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Engineering Development of Advanced Physical Fine Coal Cleaning for Premium Fuel Applications

**Topical Report
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ABSTRACT

The primary goal of this project is the engineering development of two advanced physical fine coal cleaning processes, column flotation and selective agglomeration, for premium fuel applications. The project scope included laboratory research and bench-scale testing on six coals to optimize these processes, followed by the design, construction and operation of 2 t/hr process development unit (PDU). The project began in October 1992 and is scheduled for completion by September 1997.

This report represents the findings of the PDU Advanced Column Flotation Testing and Evaluation phase of the program and includes a discussion of the design and construction of the PDU. Three compliance steam coals, Taggart, Indiana VII and Hiawatha, were processed in the PDU to determine performance and design parameters for commercial production of premium fuel by advanced flotation. For the PDU work, the coals were stage ground with water in ball mills followed by closed-circuit finish grinding in a Netzsch stirred mill. The ground slurry was cleaned by froth flotation in a 6-ft dia Microcel™ column. Wash water was distributed across the top of the column, and the products were dewatered to allow for a closed-circuit water recycle system.

It was important that the coals be ground fine enough for liberation of the mineral so that the target ash specification could be met. The wash water additions were effective for rinsing entrained clay from the froth, and the most relevant parameters effecting product quality and yield were frother dosage, aeration rate, and the recirculation rate through the in-line aerator. The following results were achieved after optimization of the operating parameters:

	<u>Feed, lb/hr</u>	<u>Grind (D₈₀), μm</u>	<u>Ash, lb/MBtu</u>	<u>Btu Rec, %</u>
Taggart	4,200	60	1.0	96.9
Indiana VII	3,200	23	2.3	82.0
Hiawatha	4,300	48	1.9	88.0

Consistent, reliable performance of the PDU was demonstrated by 72-hr production runs on each of the test coals. Its capacity generally was limited by the dewatering capacity of the clean coal filters during the production runs rather than by the flotation capacity of the Microcel™ column.

The residual concentrations of As, Pb, and Cl were reduced by at least 25% on a heating value basis from their concentrations in the test coals. The reduction in the concentrations of Be, Cd, Cr, Co, Mn, Hg, Ni and Se varied from coal to coal but the concentrations of most were greatly reduced from the concentrations in the ROM parent coals. The ash fusion temperatures of the Taggart and Indiana VII coals, and to a much lesser extent the Hiawatha coal, were decreased by the cleaning.

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EXECUTIVE SUMMARY

The goal of this project was the engineering development of advanced column flotation and selective agglomeration technologies for premium fuel applications. This project was a major step in the Department of Energy's (DOE) program to show that ultra-clean coal-water slurry fuel (CWF) can be produced from selected coals and that this premium fuel will be a cost-effective replacement for oil and natural gas now fueling some of the industrial and utility boilers in the United States, as well as for advanced combustors currently under development. The replacement of oil and gas with CWF can only be realized if retrofit costs are kept to a minimum and retrofit boiler emissions meet national goals for clean air. These concerns establish the specifications for maximum ash and sulfur levels and combustion properties of the CWF.

The engineering development work focused on two advanced coal cleaning technologies - selective agglomeration and column flotation. The scope of the project included laboratory and bench-scale research and development work on these processes as well as the design, construction, and operation of a 2 tph process development unit (PDU). The purpose of this report is to present the findings of this study as well as any conclusions and recommendations for design of commercial operations. This multi-year cost-share contract started on October 1, 1992, and is scheduled for completion by September 1997.

SPECIFIC OBJECTIVES OF PROJECT

The project had three major objectives:

- The primary objective was to develop the design base for commercial prototype advanced fine coal cleaning facilities capable of producing ultra-clean coals suitable for conversion to stable, highly loaded coal-water slurry fuels. These slurry fuels should contain less than 2 lb ash/MBtu HHV (860 grams ash/gigajoule) and preferably less than 1 lb ash/MBtu HHV (430 grams ash/gigajoule), and less than 0.6 lb sulfur/MBtu HHV (258 grams sulfur/gigajoule). The advanced fine coal cleaning technologies to be employed are advanced column froth flotation and selective agglomeration. Operating conditions during the advanced cleaning processes should recover at least 80 percent of the heating value in run-of-mine source coals at an annualized cost of less than \$2.50/MBtu (\$2.37/gigajoule), including the cost of the raw coal.
- A secondary objective of the work was to develop the design base for near-term commercial applications of these advanced fine coal cleaning technologies. These applications should be suitable for integration into new or existing coal preparation plants for the purpose of economically and efficiently processing minus 28-mesh coal fines. The design base also included the auxiliary systems required to yield a shippable, marketable product such as a dry clean coal product.

- A third objective of the work was to determine the distribution of toxic trace elements between clean coal product and refuse during the cleaning of various coals by advanced froth flotation and selective agglomeration technologies. Twelve toxic trace elements have been targeted. They are antimony, arsenic, beryllium, cadmium, chromium, cobalt, lead, manganese, mercury, nickel, selenium, and chlorine. The results show the potential for removing these toxic trace elements from coal by advanced physical cleaning.

APPROACH

The project team consisted of Cyprus Amax Minerals Company through its subsidiaries Amax Research & Development Center (Amax R&D) and Cyprus Amax Coal Company (Midwest and Cannelton Divisions), Arcanum Corporation, Bechtel Corporation, Center for Applied Energy Research (CAER) of the University of Kentucky, and the Center for Coal and Mineral Processing (CCMP) of the Virginia Polytechnic Institute and State University. Entech Global, Inc. managed the project for Amax R&D and provided research and development services. Dr. Douglas Keller of Syracuse University and Dr. John Dooher of Adelphi University were consultants to the project.

The project effort was divided into four phases which were further divided into eleven tasks including coal selection, laboratory and bench-scale process optimization research and testing, along with design, construction, and operation of a 2 ton/hr PDU. Tonnage quantities of the ultra-clean coals was produced in the PDU for combustion testing. Near-term application of advanced cleaning technologies to existing coal preparation plants was also studied.

RESULTS AND DISCUSSION

This report is concerned with advanced column froth flotation technology for production of premium fuels. Findings of the activities associated with the coal selection (Task 2), laboratory and bench-scale research and development (Task 4), and PDU-scale column flotation test work (Task 8) are summarized in this report.

Laboratory and Bench-Scale Work

Activities associated with laboratory and bench-scale column flotation testing are discussed below.

