**3.08**	**4.15**	**1.96**	**1.34**	**0.13**
Org. contrib.	**0.86**	**1.05**	**6.80**	**5.67**	**1.24**	**1.00**	**1.34**	**1.53**	**0.89**	**0.74**	**0.06**
Ash contrib.	**96.26**	**72.97**	**27.44**	**36.58**	**11.86**	**7.58**	**1.73**	**2.62**	**1.07**	**0.60**	**0.07**
Org. fraction	**0.01**	**0.01**	**0.20**	**0.13**	**0.09**	**0.12**	**0.44**	**0.37**	**0.45**	**0.55**	**0.46**

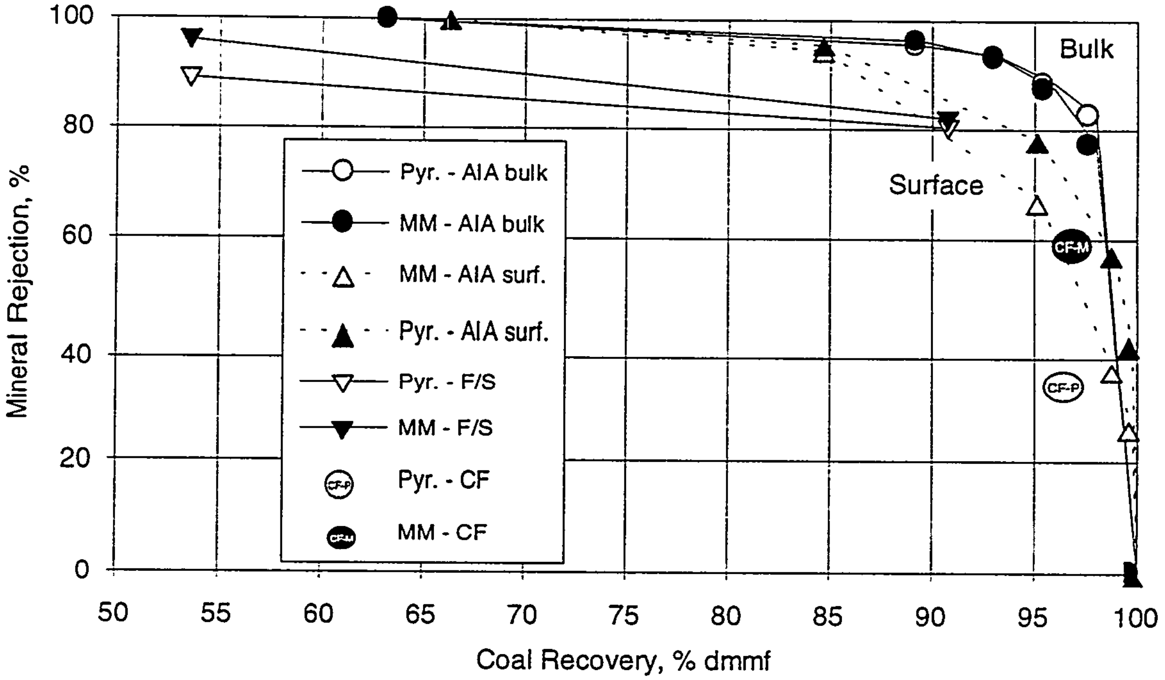

Figure 1. Rejection of minerals as a function of coal recovery for 200-mesh Pittsburgh No. 8 coal as predicted by SEM-AIA and as found by column flotation (CF) and centrifugal float sink (F/S).

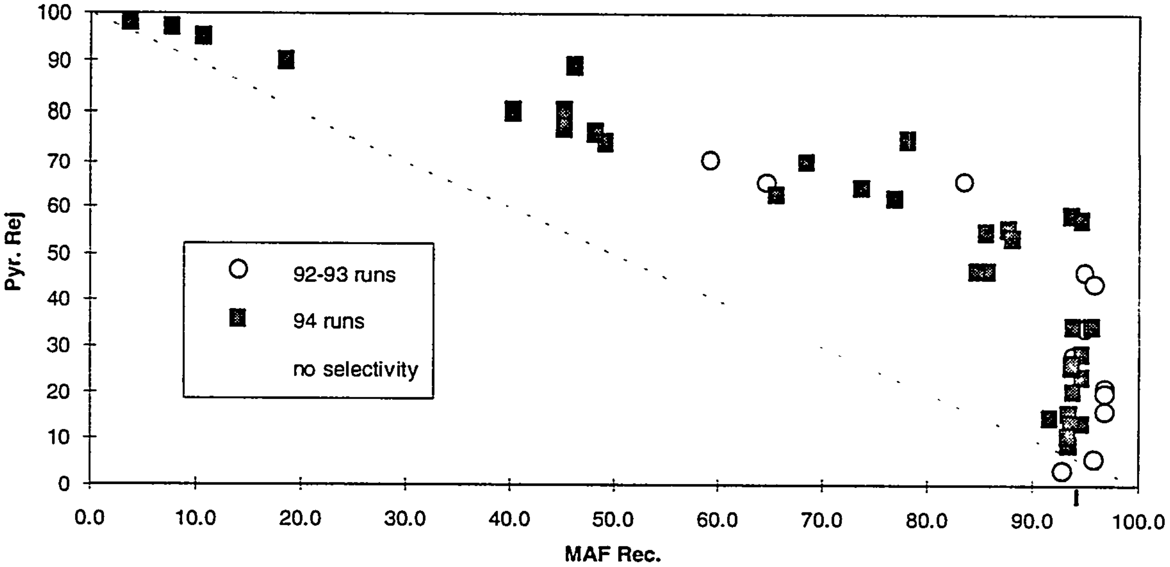

Figure 2. Pyrite rejection as a function of coal recovery for selected column flotation tests for 200-mesh Pittsburgh No. 8 coal.

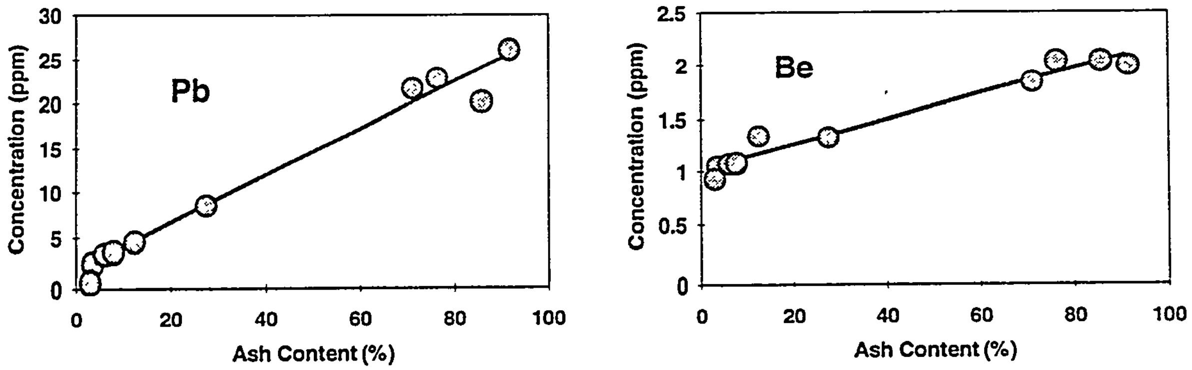

Figure 3. Concentration of Pb and Be as a function of ash content in samples of Pittsburgh No. 8 coal.

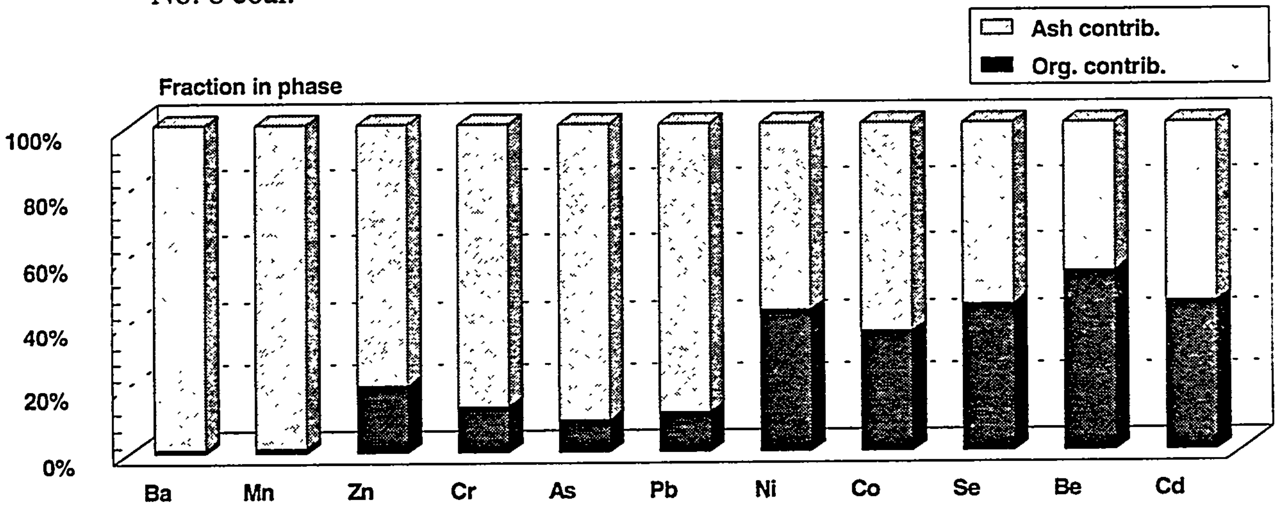

Figure 4. Distribution of trace elements between organic and ash-forming fractions in head sample of Pittsburgh No. 8 coal (27% ash content).

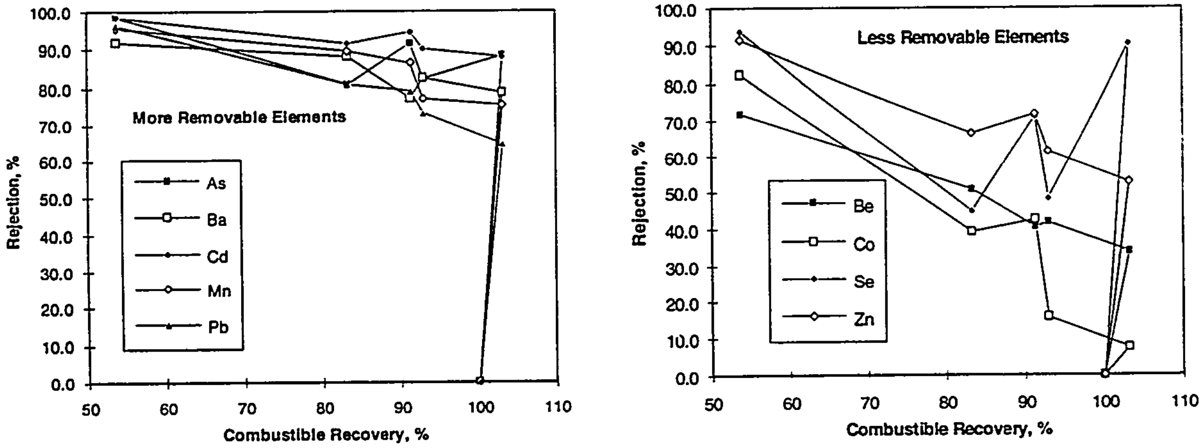

Figure 5. Rejection of trace elements as a function of recovery of 200-mesh Pittsburgh No. 8 coal using both column flotation and centrifugal float-sink data.

APPALACHIAN CLEAN COAL TECHNOLOGY CONSORTIUM

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INTRODUCTION

General

The Appalachian Clean Coal Technology Consortium (ACCTC) has been established to help U.S. coal producers, particularly those in the Appalachian region, increase the production of lower-sulfur coal. The cooperative research conducted as part of the consortium activities will help utilities meet the emissions standards established by the 1990 Clean Air Act Amendments, enhance the competitiveness of U.S. coals in the world market, create jobs in economically-depressed coal producing regions, and reduce U.S. dependence on foreign energy supplies.

The research activities will be conducted in cooperation with coal companies, equipment manufacturers, and A&E firms working in the Appalachian coal fields. This approach is consistent with President Clinton's initiative in establishing Regional Technology Alliances to meet regional needs through technology development in cooperation with industry. The consortium activities are complementary to the High-Efficiency Preparation program of the Pittsburgh Energy Technology Center, but are broader in scope as they are inclusive of technology developments for both near-term and long-term applications, technology transfer, and training a highly-skilled work force.

The consortium has three charter members, including Virginia Polytechnic Institute and State University, West Virginia University, and the University of Kentucky. Three other universities, namely, Pennsylvania State University, Ohio University, and Southern Illinois University, have been invited to join the consortium. The consortium also includes eight affiliate members[2] composed of coal companies, A&E firms, and equipment manufacturers.

Organization

The consortium is governed by a Council, which consists of representatives of the charter members, the Chairman of the Advisory Board, and the Director. The Advisory Board is made up

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[1] Current Director of the Appalachian Clean Coal Technology Consortium

[2] AMVEST Minerals; Arch Minerals Corp.; A.T. Massey Coal Co.; Carpco, Inc.; CONSOL Inc.; Cyprus Amax Coal Co.; Pittston Coal Management Co.; and Roberts & Schaefer Co.

of representatives from participating industrial affiliate members. The board identifies the areas of research priorities and the member universities submit proposals to the Advisory Board. The results of the Board's review are submitted to the Council, which makes the final selection of the proposals.

First-Year Objectives

Because of the limitations in the funding available during the first year, the R&D activities have been limited to only two areas of research: fine coal dewatering and modeling of spirals. Participating industrial companies identified fine coal dewatering as the most needed area of technology development. They also suggested that there is a need for developing new spirals that can have lower specific gravity cuts. The dewatering studies are to be conducted by the University of Kentucky's Center for Applied Energy Research (CAER) and Virginia Tech's Center for Coal and Minerals Processing (CCMP). A spiral model is to be developed by West Virginia University.

DESCRIPTION OF INDIVIDUAL PROJECTS

At the time of preparing this communication, the Advisory Board has not competed the review process. Therefore, the project descriptions given below are based on the proposals submitted rather than approved by the Council. It should also be noted that the proprietary information contained in the proposals are not presented in this communication.

Virginia Tech - Innovative Approaches to Fine Coal Dewatering

Background

The coal industry has been discarding coal fines due to difficulties in cleaning and moisture removal. Recent developments in advanced column flotation technologies have largely solved the first problem; however, there are currently no practicable solutions to the problems associated with fine coal dewatering. The mechanical dewatering methods commonly used today are not capable of reducing the moisture to levels acceptable to electrical utilities and other coal users. Thermal drying can reduce the moisture to acceptable levels; however, conventional thermal dryers are capital-intensive and costly to operate. Therefore, there is an impending need for innovative approaches to solving problems in dewatering fine coal.

For this reason, a novel dewatering process has been developed at CCMP. In this process, a wet coal is contacted with liquid butane, which displaces water from the surface of coal. The spent butane is recovered and recycled. This process is capable of reducing the moisture to levels comparable to thermal drying. Since the process of displacing water with butane is spontaneous, no energy is consumed during this step. The majority of the energy consumption occurs during the recycling stage.

Objectives

The objectives of this work are i) to test the novel dewatering process developed at CCMP on a variety on coals from the Appalachian coal fields, and ii) to determine its economic feasibility.

Scope of Work

Batch dewatering tests will be conducted on fine coal products from different coal preparation plants in the Appalachian coal fields. The tests will be conducted to study the effects of various process variables on the efficiency of the process. After establishing the optimum operating conditions with one coal, other coal samples will be tested. The test results will be used for economic analysis.

University of Kentucky - Improving Dewatering of Fine Clean Coal Using High-Pressure Filters

Background

Pressure filtration of fine coal is capable of producing a product containing as little as 20% moisture which is lower than is typically possible with other mechanical dewatering processes. Unfortunately, the operating costs for pressure filters are higher than for other conventional dewatering devices, i.e., vacuum filters and screen-bowl centrifuges. The major factor contributing to the high operating cost of high-pressure filters is air consumption. High air consumption usually occurs when the filter cake develops cracks through which air escapes. Once cracks develop, the effectiveness of the pressure or vacuum filter diminishes significantly.