Selection of Test Coals (Subtask 2.1)

Successful accomplishment of project objectives by both froth flotation and selective agglomeration processes is dependent on the selection of suitable source coals. Due to the widely varying quality and economic factors of United States coals, only selected coals could be considered as a feedstock for this project. Accordingly, guidelines were

established to evaluate a number of candidate coals and select six coals for use in this project. Overall, five bituminous coals and one low-rank coal were to be selected. The select five bituminous coals and producing states were Taggart (VA), Sunnyside (UT), Indiana VII (IN), Winifrede (WV), and Elkhorn No. 3 (KY). The single low-rank coal and producing state was Dietz (MT).

Laboratory Grinding and Mineral Liberation Studies (Subtask 4.1)

The objective of this Subtask was to study the grinding and mineral liberation characteristics of five of the six test coals. The Dietz coal (sub-bituminous), was not tested due to the inability to develop a flotation scheme for a sub-bituminous coal. The objective was achieved by parametric laboratory and bench scale testing of various grinding configurations at variable rates (100 kg/hr to 500 kg/hr).

The work indicated that a closed-circuit grinding configuration was more effective than an open-circuit configuration at providing greater capacity for a given grind size. The benefit was most evident when grinding to very fine particle sizes, such as those needed for the Indiana VII and Winifrede coals. The grinding / liberation testwork performed under Subtask 4.1 determined grinding power requirements for various coals. This data formed the design basis of the PDU grinding circuit.

Laboratory Flotation Testwork (Subtask 4.2)

The objective of the laboratory column flotation testwork performed under Subtask 4.2 was to determine the preferred column design and operating conditions for cleaning the six test coals. In order to accomplish these objectives, the work was distributed among various project team members.

Testing revealed that all five bituminous coals responded quite well to column flotation. Residual ash, sulfur, and heating value recovery targets were easily achieved with the Elkhorn No. 3, Taggart, and Sunnyside coals. Testing of the Indiana VII and Winifrede coals indicated that the product ash and heating value targets need to be relaxed. The sixth test coal (low rank Dietz coal) did not respond well to laboratory flotation cell testwork, and as a result, was not subjected to column flotation testwork.

Overall, the performance of the Microcel™ and Ken-Flote™ units were very similar and superior to that of the packed column. As a result, it was suggested that both Microcel™ and Ken-Flote™ columns be evaluated at bench scale under Subtask 4.4.

Bench Scale Column Flotation Testing (Subtask 4.4)

Based on the results of laboratory flotation testwork conducted under Subtask 4.2, five of the six original test coals were subjected to bench-scale column flotation testing. The objective of this work was to verify that the performance and operating characteristics observed in laboratory testing can be scaled up to a continuous feed

rate of 100 lb/hr. The response of each coal to cleaning in a 1-foot diameter Ken-Flote™ column and Microcel™ column was evaluated. Each coal was subjected to numerous tests in both flotation columns. Several operating parameters were varied and their effect on performance monitored. Overall, a number of useful observations were made during the testwork. They were:

- Product ash targets were met for all five coals tested in the 12-inch columns.
- There was an obvious trend showing that the unit capacity of the flotation columns increased as the particle size distribution of the feed slurry became coarser.
- Wash water requirements needed for ash rejection were lower for coarse particle size distributions.
- Reagent requirements for the Microcel™ flotation column were lower than those needed for the Ken-Flote™ unit.
- The Microcel™ unit typically had a greater feed capacity than the Ken-Flote™ unit.
- The Ken-Flote™ column incurred significantly more downtime and, as a result, required more maintenance than the Microcel™ unit.

Based on this testwork, the Microcel™ flotation column was chosen for further process development work in the 2 tph PDU flotation module.

Final Coal Selection and Procurement (Subtask 8.1)

Based on the performance of the six test coals in the laboratory and bench scale testwork, the following three coals were selected for use in the PDU Flotation Module.

Taggart Coal - Taggart coal was the only test coal that could be cleaned to less than 1.0 lb ash/MBtu ash and less than 0.6 lb/MBtu sulfur while recovering 80 percent of the heating value (raw coal basis). Because Taggart coal is low in ash and sulfur, fine grinding was not necessary. The coal responded very well to column flotation with PDU yields expected to exceed 95 percent. Clean coal slurry prepared from Taggart coal approached 70 percent solids loading by weight.

Indiana VII Coal - Indiana VII coal was recommended for use in the PDU flotation module for reasons quite opposite to that of Taggart. Specifically, Indiana VII was chosen to challenge the capabilities of the PDU flotation and selective agglomeration modules. Though the coal required fine grinding for adequate mineral and sulfur liberation (d_{80} of 20 microns), testing indicated that the clean coal ash goal of 2.0 lb/MBtu can be achieved.

Sunnyside Coal - Sunnyside coal from Utah was selected as the third and final coal for testing in the PDU flotation and agglomeration modules. It is a low-sulfur coal with the ability to easily meet the project quality goals after moderately fine grinding (d_{80} of 45 microns). In addition, a coal from the Utah / Colorado area could represent a dependable fuel source for the western United States. It should be noted that shortly before the PDU operation, Sunnyside coal became unavailable due to mine closure. Another coal (Hiawatha coal) from a nearby mine was identified as a suitable replacement for Sunnyside with similar performance, quality, and geographic considerations.

Design and Construction of the PDU Flotation Module

Activities associated with the conceptual design, detailed design, and construction of the PDU flotation module are discussed below.

Conceptual Design of PDU Flotation Module (Subtask 4.5)

The conceptual design of the PDU Flotation Module was a collaborative effort between Bechtel, Entech, CCMP, and CAER. A majority of effort was dedicated to the following areas:

Selection of Flotation Column - The Microcel™ flotation column was selected for use in the PDU primarily due to its superior performance and ease of operation. Testing indicated that very high yield and quality values could be achieved with the Microcel™ unit. In addition, its bubble generation system is designed in a manner which minimized maintenance, increases bubble generation flexibility, and is easier to scale up.

Cyclone / Fine Coal Screen Selection - A decision was made to install a cyclone / screen circuit for size classification in the grinding circuit. Not only would the equipment produce the needed size distribution for the PDU, it could do so with a significant savings in capital, operating, and maintenance costs.