In preliminary work conducted at the CAER, paper pulp was added to a coal slurry prior to filtration. During filtration, it was observed that no cracks developed in the filter cake and that the final wet cake moisture was lowered by four percent over the same coal slurry filtered without the addition of paper pulp. The wet filter cake also exhibited exceptionally high compressive strength. This suggests that the addition of inexpensive fibrous material may help minimize the consumption of air, thus making high-pressure filtration a more viable process for fine coal dewatering.

Objectives

The main objectives of this project are to i) identify the factors responsible for the formation of cracks within the filter cake during vacuum and high-pressure filtration, and ii) evaluate the addition of various types of fibers to the coal slurry to reduce or eliminate the formation of cracks. A second objective is to harden the filter cake in situ to improve the handling characteristics of the dewatered coal.

Scope of Work

Dewatering studies will be performed on froth flotation products from two coal preparation plants in the Appalachian region. Initially, dynamic filtration tests will be run to determine the effects of various operating parameters and reagent addition on the dewatering characteristics of the froth products. Variables to be studied include reagent quantity, pH, percent solids in suspension, particle size distribution, filtration kinetics, and final wet cake moisture content.

Fiber addition studies will be carried out using pulps produced from newspapers, computer/copier waste paper, carpet waste, and wood powder. Aqueous suspensions of each material will be prepared and added to the coal slurry. The slurry will then be filtered using both vacuum and high-pressure filters. Variables to be investigated include density of the pulp suspensions, percent of fibers added, and final wet cake moisture. All filter cakes will be checked for cracking during filtration. If cracks are observed, the cake will be dissected and examined via microscopy to determine the origin of the cracks. Selected filter cakes will also be examined using SEM and petrography to gain insights as to the effect of fiber arrangement on dewatering efficiency.

The wet and dried filter cakes will also be evaluated for their handling characteristics using four criteria: compressive strength, resistivity during drop tests, abrasion index, and dust reduction efficiency.

West Virginia University - Modeling of Spirals

Background

The most promising approach to improving spiral separation efficiency is through extensive computer modeling of fluid and solids flow in the various operating regions of the spiral. Previous efforts at accurate modeling have failed, primarily due to the use of incorrect physical models describing the flowing slurry stream.

As operated, the modern spiral has two functioning regions. These are the Grandy region, where the water flows, and a stable dense media section where the physical separation takes place. The Knoll line separates the Grandy from the dense media section. The Grandy is further divided into upper and lower regions. The dense media section is also divided into two regions. The radially outboard region is the low density region while the radially inboard region is the high density region. Energy and fluid in the Grandy forms and powers the dense media section of the spiral.

The addition of makeup water to the spiral greatly affects the size, location and behavior of each flow region. A detailed physical model has been developed that delineates the complex behavior of water and solids within the Grandy and dense media regions.

Several key issues must be resolved to improve the performance of spirals. Since movement of particles into the separation zone of the spiral is the key to improving efficiency, the most important issue is control of fluid and particle flow. Critical questions to be answered include determining the optimum location for makeup water addition, how to control the position of the Roberts and Knoll lines, and how to control the movement of particles from the upper Grandy into the lower Grandy. In addition, the effects of spiral diameter, pitch and height on fluid/particle behavior also need to be determined.

Objectives

The objective of this project is to use computer modeling to develop better, more efficient spiral designs for coal cleaning. The fully-developed model will predict spiral performance based on variations in spiral profile, flow rate, and pitch. Specific goals are to: i) design spirals capable of making separations at a specific gravity of 1.5, and ii) broaden the size range at which spirals make effective separations.

Scope of Work

Three models will be used in this work: i) a physical model that qualitatively describes the physics of the spiral, ii) a mathematical model that quantifies the physical model, and iii) a numerical model that permits the calculation of the mathematical model. All three models will be studied simultaneously; however, the primary focus of the first year's effort will be to fully develop the numerical model. Sufficient experimental data are available from the literature to provide a baseline against which the computer model can be checked.

In the initial stage of this project, a flow field computer program will be used to simulate pure water flow down a smooth, infinitely wide inclined plane. The results will be closely checked with experimental results reported in the literature. As the initial model is validated, the program will then be used to simulate more complex topographies and channels. Again, the simulator results will be compared with those from the literature.

Following the studies with pure water flow, two approaches will be followed. The first will be to numerically simulate curvilinear flow and the second will be to simulate slurry flow on linear surfaces. Once these models are working properly, they will be combined. The combined model will simulate flow in the upper and lower Grandy regions. Experimental data used to validate the combined numerical model will be provided by Carpco, Inc.

ACKNOWLEDGMENT

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The following manuscript was unavailable at time of publication.

PRODUCTION OF COMPLIANCE COAL FROM ILLINOIS BASIN COALS

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Please contact author(s) for a copy of this paper.

The following manuscript was unavailable at time of publication.

EPRI UPGRADED-COAL INTEREST GROUP

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The following manuscript was unavailable at time of publication.

OXIDATIVE STABILIZATION OF A DRIED LOW-RANK COAL

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THE KINETICS OF FOSSIL RESIN EXTRACTION FROM A FLOTATION CONCENTRATE

DE-AC22-93PC92251

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ABSTRACT

The kinetics of fossil resin extraction from a flotation concentrate by heptane were investigated as a function of process variables using monosize particles. Experimental results provide for a better understanding of the refining process and the basis for subsequent design and construction of a continuous resin refining circuit. Based on the effect of process variables (particle size, stirring speed, and temperature) the resin extraction rate appears to be controlled by surface solvation phenomena. The initial extraction rate was found to be inversely proportional to the initial particle size and a kinetic model is being developed to describe the experimental results.

INTRODUCTION

Certain bituminous coals of the western U.S. are known to contain appreciable quantities of macroscopic fossil resin. Such resinous coals are found in the states of Arizona, Colorado, New Mexico, Utah, Washington, and Wyoming, etc. The Wasatch Plateau coal field in Utah has a particularly high content of fossil resin. It has been reported that many seams in this field average as much as 5% resin.[1-2] Macroscopic fossil resins are friable and tend to concentrate into the fine sizes during coal preparation. Therefore it is not unusual to find that fine coal streams in such coal preparation plants contain more than 10% recoverable macroscopic resin, even when the run-of-mine coal contains only 3% resin.[5]

Fossil resins have been recovered intermittently from the Wasatch Plateau coal field in Utah since 1929 by gravity and/or flotation processes. The production, however, has been on a very small scale and the technologies used have limited the development of a viable fossil resin industry. Resin flotation concentrates can be refined by solvent extraction and evaporation of the solvent. Solvent-purified resins have a market value of at least $1.00/kg as a chemical commodity and can be used in the ink, adhesive, rubber, varnish, enamel, paint and coatings, and thermoplastics industries.[3-6] Unfortunately, process technology for the recovery and utilization of fossil resins from coal has not received sufficient attention. Because of the lack of technology and the competition from synthetic resins, the valuable fossil resin resource from western coal 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 as fuel has a value of $100 million as a chemical commodity - equivalent to the value of the coal itself! In this regard, significant efforts have been made to develop technology for a fossil resin industry in the western coal fields. As a result, several new flotation technologies for the recovery of fossil resin from coals have been developed.[5-8] These flotation technologies provide a highly efficient means to selectively recover fossil resin from coal (more than 80% recovery at about 80% concentrate grade). However, little attention has been given to the refining of the fossil resin flotation concentrate although solvent refining is a critical step in order to increase the market value of the fossil resin. The objective of this DOE sponsored research project was to study the details of the refining technology so that the process for fossil resin recovery can be optimized and the quality of refined fossil resin products can be controlled to meet market specifications.

In this paper, the resin extraction kinetics from a Wasatch Plateau fossil resin concentrate are discussed based on the physical/chemical properties of the resin. The effects of important extraction variables are examined and a kinetic model for batch resin extraction with heptane is under development.

CHARACTERIZATION OF RESIN CONCENTRATE

Fossil Resin Flotation Concentrate

The fossil resin flotation concentrate used in this study was obtained from Wasatch Plateau coal of south central Utah and generated by pilot-plant flotation tests under a previous DOE-funded program "Selective Flotation of Fossil Resin from Wasatch Plateau Coal." The fossil resin flotation concentrate has a heptane soluble resin content of approximately 75%. It was naturally dried, sampled and stored in plastic bags for future research use. The ash and moisture contents of the resin concentrate were found to be 1.23% and 1.03% respectively. 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 400 mesh (37 microns).

Heterogeneity of the Fossil Resin

Since fossil resin fluoresces under blue light, fluorescence microscopy can be used to examine its macroscopic composition. Under blue light, fossil resin grains can be distinguished and sorted into four resin types: yellow, amber, light-brown and dark-brown resin.[9] 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. It was found from this petrographic analysis that the resin concentrate consists predominately of amber (29%) and light-brown resins (35%) with some yellow resin (12%) and dark-brown resin (11%). A significant amount of coal fines (13%) were also found in the resin flotation concentrate. It is clear that the fossil resin concentrate is a heterogeneous material and preliminary results indicate that different extraction rates are associated with the four different resin types.[10]

Even at the molecular level fossil resin is known to be a complicated organic polymer. Fossil resin from the Wasatch Plateau coal field was classified by Anderson[11] et al. to belong to the class II resinites, which are believed to be derived from diterpenoic acid rich precursors. Based on the results of Py-GC/MS analysis, the bulk component of the Utah resin appears to be a polymer consisting of sesquiterpenoid ($C_{15}H_{24}$) repeat units. It has been reported that approximately 10% of the resin consists of a mixture of cyclic terpenoids.[12] In view of the foregoing, it is evident that the composition and structure of the Utah resin are not uniform. However, based on present results an approximate description can be made. The average elemental composition of heptane extracted resin is as follows:

C	H	N	S	O
86.88%	10.85%	0.17%	0.30%	1.80%

Only small amounts (2.3%) of heteroatoms (N, S and O) are found in the heptane extracted resin, which implies that the polar functional groups are rare. By adsorption chromatography with silica gel as the static phase and heptane as the elution solvent, a distinct component (7.7%) was separated from the heptane extracted resin. The IR spectra of this component and the remaining more-polar fraction are shown in Figures 1a and 1b, respectively. Figure 1a has typical alkane features. For the more-polar fraction in Figure 1b, the peaks of the CH stretching vibrations (2850 to 2960 cm^{-1}) and CH bending vibrations (1462 and 1382 cm^{-1}) obviously predominate. However, there is a signal which shows significant carbonyl (C=O bond) stretching vibrations at 1711 cm^{-1} for the more-polar fraction. In conclusion, the fossil resin from the Wasatch coal field is heterogenous and nonuniform in its chemical composition. It mainly consists of hydrocarbons with small amounts of functional groups containing heteroatoms.

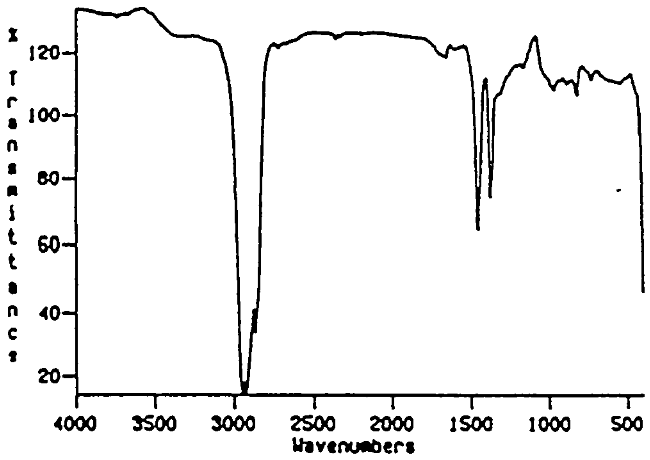

Figure 1a. IR spectrum of the component separated by elution chromatography from the heptane extracted resin.

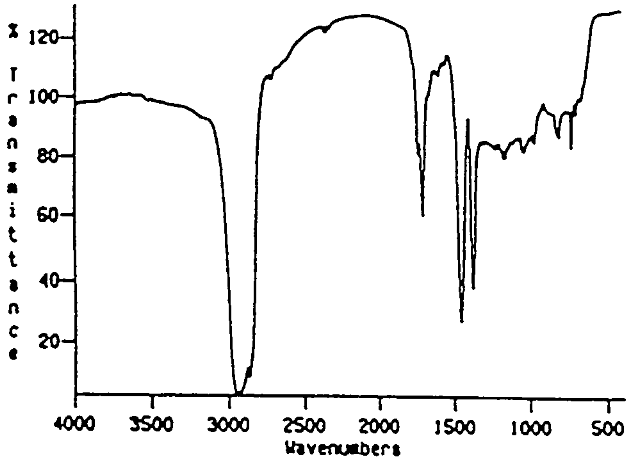

Figure 1b. IR spectrum of the remaining more-polar fraction separated by elution chromatography from the heptane extracted resin.

EXPERIMENTAL

A series of experiments for resin extraction by heptane were designed to study the effect of process variables on the rate of the resin extraction from monosize particles at a low solids concentration. Process variables studied included temperature, particle size and agitation intensity. The resin extraction kinetics were determined by analysis of the resin in heptane solutions taken at certain time intervals during extraction process.

Technical grade normal heptane (C_7H_{16}) was used as the solvent for the kinetics study. Heptane has a molecule weight of 100.21, density of 0.684 gram/cm^3 and boiling point of 98 ^0C. Four monosize samples (8×10, 28×35, 48×60, and 100×150 mesh) were prepared by wet screening from the flotation concentrate. The heptane-soluble resin content in all samples was determined to be between 88% to 95% with a TX-6 Soxhlet extraction unit at the boiling point of heptane (98 ^0C) for 6 hours.

The resin concentration in heptane was determined by a UV/Vis spectrophotometer (Beckman DU-7) at room temperature. It was found that a good linear relationship between UV absorbance at 300 nm and resin concentration in the heptane solution exists as long as the resin

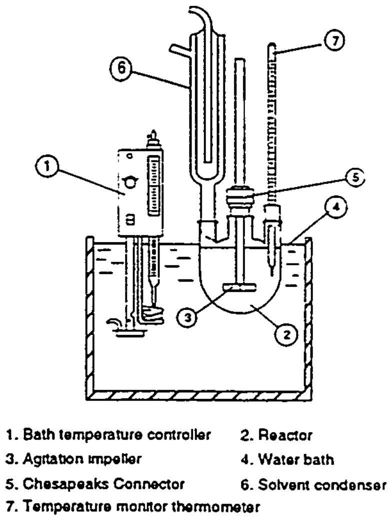

Figure 2. Schematic drawing of resin extraction apparatus.

concentration is below 500 ppm.

A schematic drawing of the experimental apparatus used for the kinetics study is presented in Figure 2. The reactor is a one liter round bottom 3-neck distillation flask. One liter of heptane was taken into the flask and heated to the desired temperature at which time 1.5 grams of resin sample was added to initiate the reaction. During the extraction process, about 3 ml of resin solution was taken from the suspension by a syringe with a prefilter at each desired time interval. 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 the heptane-soluble resin content in the samples.

EXPERIMENTAL RESULTS AND DISCUSSION

Effect of Resin Particle Size

The effect of particle size on heptane extraction of resin at 20 °C and 500 RPM is presented in Figure 3. It is evident that the extraction rate decreases with an increase in extraction time, which is to be expected from geometric considerations and from the heterogeneity of the fossil resin. As shown in Figure 3, a strong dependence of resin extraction rate on particle size was found. The initial resin extraction rates ($d\alpha/dt$) are plotted against the inverse of the initial resin particle size in Figure 4 and the linear relationship suggests that the initial rate may be limited either by mass transfer or by surface reaction.