Selection of Fine Grinding Mill - Due to the increased liberation requirements of the Indiana VII coal (d_{80} of 20 microns) and the inability of conventional ball mills to achieve this product size analysis, a fine grinding mill was incorporated into the grinding circuit. Several fine grinding mills were evaluated for use in the PDU - a tower mill, attritor mill, and horizontal bead mill. The horizontal bead mill, supplied by Netzsch, Inc. was selected due to its compact size and prior operating record in the coal processing industry.

Elimination of Rougher / Cleaner Flotation Circuit - A decision was made to use a single Microcel™ flotation column as opposed to a rougher / cleaner flotation circuit. Studies indicated that a single unit could produce the target high grade product with a significant capital savings over a multi-stage circuit.

Sizing of Microcel™ Flotation Column - Because the size requirements of a flotation column vary from coal to coal, a decision was made to size the single Microcel™ flotation column based on the test coal with average bench-scale performance characteristics. As a result, a 6-foot diameter unit was chosen based on the bench-scale performance of the Sunnyside coal. The column would be oversized for the Taggart coal and undersized for the Indiana VII coal.

Use of Existing DOE Filters - A decision was made to use a rotary drum filter as well as a pair of Netzsch plate and frame filters utilized in a previous DOE project. Though the filters were not ideally suited for the steady state production capacities of each PDU test coal, the capital savings to the project were significant.

Selection of High Capacity Thickener - In an effort to conserve capital costs, operating costs, and space, a decision was made to use a high capacity Enviro-Clear thickener in the PDU tailings circuit. The unit, which was installed inside the pilot plant, eliminated the high capital cost associated with a large static thickener to be installed outside as well as operating and maintenance costs associated with cold weather.

Detailed Design of PDU Flotation Module (Task 5.0)

The detailed design of the PDU Flotation Module was performed by Bechtel Corporation of San Francisco, CA with support from Entech Global engineers. All structural drawings as well as P&ID's were completed by Bechtel and issued for construction. Electrical drawings were issued by Control Technologies, Inc. and approved by Bechtel Corporation.

Entech Global managed the procurement of all instrumentation as well as new and refurbished capital equipment items used in the PDU Flotation Module. Altogether, 47 pieces of new equipment and 52 pieces of instrumentation / control equipment were purchased. Twelve pieces of existing Amax / DOE equipment were refurbished.

Construction of PDU Flotation Module (Subtask 8.2)

Request for Quotation (RFQ) packages were issued to eight companies for the PDU construction subcontract during the first quarter of 1995. A site inspection for interested bidders was held on February 2, 1995 and February 16, 1995. Bids were received from three companies on February 28, 1995. After careful evaluation, the subcontract for the construction of the PDU Flotation Module of the PDU was awarded to TIC - The Industrial Company, of Steamboat Springs, Colorado. An award meeting was conducted on March 15, 1995 and site mobilization occurred March 27, 1995.

TIC was responsible for the installation of all process equipment, instrumentation, structural steel, concrete, process piping, power systems, and control systems related to the operation of the PDU Flotation Module. Mechanical and electrical completion of the PDU was achieved on July 31, 1995 and August 31, 1995 respectively.

PDU Flotation Module - Start-up and Shakedown

Activities associated with the staffing, start-up, and shakedown of the PDU flotation module are discussed below.

PDU Flotation Module - Operating Personnel / Staffing

The PDU flotation module required many different crafts and skills to properly operate and maintain the equipment found in each unit operation. Because research and development was the main thrust behind this project, technicians who possessed strong operating, maintenance, and analytical skills were utilized.

Entech Global, Inc. management established a staffing schedule to ensure that these objectives were realized. A total of four operating personnel, one laboratory technician, and one project engineer were required for safe and effective operation of the PDU during one shift operation. Additional staff was provided during round-the-clock operations needed for production runs.

PDU Flotation Module - Equipment Shakedown

Efforts related to the PDU module shakedown commenced during the third quarter of 1995 and concluded during the fourth quarter of 1995. Any problems, mechanical or electrical, were corrected by TIC or Entech personnel.

Some additional problems (mostly electrical) were discovered during initial shakedown efforts. As a result, a TIC electrician was retained through the latter part of September, 1995 to complete the associated changes. Proportional Integral Derivative (PID) Loops were then configured and tested.

PDU Flotation Module Test Plan

The PDU flotation module test plan was completed and submitted to DOE on December 14, 1995 for review and approval. A training session, described in the test plan, was attended by all technicians on December 7, 1995. The session covered startup, operation, and shutdown of the PDU flotation module.

Though parametric testing of the three test coals was initially scheduled for December, 1995, unexpected problems encountered during startup and shakedown of the PDU flotation module pushed the start of test work to January, 1996. This schedule adjustment did not impact the overall project schedule since PDU process improvements allowed more testing to be completed each day. Testing was first performed on the Taggart coal followed by the Indiana VII and Hiawatha coals.

PDU Flotation Module - Operation and Testwork

Operation of the PDU Flotation Module commenced in January, 1996 with the Taggart coal and concluded in September, 1996 with the Hiawatha coal. Activities associated with the operation and testwork of the Taggart, Indiana VII, and Hiawatha coals are discussed below.

Taggart Coal

Parametric testing of the Taggart coal in the PDU Flotation Module commenced in January, 1996 and was concluded during March, 1996. The operation of the PDU with Taggart coal was quite successful with project goals achieved on numerous occasions.

Testing and Optimization of Grinding Circuit (Taggart Coal) - This test work was performed to determine the best grinding scenario for optimum liberation of mineral matter. Laboratory testing had shown that adequate liberation was achieved when the Taggart coal was ground to a d_{80} of 50 microns (80 percent passing 50 microns). The challenge faced by the PDU staff was to determine which grinding arrangement would produce a similar size distribution. Twenty-five tests aimed at optimizing the grinding circuit were conducted. An evaluation of the resulting data indicated that the desired clean coal quality of 1 lb ash/MBtu could be achieved in the PDU at a d_{80} of 52 microns achieved by using both ball mills, 100 mesh screens and 3-inch cyclones.

Parametric Testing of Microcel™ Flotation Column (Taggart Coal) - A test matrix was established to determine the effects of independent variables such as air rate, percent solids, feed rate, wash water, and reagent dosage on response variables such as product ash and yield. Like the Taggart coal evaluated in the 12-inch Microcel™ unit, the feedstock used in the PDU flotation module was easily floatable. In fact, the natural floatability of the Taggart coal produced comparable yield and quality values regardless of the change in the input parameters. Noticeable changes in the yield and quality were typically observed only when the input parameters were varied dramatically. Overall, the quality goal of 1 lb ash/MBtu was met or exceeded in four tests. The clean coal yield varied from 58.5 to 96.6% while the energy recovery and product quality varied from 60.1 to 98.0% and 0.77 to 1.23 lb ash/MBtu respectively.

Development of Regression Equations (Taggart Coal) - Data from PDU Flotation Module parametric testing was compiled and multiple regression models (equations) were generated. Forward stepwise regression produced equations which link output variables such as yield and clean coal quality to input variables such as feed rate, wash water rate, air rate, collector addition, and frother addition.

The Coefficient of Determination (R^2) and Adjusted Coefficient of Determination (Adjusted R^2) both indicated that the each equation fits the data quite well. In addition, the t-statistic, which indicates the relative importance of each independent variable to the response equation, revealed that the most important variable that affects yield and clean coal ash is the frother dosage.

Optimization of Flotation Column (Taggart Coal) - The previously developed equations were used to determine optimum Microcel™ setpoints needed to achieve the process development goals of 1 lb ash/MBtu and over 80% energy recovery. A total of seven tests were performed. The overall quality goal of 1 lb ash/MBtu was achieved in two tests. The clean coal yield varied from 87.5% to 88.3% while the product ash varied from 0.88 lb ash/MBtu to 0.97 lb ash/MBtu. It is important, however, to note that the aforementioned results correspond to a d_{80} of approximately 60 microns. Parametric testing, on the other hand, indicated improved yield and energy recovery when the d_{80} was close to 50 microns. As a result, the yield and product ash values of 94.4% and 0.99 lb ash/MBtu achieved during parametric test T-25 was considered optimum and used for the production run.

Extended Production Run of PDU Flotation Module (Taggart Coal) - An extended production run of the PDU Flotation Module was successfully completed during the week of March 25, 1996. The effort commenced Monday, March 25 at 11:30 AM and concluded 72 hours later on Thursday, March 28 at 11:30 AM. Aside from a failed belt splice, uninterrupted operation was achieved showing excellent reliability of the operation.

The PDU was operated at a feed rate of approximately 3,800 lb/hr due to filter capacity limitations previously determined during parametric testing. Had the feed rate been greater than 3,800 lb/hr, the PDU would have shut down prematurely due to a lack of clean coal slurry storage capacity. Overall, 275,340 pounds of coal (137.67 tons) was processed in the PDU Flotation with a yield of 95.3%, energy recovery of 96.9% , and clean coal quality of 1.22 lb ash/MBtu.

Disposal of Clean Coal Product (Taggart Coal) - Communications with DOE/PETC indicated that Penn State University (PSU) was interested in procuring the Taggart clean coal produced during the extended production run of the PDU Flotation Module. Fifty supersacks of Taggart clean coal product was shipped to Penn State. The remaining clean coal product was either shipped to Western Aggregates in Golden, CO for use as a fuel or disposed at a local landfill.

Indiana VII Coal

Parametric testing of the Indiana VII coal in the PDU Flotation Module commenced in April, 1996 and was concluded during July, 1996. Though the product ash goal of 2 lb ash/MBtu was difficult to achieve, the operation of the PDU with Indiana VII coal was considered quite successful.

Laboratory Release Analysis (Indiana VII Coal) - To better define the theoretical grade-yield curves associated with different feed particle size distributions, release analysis test work was performed on the Indiana VII coal. Two Microcel™ feed slurries, one having a d_{80} of 22 microns and a second with d_{80} of 19 microns, were evaluated. It was discovered that the product goal of 2 lb ash/MBtu can be achieved at yields of 74% and 83% for feed slurries having a d_{80} of 22 and 19 microns, respectively. The increase

in yield can be attributed to enhanced liberation of carbon and mineral matter at the finer grind. It is important to note that though the additional yield associated with a d_{80} of 19 microns may be desirable, the production of a similar size consist in the PDU flotation module would result in a dramatic reduction in filtering capacity as well as an increase in product moisture. As a result, all PDU test work was performed at a d_{80} of 22 microns.

Testing and Optimization of Grinding Circuit (Indiana VII Coal) - This test work was performed to determine the best grinding scenario for optimum liberation of mineral matter. Laboratory testing had shown that adequate liberation was achieved when the Indiana VII coal was ground to a d_{80} of 20 microns (80 percent passing 20 microns). The challenge faced by the PDU staff was to determine which grinding arrangement would produce a similar size distribution. An evaluation of the resulting data indicated that the desired clean coal quality of 2 lb ash/MBtu could be achieved in the PDU at a d_{80} of 20 microns, achieved with both ball mills and the fine grinding mill, 270 mesh screens and 2-inch cyclones.

Modification of Area 100 Grinding Circuit (Indiana VII coal) - The fine liberation requirements of the Indiana VII coal led to several unexpected operating problems in the PDU grinding circuit. Specifically, degradation and loss of grinding media from the ball mills resulted in screen blinding, cyclone plugging, increased d_{80} values, and unexpected downtime.

Consultations with Mineral Resource Development Inc., a firm specializing in grinding and size reduction, revealed that the speed of each mill was too fast, the ball size distribution was too coarse, the ball charge was too heavy, and the mill solids concentration was too dilute. The correction of these problems resulted in smooth operation of the PDU grinding circuit and consistent d_{80} values.

Parametric Testing of Microcel™ Flotation Column (Indiana VII Coal) - A test matrix was established to determine the effects of independent variables such as air rate, % solids, feed rate, wash water, and reagent dosage on response variables such as product ash and yield. The quality goal of 2.00 lb ash/MBtu was achieved on five occasions. Unfortunately, the product yield and energy recovery suffered significantly during these tests. Overall, the clean coal yield varied from 12.0% to 89.7% while the energy recovery and product ash varied from 13.2% to 96.4% and 1.81 lb ash/MBtu to 3.25 lb ash/MBtu, respectively.

Development of Regression Equations (Indiana VII Coal) - Data from PDU Flotation Module parametric testing was compiled and multiple regression models (equations) were generated. Forward stepwise regression produced equations which link output variables such as yield and clean coal quality to input variables such as feed rate, wash water rate, air rate, collector addition, and frother addition.

The Coefficient of Determination (R^2) and Adjusted Coefficient of Determination (Adjusted R^2) both indicated that the each equation fits the data quite well. In addition, the t-statistic, which indicates the relative importance of each independent variable to

the response equation, revealed that the most important variable that affects yield and product ash is frother dosage and air rate, respectively.