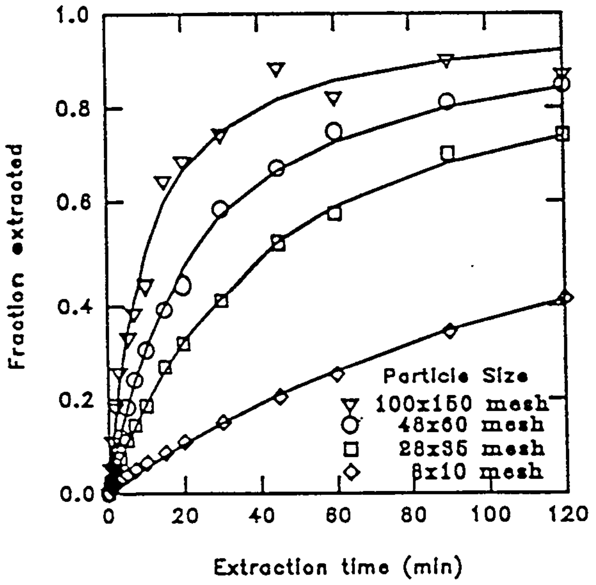

Figure 3. Effect of particle size on heptane extraction of resin at 25 °C and 500 rpm.

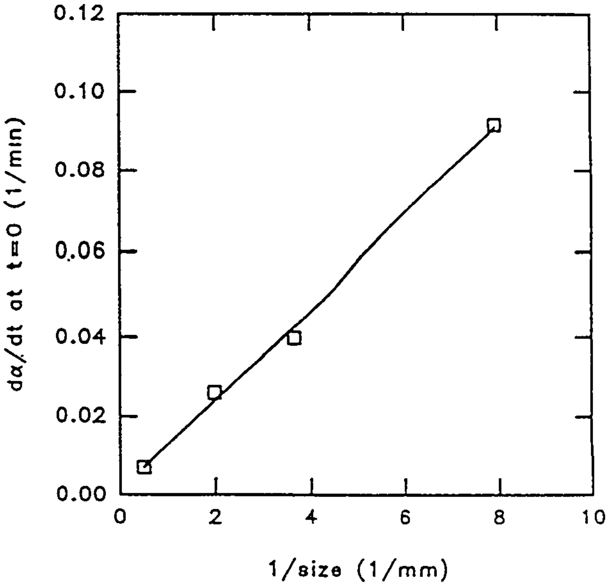

Figure 4. Initial extraction rates vs. the inverse of the particle size from data presented in Figure 3

Effect of Temperature

Several tests on heptane extraction of resin were performed at different temperatures in order to determine the effect of temperature on the extraction rate and the results are presented in Figure 5. As expected, the resin extraction rate was found to increase with an increase in extraction temperature. The effect of temperature is very significant. The initial extraction rates

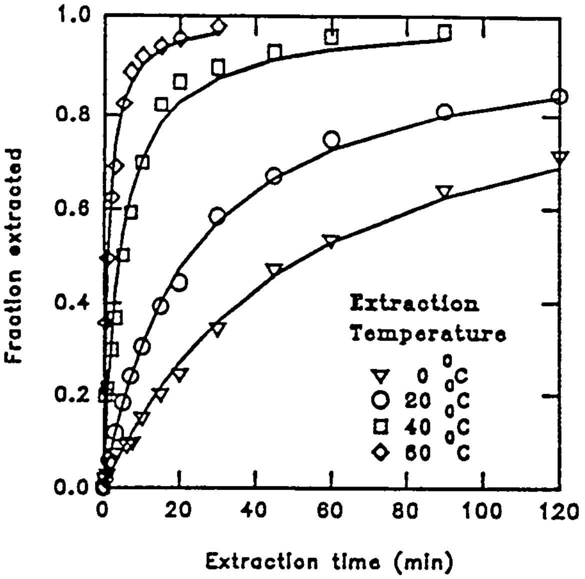

Figure 5. Effect of temperature on heptane extraction of 48×60 mesh resin particles and 500 rpm.

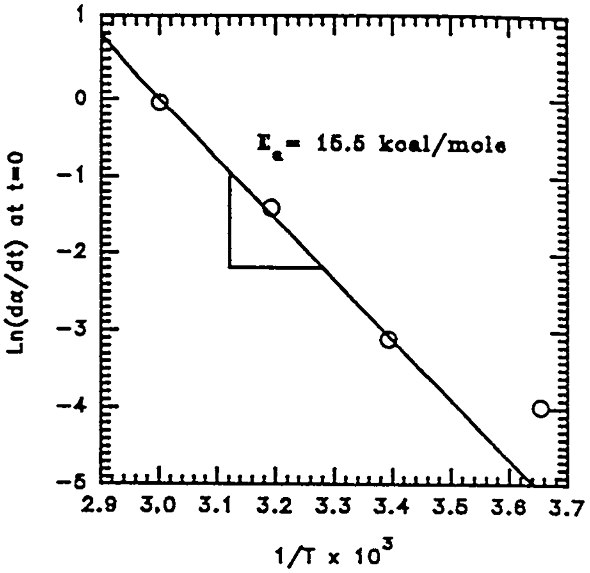

Figure 6. Arrhenius plot for heptane extraction of 48×60 mesh resin particles from data presented in Figure 5.

at different temperatures are plotted according to e Arrehenius equation in Figure 6 and a good linear relationship between the logarithm of the initial extraction rate and inverse temperature was found. From the slope of this linear relationship, an activation energy of 15.5 kcal/mole can be calculated. The magnitude of the activation energy indicates that the extraction process is controlled by a surface reaction mechanism involving the dissolution of resin molecules into the heptane solution. In this regard, the energetics of such solvation reactions are expected to be related to the observed activation energy.

Effect of Agitation Intensity

The effect of agitation intensity on heptane extraction of resin is presented in Figure 7 in terms of stirring speed (RPM). It was found that the agitation intensity has hardly any effect on the resin extraction rate, again providing evidence that the extraction process is not under diffusion or mass transfer control for these experimental conditions (room temperature, low solids concentration, and moderate stirring speed).

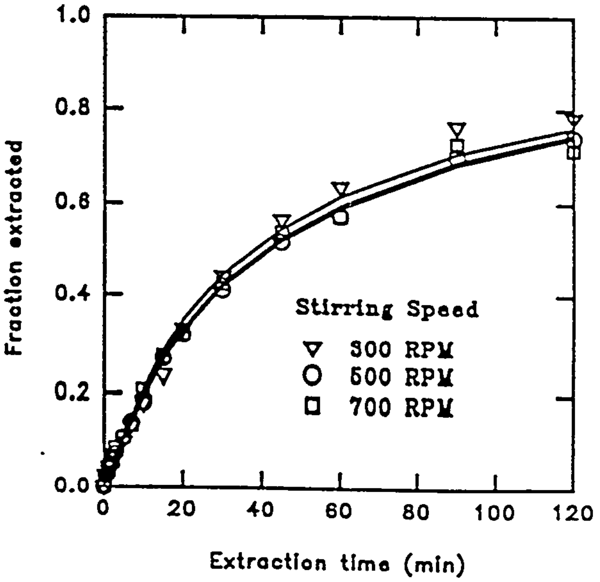

Figure 7. Effect of stirring speed on heptane extraction of 48×60 mesh resin particles at 25 °C and 500 rpm.

Solvent extraction and purification of fossil resin from a resin flotation concentrate is accomplished by dissolving the resin compounds into an organic solvent in which other coal macerals will not be dissolved. It should be possible to describe the resin extraction reaction by a typical kinetic analysis for shrinking core geometry. However, it should be noted that the resin extraction reaction has special features. As mentioned before, the fossil resin particles are heterogeneous in terms of their structure (both macroscopic and molecular) and their chemical composition. And they are complex polymers and copolymers of varied molecular weight. Different components of the fossil resin, even components with the same structural characteristics but different in their molecular weight, are expected to have different dissolution rates in organic solvents such as heptane.

For nonporous uniform particles of spherical shape, the relationship between the extent of extraction (α) and the extraction time (t) for a batch reaction under surface reaction control can be described by the following equation,[13]

$$1 - (1 - \alpha)^{\frac{1}{3}} = kt \tag{1}$$

where k is the overall rate constant which includes reactant concentration and many other factors such as surface area, intrinsic rate constant, initial particle size, shape factor, reactive sites on the particle surfaces et cetera. The constant, k, should be inversely proportional to the initial particle size. However, this traditional shrinking core model does not fit the data shown in Figure 3. It is expected that the rate constant varies with time and thus the kinetic analysis must be considered in more detail.