Optimization of Flotation Column (Indiana VII Coal) - The previously developed equations were used to determine optimum Microcel™ setpoints needed to achieve the process development goals of 2 lb ash/MBtu and over 80% energy recovery. A total of four tests were performed. The product quality goal of 2 lb ash/MBtu was not achieved. It is suspected that a buildup of frother in the clarified water circuit resulted in higher recovery of unwanted middlings material which in turn increased the clean coal yield and ash content. The frother buildup was visible as white foam on the clarified water tank surface and also at screen sprays.

Extended Production Run of PDU Flotation Module (Indiana VII Coal) - An extended production run of the PDU Flotation Module was successfully completed during the week of July 22, 1996. Like the Taggart production run, a failed belt splice was the only operational difficulty. Due to the extremely poor filtering characteristics of this coal, 16 hours each day was dedicated to operation of the PDU while the remaining 8 hours were used for filtering accumulated clean coal slurry. Overall, 154,170 pounds of coal (77 tons) were processed in the PDU Flotation with a yield of 75.2%, energy recovery of 82.0% , and clean coal quality of 2.33 lb ash/MBtu.

Disposal of Clean Coal Product (Indiana VII Coal) - Continued communications with Penn State University (PSU) indicated that they were also interested in procuring the Indiana VII clean coal produced during the extended production. Due to its poor shipping characteristics, only one 2,000 pound super sack of Indiana VII was transported to Penn State. The remaining clean coal product was disposed at a local landfill.

Hiawatha Coal

The testing of the Hiawatha coal in the PDU Flotation Module commenced in August, 1996 and was concluded by September, 1996. The operation of the PDU with Hiawatha coal was very successful with project goals achieved on numerous occasions.

Laboratory Release Analysis (Hiawatha Coal) - Because the Hiawatha coal was not evaluated in the 12-inch Microcel™ during Subtask 4.4 (Hiawatha replaced Sunnyside), no data was available for indicating expected performance. As a result, to better define the theoretical grade-yield curves associated with different feed size distributions, laboratory release analysis test work was performed on the Hiawatha coal. Two Microcel™ feed slurries, one having d_{80} of 54 microns and a second with d_{80} of 49 microns, were evaluated.

An analysis of the data showed that though the quality goal of 2.00 lb ash/MBtu can be achieved at both particle size distributions, there is a subtle change in the flotation characteristics when the d_{80} is reduced from 54 microns to 49 microns. At a product quality value of 2.00 lb ash/MBtu, the yield improves from about 92% to 93% while the

energy recovery rises from 98% to about 99%. However, at a product quality of 1.90 lb ash/MBtu, the yield increases from 89% to 92% while the energy recovery improves from about 94% to 98%.

Testing and Optimization of Grinding Circuit (Hiawatha Coal) - The testing was performed to determine the optimum grinding circuit arrangement needed to achieve adequate mineral liberation for the Hiawatha coal. Release analysis of the Hiawatha coal suggested that the product ash quality goal of 2.00 lb/MBtu can be achieved with size distributions having d_{80} values of 54 and 49 microns, respectively. However, because the release analysis indicated improved yield and energy recovery at a d_{80} of 49 microns, this value was targeted. Based on the similar size distribution needed in for the Taggart coal, the 49 micron d_{80} target was easily achieved.

Parametric Testing of Microcel™ Flotation Column (Hiawatha Coal) - A test matrix was established to determine the effects of independent variables such as air rate, % solids, feed rate, wash water, and reagent dosage on response variables such as product ash and yield. Overall, the clean coal yield varied from 12.3% to 94.0% while the energy recovery and product ash varied from 13.8% to 98.7% and 1.43 lb ash/MBtu to 2.87 lb ash/MBtu, respectively. The Microcel™ feed d_{80} values averaged 50 microns with a standard deviation of 3 microns.

Development of Regression Equations (Hiawatha Coal) - Data from PDU Flotation Module parametric testing was compiled and multiple regression models (equations) were generated. Forward stepwise regression produced equations which link output variables such as yield and clean coal quality to input variables such as feed rate, wash water rate, air rate, collector addition, and frother addition.

The Coefficient of Determination (R^2) and Adjusted Coefficient of Determination (Adjusted R^2) both indicated that the each equation fits the data quite well. In addition, the t-statistic, which indicates the relative importance of each independent variable to the response equation, shows that the most important controllable variables that affect clean coal ash are Recirculation Rate (affects bubble size) and Wash Water. However, the variables that have the most significant impact on yield are Feed Ash % and Frother Dosage.

Optimization of Flotation Column (Hiawatha Coal) - The previously developed equations were used to determine optimum Microcel™ setpoints needed to achieve the process development goals of 2 lb ash/MBtu and over 80% energy recovery. A total of eight tests were performed and the overall quality goal of 2 lb ash/MBtu was achieved during two tests.

Extended Production Run of PDU Flotation Module (Hiawatha Coal) - An extended production run of the PDU Flotation Module on Hiawatha coal was successfully completed during the week of September 26, 1996. The PDU operated 72 continuous hours without interruption. Overall, 310,270 pounds of coal (155 tons) were processed

in the PDU Flotation with a yield of 81.8%, energy recovery of 88.0% , and clean coal quality of 1.89 lb ash/MBtu.

Disposal of Clean Coal Product (Hiawatha Coal) - Continued communications with Penn State University (PSU) indicated that they were interested in procuring the Hiawatha clean coal produced during the extended production. Overall, 131 bags (supersacks) were produced during the Hiawatha production run. Of this amount, 44 bags were shipped to Penn State with the remainder disposed at a local landfill.

Clean Coal Ash Properties

Hazen Research Inc., Golden, CO, determined the ash chemistry and fusion properties of feed and clean coal samples from the extended PDU production runs utilizing the Taggart, Indiana VII, and Hiawatha coals. It was found that the PDU Microcel™ flotation column cleaning consistently increased the base/acid ratio of the ash and decreased the silica/alumina ratio. The overall results were substantial declines in the reducing atmosphere fusion temperatures of the ash in the Taggart and Indiana VII coals, and a smaller decline in the fusion temperatures of the ash in the Hiawatha coal. Except for titania, and iron oxide in the case of the Taggart coal, the concentrations of the ash constituents were significantly reduced on a heating value (lb/MBtu) basis, by advanced flotation cleaning in the PDU.

Toxic Trace Elements Reduction

Samples of the crushed feed coal, ground feed coal, clean coal, and fine refuse from the extended PDU production runs utilizing the Taggart, Indiana VII, and Hiawatha coals were submitted to Huffman Laboratories, Golden, CO, for determination of the concentrations of twelve toxic trace elements. The toxic trace elements were antimony, arsenic, beryllium, cadmium, chromium, cobalt, lead, manganese, mercury, nickel, selenium, and chlorine.

The testwork indicated that there were substantial reductions, over 25% on a heating value basis, in the residual concentrations of arsenic, lead, and chlorine for all three as-received test coals. The reduction in the concentrations of beryllium, cadmium, chromium, cobalt, manganese, mercury, nickel, and selenium varied from coal to coal. Flotation did not appear to reduce the heating value basis concentration of antimony in any of the coals.

Reduction in trace-element concentrations from the amounts in the parent Taggart and Indiana VII ROM coals was greater on a heating value basis than the reduction from the amounts in the as-received test coals. The residual concentrations of arsenic, beryllium, cadmium, chromium, cobalt, lead, manganese, mercury, and selenium in the two clean coals were especially lower than their concentrations in the two ROM coals. Mixed results were seen for nickel and chlorine, and the concentration of antimony did not appear to have been reduced at all by the combination of preparation plant cleaning and column flotation. Except for chlorine, the rejection of the toxic trace elements from

the Taggart and Indiana VII ROM coals was greater than their rejection from the Hiawatha test coal even though the Hiawatha coal had not been washed prior to flotation in the PDU.

Microcel™ Scale-up Testwork

To better understand and determine the similitude between the 12-inch Microcel™ unit and the 6-foot Microcel™ unit, comparative tests were conducted on the 12-inch unit at conditions similar to those used in production runs and parametric testing. Testwork performed on all three test coals indicated that the 12-inch Microcel™ unit consistently produced clean coal products with better quality (lower ash) but at lower yields. The reasons for the variance in performance may be attributable to the following:

- Bubbles generated in the 12-inch column were considerably larger (2 mm) than those in the 6-foot column (1 mm). Large bubbles typically result in a lower carrying capacity (low yield) and more selectivity (higher quality) than smaller bubbles. Investigations into this difference revealed that the recirculation velocity through the 12-inch unit's in-line mixer was lower than that of the 6-foot unit.
- The retention time of the 12-inch column is only 73% of the 6-foot column (9.0 minutes versus 12.4 minutes). This low retention time may have resulted in the rejection of desired middlings to the tailings stream. Had the retention time been longer (geometry of 12-inch column similar to 6-foot column) the middlings might have reported to the clean coal stream increasing the yield and product ash.

Lessons Learned

Based on the testwork and operation of the PDU Flotation Module, the following general lessons were learned:

- Feed coal should be stored in a silo for protection from the elements. Coal left uncovered may result in material handling problems due to freezing or sticking at transfer points. These problems were particularly noticeable with the Indiana VII coal.
- Sumps should be designed with enough capacity that small changes in volume do not produce large fluctuations in level readings. Specifically, sumps and tanks should favor additional width with less height. PDU sumps showed widely fluctuating levels due to small width dimensions.
- Sumps should also have enough capacity to absorb process upsets without causing downtime.

- Proposed ball mill charges should be reviewed for proper loading and ball size. PDU ball mills were improperly charged (initially) which resulted in inefficient grinding and premature ball wear.
- Ball mill circuits should be designed for feed streams of 40% - 50% solids (by weight). PDU circuits initially operated at about 25% solids which compounded the problems of inefficient grinding and premature ball wear.
- Ball mills should only treat the size fraction that need grinding. Grinding the entire feed stream, as in the PDU, increases mill size requirements as well as operating costs. Size classification equipment, such as screens or cyclones, should be used as needed.
- Ball mill discharge magnets should be included in future designs for removal of grinding media degradation. Fine iron from the ball mill grinding media clogged pumps and cyclone apexes in the PDU.
- Baffles should be used in all agitated tanks. Many PDU tanks were initially designed without baffles. This resulted in vortexing, pump cavitation, and inaccurate sump level readings. The problem was corrected with the addition of baffles to the tanks.
- Nuclear density gauges should monitor only pipes that are free of air bubbles. The PDU density gauge provided radical and inaccurate fluctuations in density due to entrained air bubbles. The solution to this problem is the simple deaeration of slurry before entering the nuclear gauge. This can be accomplished with a deaeration tank upstream of the feed pump.
- Microcel™ flotation column wash water lines should be plumbed with the option of using fresh water. Though this option may not be used with high frequency, it allows the plant to operate at times when clarified water may cause contamination of the clean coal product.
- The Microcel™ flotation column should be designed with a fill water port (6 - 8 inches in diameter) for fast filling resulting in minimal downtime.
- Clean coal product sumps should be located immediately adjacent to the Microcel™ flotation column with large vertical pipe feeds. Sumps initially used in the PDU were located over 30 feet from the Microcel™ unit and were considerably undersized. The problems caused by this oversight included pipe plugging and unwanted downtime.
- Microcel™ column interface levels should be monitored with a ball float. This monitoring method proved to be the most reliable in such a harsh environment. Continuous data acquisition should be considered with such a unit.
- A variable speed recirculation pump proved to be invaluable to the proper operation of the Microcel™ column. The ability to adjust pump speed provides tremendous flexibility in varying bubble size and grade / yield.
- Dewatering equipment should be designed specifically for the intended use. The filters utilized in the project were used in a prior DOE effort, and as such,

were not a perfect match for the PDU application. The result was low filtering capacity and unscheduled downtime.

CONCLUSIONS AND RECOMMENDATIONS

The work completed in this study has provided considerable insight to the scale-up, design, operation, and performance of flotation columns (and related unit operations) as well as the need for further research in this area. A summary of relevant conclusions and recommendations follows.

Conclusions

Success of Program

The work and results related to this project should be considered entirely successful. The 2 tph flotation module was operated from January, 1996 through September, 1996 processing over one thousand tons of the Taggart, Indiana VII, and Hiawatha coals. Parametric testing was performed on each test coal followed by optimization testwork and a round-the-clock production run. A substantial amount of each coal's clean product was transported to Penn State University for combustion testing. Overall, the Taggart coal was cleaned to 1 lb ash/MBtu while the Indiana VII and Hiawatha coals were cleaned to 2 lb ash/MBtu. Not only were the project goals achieved, the process equipment performed extremely well in terms of reliability, control, and repeatability of results. A commercial plant cost study performed by Bechtel, estimated the cost of production for premium quality coal water slurry fuel to be \$2.15/MBtu which met the overall project goal.