From characterization studies, it was found that the fossil resin particles are heterogeneous and not of uniform composition based on microscopic examination and chemical analysis. During extraction, it is expected that the resin components with high solubility will be extracted more rapidly than those with low solubility. Therefore, the composition of the extracted resin will change as a function of time and the rate will decrease more than might be expected as the extraction reaction proceeds. A sequential extraction test was conducted to examine this hypothesis. Sequential extraction of the concentrate was carried out for different time intervals and heptane extraction temperatures. One hundred and eighty grams of the concentrate and 1800 ml of heptane were taken into the reactor for the first stage of extraction. After the first extraction, the pregnant resin solution was filtered and the first resin product was obtained by evaporation. The residual resin concentrate was processed with fresh heptane using the same extraction procedure as the first extraction for desired times and temperatures. A stirring speed of 700 RPM was used throughout all experiments. Products from these sequential tests were recovered and the conditions with the analytical results are summarized in Table 2. A Klett-Summerson Photoelectric Colorimeter manufactured by Klett Manufacturing Co., Inc. was used to analyze the resin products obtained. The light beam of the colorimeter has a visible spectral range, from approximately 400 to 465 nm. Toluene was used as the solvent for these transmittance measurements, and the concentration of the resins in the toluene was fixed at 0.1 gram per 50 ml of toluene. The analytical results obtained from the transmittance measurements show that the extracted resin composition changes with an increase in extraction time.

Thus it can be imagined that the resin extraction kinetics must take into consideration the fact that the resin is nonuniform and should be characterized either by a distribution of rate constants or by taking into consideration the fact that the composition of the unreacted particle changes with time.

Table 2. Sequential extraction results and analysis of products

Extracted resin product	Extraction temperature (°C)	Extraction time (min)	Transmittance (%)	Extracted product	
				Wt. (%)	Cumulative Wt. (%)
Product 1	27	10	53.75	48.97	48.97
Product 2	27	20	46.78	12.76	61.73
Product 3	27	90	37.00	7.28	69.01
Product 4	60	20	23.44	3.67	72.68
Final residue			<1	27.32	100.00

CONCLUSIONS

Although fossil resin is heterogeneous in terms of its macroscopic and chemical composition, it is mainly composed of non-polar hydrocarbon compounds with a small number of functional groups containing heteroatoms. The solvent, heptane, is also a nonpolar aliphatic compound. It is expected that no chemical reactions occur in such an extraction process at low temperature (0-90 °C). The main interaction between resin and heptane is expected to be the van der Waals forces of interaction associated with solvation phenomena. In this regard, the resin extraction rate was found to be largely independent of agitation intensity at room temperature, low solids concentration and moderate agitation. Further the initial extraction rate was found to be inversely proportional to the initial particle diameter. Temperature has a significant effect on extraction rate and the activation energy for resin extraction by heptane was found to be 15.5 kcal/mole. On this basis it is expected that the extraction rate is controlled by surface solvation phenomena. Additional research is in progress to develop a kinetic model for the fossil resin extraction reaction.

ACKNOWLEDGMENTS

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BENCH-SCALE TESTING OF A MICRONIZED MAGNETITE, FINE-COAL CLEANING PROCESS

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ABSTRACT

Custom Coals, International has installed and is presently testing a 500 lb/hr. micronized-magnetite, fine-coal cleaning circuit at PETC's Process Research Facility (PRF). The cost-shared project was awarded as part of the Coal Preparation Program's, High Efficiency Preparation Subprogram. The project includes design, construction, testing, and decommissioning of a fully-integrated, bench-scale circuit, complete with feed coal classification to remove the minus 30 micron slimes, dense medium cycloning of the 300 by 30 micron feed coal using a nominal minus 10 micron size magnetite medium, and medium recovery using drain and rinse screens and various stages and types of magnetic separators. This paper describes the project circuit and goals, including a description of the current project status and the sources of coal and magnetite which are being tested.

INTRODUCTION

A recent emphasis of the Department of Energy's (DOE's), Coal Preparation Program has been the development of high-efficiency technologies that offer near-term, low-cost improvements in the ability of coal preparation plants to address problems associated with coal fines. In 1992, three cost-shared contracts were awarded to industry, under the first High-Efficiency Preparation (HEP I) solicitation. All three projects involved bench-scale testing of various emerging technologies, at the Pittsburgh Energy Technology Center's (PETC's), Process Research Facility (PRF). The first project, completed in mid-1993, was conducted by Process Technology, Inc., with the objective of developing a computerized, on-line system for monitoring and controlling the operation of a column flotation circuit. The second project, completed in mid-1994, was conducted by a team led by Virginia Polytechnic Institute to test the Mozely Multi-Gravity Separator in combination with the Microcel Flotation Column, for improved removal of mineral matter and pyritic sulfur from fine coal. The third project, which is the subject of this paper, is presently being conducted by Custom Coals, International (CCI), to evaluate and advance the micronized magnetite-based, fine-coal cycloning technology through integrated circuit operation.

TECHNOLOGY DESCRIPTION

Over the last ten years, the use of micronized-magnetite cycloning for beneficiating fine coal has been researched by both the DOE and Genesis Research Corporation. Based on its work, the DOE received a patent in 1991 titled "Fine-Coal Cleaning via the Micro-Mag Process". Likewise, Genesis Research received patents in 1992 on more complicated processes (ie., Carefree and Self-Scrubbing Coal Processes), involving the micronized-magnetite cycloning technology. In 1993, Custom Coals brought together these technologies by purchasing the rights to the various DOE and Genesis Research patents, and is actively marketting and commercializing the technology both domestically and internationally. Custom Coals is currently constructing a 500 TPH commercial cleaning plant, in Somerset County, PA, employing these technologies, under the DOE's Clean Coal Technology Program. The DOE cofunded demonstration project will occur through 1996, after which the plant will continue to operate commercially, producing compliance coal for local and regional utilities.

The micronized-magnetite coal cleaning technology is based on widely used conventional dense-medium cyclone applications, in that it utilizes a finely ground magnetite/water suspension as a separating medium for cleaning fine coal, by density, in a cyclone. However, the micronized-magnetite cleaning technology differs from conventional systems in several ways:

- It utilizes significantly finer magnetite (about 5 micron mean particle size), as compared to normal mean particle sizes of 10-20 microns.
- It can effectively beneficiate coal particles down to 30 microns in size, as compared to the most advanced, existing conventional systems that are limited to a particle bottom size of about 150 microns.
- Smaller diameter cyclones (ie., 4 to 10 inch) are used to provide the higher G-force required to separate the finer feed coal.
- Cyclone feed pressures 3 to 10 times greater that those used in conventional cleaning systems are employed to enhance the separating forces.
- More advanced magnetite recovery systems, including rare-earth drums or high-gradient magnetic separators, are required for recovery and reuse of the medium.

While the similarity of the micronized-magnetite technology to existing circuitry has contributed to its fairly rapid commercialization, only limited work has been done on the magnetite recovery aspects of the circuit, particularly in an integrated, continuous application. Custom Coals HEP-I project is being undertaken to evaluate and resolve some of these remaining issues, to better understand and improve the overall process economics.

PROJECT OBJECTIVES, DESCRIPTION, AND SCHEDULE

The general objective of the project is to design, construct, and operate a fully integrated, 500 lb/hr. continuous micronized-magnetite cycloning circuit for cleaning fine coal. The work will focus on the medium recovery circuit and the impact of recirculating medium quality on the separation performance of the cyclone. Past semi-continuous research has indicated that highly efficient separations can be achieved in the small diameter cyclone, for the entire 600 by 30 micron particle size range, with magnetite losses less than 10 pounds per ton of coal processed. The testing is designed test these conclusions in a fully-integrated continuous circuit.

In addition, the specific objectives of the project are:

- Establish classifying circuit operating conditions and efficiency for desliming at 30-40 microns.
- Verify past cyclone performance finding, relative to cyclone operating variable optimization.
- Quantify the amount and size of magnetite losses for various recovery circuit configurations.

- Determine the effects of magnetite particle size and medium purity changes on dense-medium cyclone performance.

- Assess the technical and economic feasibility of various magnetite recovery circuit options.

Technically, the testing and data evaluation will focus on establishing the least complicated, easiest to operate circuit, that will provide the proper recirculating medium and cyclone performance. Economically, the evaluation will focus on tradeoffs between circuit capital and operating costs versus overall system performance, including cost of magnetite losses.