Operation and Performance of Microcel™ Flotation Column

The operation and performance of the Microcel™ flotation column was very successful. Not only was the unit simple for the technicians to operate and maintain, it was easily capable of producing premium quality fuel. Overall, the unit could reach steady state within 20 minutes and maintain production levels with little variance. The bubble generation system proved to be extremely reliable with no unplanned downtime. The wash water system also performed reliably with only a small amount of maintenance needed to clean the discharge orifices. Extended production runs indicated that the Microcel™ flotation column is a dependable and cost effective means of cleaning coal to high quality levels.

Important Process Variables

Testing of the three coals in the PDU flotation module indicated that several process variables were important to proper operation. The most important variables and their effect on each coal's performance in the Microcel™, are discussed below:

Frother Dosage - This variable was found to have the most significant impact on yield for all three coals. It was also found to be very closely linked to product ash for the Taggart coal.

Air Rate - This variable was found to have the greatest impact on product ash for the Indiana VII coal. Specifically, increases in air rate increased the product ash.

Wash Water - The ability of this parameter to remove entrained mineral matter from clean coal froth resulted in a very large impact on product ash for the Hiawatha coal.

Recirculation Rate - The Hiawatha coal product ash was also greatly influenced by the recirculation rate. Variations in this parameter resulted in highly selective bubble size fluctuations.

Test Coal Grind Size

Testing performed in this program has indicated the size distribution needed for adequate mineral liberation in each coal. Taggart and Hiawatha coals have very similar liberation requirements while those for the Indiana VII coal are much more aggressive. Each test coal and corresponding d_{80} value is summarized below.

Coal	d_{80} Value	Ash - lb/MBtu	Sulfur - lb/MBtu	Yield %	Energy Recovery %
Taggart	51	0.99	0.46	94.4	96.9
Indiana VII	22	2.07	0.42	65.8	71.9
Hiawatha	51	1.89	0.44	81.8	88.0

It should be noted that the theoretical yield (67%) and energy recovery (73%) for Indiana VII coal could not be achieved due to excess frother in the closed water loop. Additional testing may allow these targets to be achieved.

Scale-up Criteria

Testing has determined that many criteria affect the scale-up similitude between the 1-foot and 6-foot columns. For all test coals, similar operating conditions in each column produced varying results. The 1-foot column consistently produced clean coal products with lower ash and yield values than the 6-foot unit. The difference in operation was attributed to differences in recirculation line velocity (which affects bubble size) and retention time. The lower recirculation rate of the 1-foot column produced

bubbles that were visibly larger than the 6-foot column. These larger bubbles characteristically produce lower yields and high quality products. In addition, the retention time of the 1-foot column, though coal dependent, was significantly smaller than that of the 6-foot column.

It was found, however, that the clean coal carrying capacity (lb/hr/ft²) values of the 1-foot and 6-foot units were similar. These values are shown below:

Microcel™ Size	Taggart Coal	Indiana VII Coal	Hiawatha Coal
1-Foot	129	74	116
6-Foot	127	86	125

Ash Property Changes

It was found that the PDU Microcel™ flotation consistently increased the base/acid ratio of the ash and decreased the silica/alumina ratio. The overall results were substantial declines in the reducing atmosphere fusion temperatures of the ash in the Taggart and Indiana VII coals, and a smaller decline in the fusion temperatures of the ash in the Hiawatha coal. Except for titania, and iron oxide in the case of the Taggart coal, the concentrations of the ash constituents were significantly reduced on a heating value (lb/MBtu) basis, by advanced flotation cleaning in the PDU.

Toxic Trace Element Removal

There were substantial reductions, over 25% on a heating value basis, in the residual concentrations of arsenic, lead, and chlorine for all three as-received test coals. The reduction in the concentrations of beryllium, cadmium, chromium, cobalt, manganese, mercury, nickel, and selenium varied from coal to coal. Flotation did not appear to reduce the heating value basis concentration of antimony in any of the coals.

Reduction in trace-element concentrations from the amounts in the parent Taggart and Indiana VII ROM coals was greater on a heating value basis than the reduction from the amounts in the as-received test coals. The residual concentrations of arsenic, beryllium, cadmium, chromium, cobalt, lead, manganese, mercury, and selenium in the two clean coals were especially lower than their concentrations in the two ROM coals. Mixed results were seen for nickel and chlorine, and the concentration of antimony did not appear to have been reduced at all by the combination of preparation plant cleaning and column flotation. Except for chlorine, the rejection of the toxic trace elements from the Taggart and Indiana VII ROM coals was greater than their rejection from the Hiawatha test coal even though the Hiawatha coal had not been washed prior to flotation in the PDU.

RECOMMENDATIONS

Recommendations for Design of Commercial Plant

The design of any commercial column flotation application should be based on sound scale-up data. Designers should use as large a flotation column as possible with the ability to vary the retention time through column height. A six-foot unit would prove very useful for such an endeavor. However, a three foot unit should be considered secondary with a 1-foot column as a final choice. In addition, it is imperative that the test column be equipped with a variable speed pump for bubble size control. The inability of the 12-inch and 6-foot columns to produce similar results was attributed to variances in bubble size and retention time.

Designers should also give consideration to maintenance requirements of the flotation column - specifically the bubble generation system. The simple design of the Microcel™ column provides almost worry-free operation with little maintenance. As a result, Microcel™ flotation columns are currently recommended for commercial applications.

In addition, design engineers should be mindful of the process control scheme developed for the flotation column. Because many different parameters affect the performance of the column (frother dosage, collector dosage, air rate, wash water rate, recirculation rate), careful control of these parameters is necessary for consistent product yield and quality. As a result, instrumentation and control equipment are vital and highly recommended.

The operation and performance of the Microcel™ flotation column is very predictable when feedstock characteristics are known and operating conditions are continually monitored and controlled by a computerized control and data acquisition system (CDAS). Though the resources that are needed to generate this performance data may be tedious and somewhat expensive, the resulting data is invaluable. Not only can the information be used to predict column performance, if used by a trained professional, it can be used to optimize performance for maximum economic benefit and return on investment (ROI). This optimization step was proven during this testwork and is recommended for future commercial and near-term applications.