To accomplish the various objectives, the project has been divided into 11 task series, which include:

- Task 100: Project Planning and Management
- Task 200: Final Circuit Design
- Task 300: Equipment and Supplies, Procurement and Fabrication
- Task 400: Magnetite and Coal Procurement
- Task 500: Circuit Installation
- Task 600: Circuit Commissioning
- Task 700: Circuit Testing
- Task 800: Analytical
- Task 900: Circuit Decommissioning
- Task 1000: Data Evaluation
- Task 1100: Final Reporting

Custom Coals is responsible for the performance of all the above-mentioned project tasks, in accordance with DOE's Environmental, Safety, and Health (ESH) regulations. They will also provide all personnel required for operation of the emerging technology circuit, including sampling and sample handling, and circuit maintenance.

The DOE staff at the PRF is responsible for (1) receiving and handling of raw coal, (2) preparation and delivery of 500 lb/hr. feed slurry to Custom Coals at the specified coal topsize and solids concentration, (3) receiving, handling, and disposing of all products and discharges from the Custom Coals circuit, and (4) providing all necessary air, water, and electrical utilities.

Table 1 shown below contains the original 16-month schedule for the project, broken down by the task series on the critical path. As of early May, 1995, when this paper is being written, the project has progressed in close proximity to the initial schedule. The circuit commissioning has been completed and the circuit testing is scheduled to begin on May 15th, once all the commissioning test data has been compiled and reported.

TABLE 1
ORIGINAL PROJECT 16-MONTH SCHEDULE
(Task Series on Critical Path)

Task	Task Description	Duration (Months)	Proposed Period
200	Final Circuit Design	4	September through December 1994
500	Circuit Installation	3	January through March 1995
600	Circuit Commissioning	1	April 1995
700	Circuit Testing	5	May through September 1995
900	Circuit Decommissioning	1	October 1995
1,100	Final Reporting	2	November through December 1995

PROJECT TEAM

The project team has been assembled to ensure that all the project objectives, and the specific task series can be completed in accordance with the preceding project schedule and the overall project budget ($1.2M). The project team members, include:

- Custom Coal's as project manager and also to provide site engineering for the installation, commissioning, testing, and decommissioning tasks.
- DOE/PETC staff for technical and financial project oversight.
- Gilbert Commonwealth staff at PETC to operate the existing PRF circuit, which provides feed and product handling for Custom Coals emerging technology (ET) circuit.
- CLI Corporation, which was subcontracted to design the ET circuit and assist with equipment selection.
- Rizzo & Sons Construction, which was subcontracted to install and decommission the ET circuit, and also assisted in the commissioning efforts.
- Commercial Testing & Engineering Co. (CT&E), which is providing the operating technicians for the testing task and conducting all routine coal sample analyses (proximate analyses, sulfur forms, screen analyses, and washabilities).
- MTU's Institute of Materials Processing (IMP), which has been subcontracted to perform the more complex magnetite-related analyses required in the project (ie., magnetic separations, magnetic moment measurements, fine particle size analyses, and magnetite rheology measurements).

The fineness of the micronized magnetite has made the sampling, sample preparation, and analytical requirements for this project more complex and challenging than in conventional coal cleaning applications.

CIRCUIT DESCRIPTION

Figure 1 contains the block flow diagram of the ET circuit that Custom Coals has installed in the PRF, and is presently testing. The circuit consists of three subcircuits:

- **Classification Circuit** - Which consists of a 2" Krebs Classifying Cyclone in combination with a Sizetec Inclined Desliming Screen. This equipment will be equipped with various cyclone orifices and screen panel sizes to make a separation at 30-40 microns, removing the ultrafine particles to the waste via the cyclone overflow. The classification circuit will receive feed slurry from the PRF at about 40Wt% solids, and will deliver a final product to the dense-medium cycloning circuit at about 50-60Wt% solids (ie., 500lb/hr of prinicipally 300 by 30 microns particles).

- **Dense-Medium Cycloning Circuit** - Which consists of two parallel Krebs Dense-Medium Cyclones (2" & 4" diameter). The 4" cyclone products will always be recirculated back to feed, and will be used for closed-loop testing. The 2" cyclone products will progress to the magnetite recovery circuit, and will be used for the open circuit testing. Magnetite will be added on a controlled basis from the Magnetite Bin to maintain the desired correct media specific gravity.

- **Magnetite Recovery Circuit** - Which consists of a Sizetec Inclined Desliming Screen (Drain Screen), and a Sizetec Horizontal Dewatering Screen (Rinse Screen). The circuit also consists of four Eriez Conventional, Wet-Drum Magnetic Separators configured as primary, secondary, tertiary, and cleaner separators, and an Eriez High Gauss, Rare-Earth Magnetic Separator to be used as a final scavenger. The final magnetic concentrates will report to the Correct Medium Sump and the scavenger tailings to the Waste Sump. The Waste Sump discharge will be dewatered using the PRF's existing Sharples Horizontal Centrifuge and Thickener, and all clarified water will be recirculated for reuse in the circuit.

In addition to the equipment shown in Figure 1, the ET circuit also contains a clarified water head tank and pump to provide all water additions to the circuit. A Motor Control Center (MCC) and Control Cabinet provide the power distribution and the required circuit control.

COAL AND MAGNETITE DESCRIPTION

Custom Coals has selected two coals for testing during the project. The two coals are a Pittsburgh No. 8 Seam Raw Coal from Belmont County, Ohio, and a Lower Kittanning "B" Seam Raw Coal from Somerset County, PA. Table 2 contains a description and raw coal analyses for both coals.

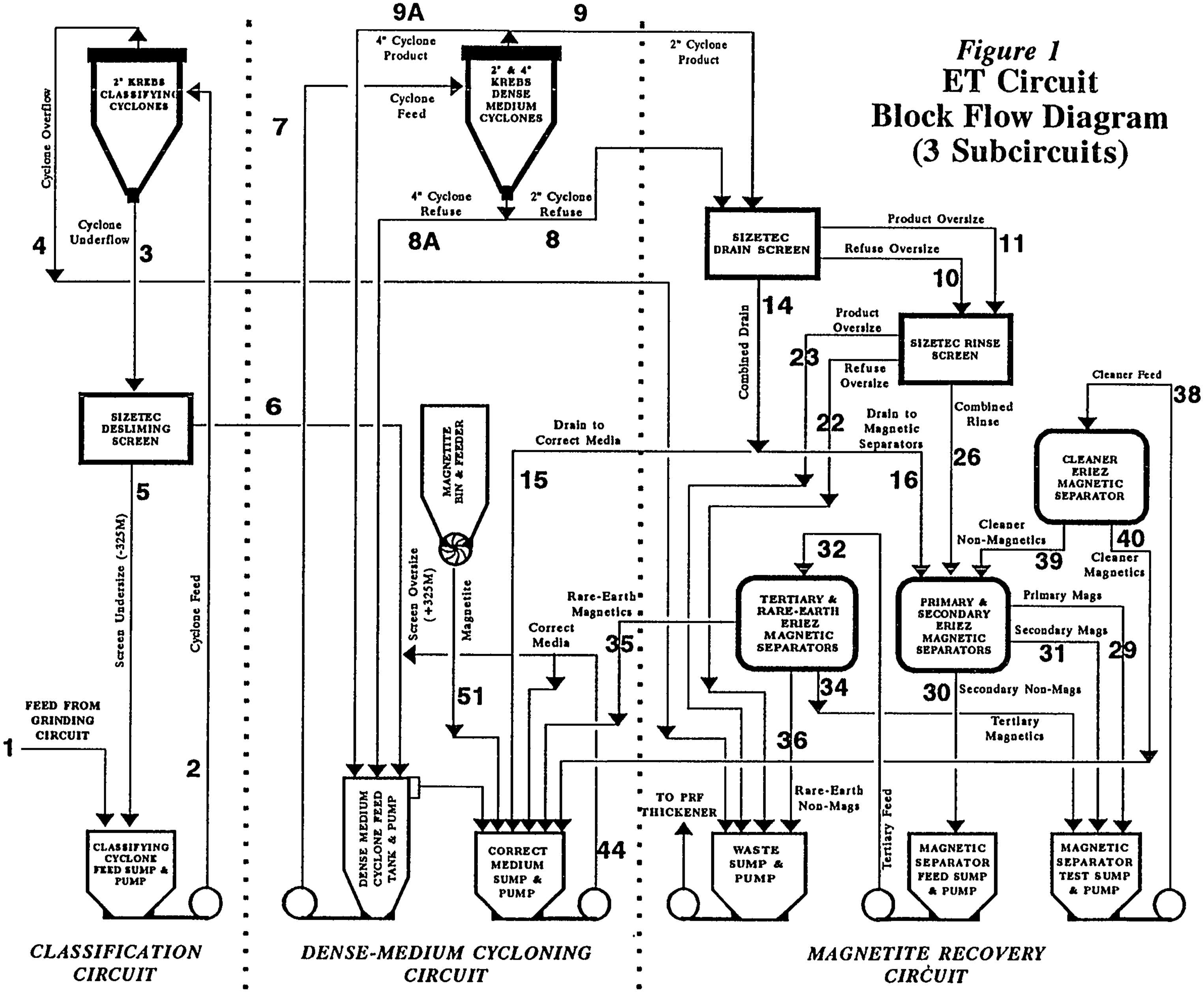


Figure 1
ET Circuit
Block Flow Diagram
(3 Subcircuits)

TABLE 2
TEST COAL CHARACTERISTICS

Description	Coal #1 (75-100 Tons)	Coal #2 (50-75 Tons)
Raw Coal Seam	Pittsburgh No. 8 Seam	Lower Kittanning "B" Seam
Company	Ohio Valley Coal Company	PB&S Coal Company
Mine	Powhatan #6 Mine	Longview Mine
County, State	Belmont County, OH	Somerset, PA

	Pittsburgh No. 8 Seam		Lower Kittanning "B" Seam	
Raw Coal Analyses	**As Received**	**Dry Basis**	**As-Received**	**Dry Basis**
Moisture (Wt%)	6.0	--	4.7	--
Ash (Wt%)	25.9	27.6	21.8	22.9
Sulfur (Wt%)	4.2	4.5	2.1	2.2
Pyritic Sulfur (Wt%)	2.1	2.2	1.3	1.4
Hardgrove Grind Index (HGI)		60-70		90-100

The two coals in Table 2 were selected for the testing because they both possess some common favorable characteristics, that include:

- they contain dry ash contents between 20 and 30 Wt%,
- that have over half of sulfur present in the pyritic form, and
- they have anticipated yields of 70-80Wt%, when cleaned at 1.60 SG.

These characteristics make them good candidates for aggressive cleaning studies. In addition, the large difference in the hardness of the two coals (see Table 2) will form an interesting comparison of how attrition affects fine-coal contamination of the recirculating medium, and subsequent medium recovery. Lastly, the Lower Kittanning "B" Seam raw coal is of specific interest, because it will be one of the major feed coals for eventual commercial operation, at the 500 TPH Cleaning Plant that Custom Coals is presently building in Somerset County, PA.

Initially, Custom Coals considered two potential sources of magnetite for this project and its commercial cleaning plant. They included:

- Synthetic Magnetite - Made from an iron chloride pickle liquor. Samples of this magnetite were previously provided by Hazen Research and tested in earlier research.

- Natural Magnetite - Made from dry grinding and classifying conventional Grade-B magnetite. Samples of this magnetite were provided by PennMag Inc.

Table 3 contains a comparison of the size consist of these two micronized magnetite options, and conventional Grade-B and Grade-E magnetite used by industry. It can be noted that the two micronized magnetites are considerably finer, and principally consist of 10 by 2 micron particles.

TABLE 3

Magnetite Types & Size Distribution

- MICRONIZED MAGNETITE OPTIONS
  - Penn Mag Grade-K: Natural Magnetite
  - Hazen High Temp: Synthetic Magnetite

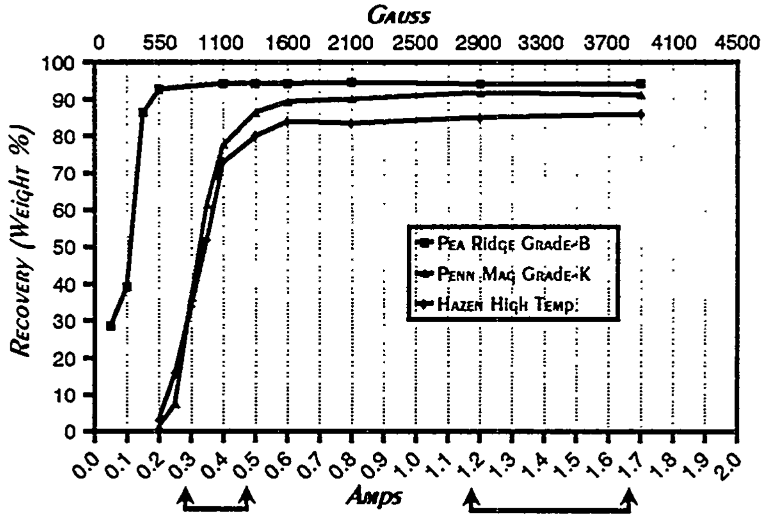
Magnetite Type	Topsize (microns)	90% Passing Size (microns)	50% Passing Size (microns)	10% Passing Size (microns)
Commercial Grade-B *(1)*	100-150	40-50	15-20	5-10
Commercial Grade-E *(1)*	60-100	30-40	10-15	3-5
Hazen High Temp *(2)*	60	10	6.5	2.0
PennMag Grade-K *(2)*	20	11	4.8	2.0

(1) From Sieve Analyses
(2) From Microtrac Analyses

FIGURE 2

Davis Tube Recovery Profiles

Force = Mass * Magnetite Susceptibility * Field Strength * Field Gradient



Conventional Wet-Drum Separator
(Recovery Limit)

Rare-Earth Wet-Drum Separator
(Recovery Limit)

Figure 2 contains a comparison of the recoverability of the two micronized magnetites and a Grade-B magnetite, determined at various settings of a Davis-Tube. The lower magnetic susceptibility of the micronized magneitites is readily apparent, from the higher amperage/gauss levels required to recover the magnetite. Many of the particles are recovered at a Gauss Range (1000-1500 Gauss) that is near the limits of recoverability in conventional wet-drum magnetic separators. For this reason, Custom Coals and Eriez Magnetics have specified rare-earth drum magnetic separators as scavengers in the magnetite recovery circuits to ensure adequate micronized magnetite recovery.

At present, Custom Coals is planning to use natural magnetite provided by PennMag Inc., for the HEP-I project and the commercial plant. This natural magnetite was preferred over the synthetic magnetite, provided by Hazen Research, because:

- It can be provided at a lower cost ($160 per ton) than the synthetic magneitite,
- It can be made at high purity (95-98Wt% recovery at 1.70 amps on the Davis Tube) with little oversize (20 micron topsize), and
- The process for making it is not very complex or difficult to adapt for existing suppliers.

Custom Coals has obtained and plans to test two different sizes of micronized magnetite provided by PennMag during the HEP-I project. They include a Grade-J magnetite at 8-10 micron mean particle size, and the Grade-K magnetite at 4-6 micron mean particle size. A third, even finer grade of magnetite, will be tested, if these magnetites prove to make unstable mediums at high cyclone pressures.

TESTING PLAN

The circuit testing task is just beginning, and should occur over a 5-month period (May through September 1995). The circuit testing will be broken down into two phases for the two coal and two magnetite options, which include:

- Component Testing
- Integrated Testing

The Component Testing will focus on closed-loop testing of each subcircuit to optimize the individual unit operations and observe the impact of key process variables. The testing for each coal and magnetite will then culminate with the Integrated Testing, where the entire circuit is operated in longer duration, primarily to quantify magnetite losses, and determine the impacts of changing medium quality on cyclone performance. Several different feed coal topsizes and medium densities are planned for the Integrated Testing.

SUMMARY

This project is expected to provide significant insight into the effects of various medium recovery circuits on the recirculating medium quality and separation performance of a micronized-magnetite cycloning system for fine-coal cleaning. The technical and economic feasibility of the selected circuitry and operating conditions will be fully evaluated and translated into a practical assessment for the potential commercial application of the technology.