Recommendations for Future R&D Work

Each year, hundreds of thousands of recoverable tons of fine coal are lost to refuse disposal. This may be the result of poor flotation cell / column performance in an existing preparation plant or even the lack of an economical fine coal cleaning process itself. If methods for optimizing existing flotation columns could be developed, considerable economic benefits would be realized by United States coal producers. Optimization of column flotation has practical applications with real-world benefits. The ability to optimize the performance of a commercially installed flotation column would result in tremendous economic benefits to domestic mining companies. As a result of

this study, it is recommended that future research in the areas of near-term flotation column performance optimization be explored.

INTRODUCTION

The goal of this project is the engineering development of advanced column flotation and selective agglomeration technologies for premium fuel applications. Development of these technologies is an important step in the Department of Energy (DOE) program to show that an ultra-clean coal-water slurry fuel (CWF) can be produced from selected United States coals and that this fuel will be a cost-effective replacement for a portion of the oil and natural gas burned by electric utility and industrial boilers in this country, as well as for advanced combustors currently under development. Capturing even a relatively small fraction of the total utility and industrial oil-fired boiler fuel market would have a significant impact on domestic coal production and reduce national dependence on petroleum fuels. Significant potential export markets also exist in Europe and the Pacific Rim for cost-effective premium fuels prepared from ultra-clean coal.

The replacement of oil and natural gas with CWF can only be realized if retrofit costs and boiler derating are kept to a minimum. Also, retrofit boiler emissions must be compatible with national clean air goals. These concerns establish the specifications for the ash and sulfur levels and combustion properties of ultra-clean coal.

Two advanced coal cleaning technologies were evaluated during this study - selective agglomeration and column flotation. The scope of the project involved the development of laboratory and bench-scale processes as well as the design and operation of a 2 tph pilot-scale plant. The purpose of this report is to present the findings of this study as well as any conclusions and recommendations for the advanced column flotation technology. This multi-year cost-share contract started on October 1, 1992, and is scheduled for completion by September 1997.

SPECIFIC OBJECTIVES OF THE PROJECT

The three main objectives of this project are discussed below.

The primary objective was to develop the design base for commercial prototype advanced fine coal cleaning facilities capable of producing ultra-clean coals suitable for conversion to stable, highly loaded coal-water slurry fuels. These slurry fuels should contain less than 2 lb ash/MBtu HHV (860 grams ash/gigajoule) and preferably less than 1 lb ash/MBtu HHV (430 grams ash/gigajoule), and less than 0.6 lb sulfur/MBtu HHV (258 grams sulfur/gigajoule). The advanced fine coal cleaning technologies to be employed are advanced column froth flotation and selective agglomeration. Operating conditions during the advanced cleaning processes should recover at least 80 percent of the heating value in run-of-mine source coals at an annualized cost of less than \$2.50/MBtu (\$2.37/gigajoule), including the cost of the raw coal.

A secondary objective of the work was to develop the design base for near-term commercial applications of these advanced fine coal cleaning technologies. These applications should be suitable for integration into new or existing coal preparation

plants for the purpose of economically and efficiently processing minus 28-mesh coal fines. The design base also included the auxiliary systems required to yield a shippable, marketable product such as a dry clean coal product.

A third objective of the work was to determine the distribution of toxic trace elements between clean coal product and refuse during the cleaning of various coals by advanced froth flotation and selective agglomeration technologies. Twelve toxic trace elements have been targeted. They are antimony, arsenic, beryllium, cadmium, chromium, cobalt, lead, manganese, mercury, nickel, selenium, and chlorine. The results show the potential for removing these toxic trace elements from coal by advanced physical cleaning.

APPROACH

A team headed by Amax Research & Development Center (Amax R&D) was formed to accomplish the project objectives. Figure 1 shows the project organization chart. Entech Global, Inc. is managing the project for Amax R&D (now part of Cyprus Amax Minerals Company) and performed laboratory research and bench-scale testing. Entech Global is also responsible for the operation and evaluation of the 2 t/hr process development unit (PDU). Cyprus Amax Coal Company provided operating and business perspective, the site for the near-term testing, and some of the coals used in the program. Bechtel Corporation provided engineering and design capabilities, and the operating experience it gained while managing similar proof-of-concept projects for DOE. The Center for Applied Energy Research (CAER) at the University of Kentucky and the Center for Coal and Mineral Processing (CCMP) at the Virginia Polytechnic Institute and State University provided research and operating experience in the column flotation area. Arcanum Corporation provided similar experience in the selective agglomeration area. Dr. Douglas Keller of Syracuse University served as a consultant in the area of selective agglomeration and Dr. John Dooher of Adelphi University served as a consultant in the area of coal-water slurry formulation. Robert Reynouard was retained as a consultant to help with electrical and instrumentation systems in the PDU, which was built by TIC and Mech El, Inc., two Colorado based construction companies.

The overall engineering development effort was divided into four phases with specific activities as discussed below. As shown in Table 1, Work Breakdown Structure, the four phases of the project were further divided into tasks and subtasks, with specific objectives which may be inferred from their titles. Figure 2 shows the project schedule.

Phase I

Phase I encompassed preparation of a detailed Project Work Plan, selection and acquisition of the test coals, and laboratory and bench-scale testing. The laboratory and bench-scale work determined the cleaning potential of the selected coals and established design parameters and operating guidelines for a 2 t/hr PDU containing both advanced column flotation and selective agglomeration modules. A conceptual

engineering design was prepared for a fully integrated and instrumented 2 t/hr PDU incorporating the features determined from the laboratory and bench-scale studies.

Additional activities during Phase I included:

- Production of ultra-clean coal test lots by bench-scale column flotation and selective agglomeration for end-use testing
- Determination of toxic trace element distribution during production of these test lots
- Evaluation of the rheological properties of slurry fuels prepared from ultra-clean coals
- Evaluation of methods for applying these advanced cleaning technologies to existing coal preparation plants in the near term

Phases II and III

Phases II and III covered the construction and operation of the 2 t/hr PDU. Phase II was for advanced column flotation while Phase III was for selective agglomeration. Process performance was optimized at the PDU-scale, and bulk lots of ultra-clean coal were produced by each process for each of the three test coals. The toxic trace element distribution was also determined during the production runs. The ultra-clean coal bulk lots were delivered to a DOE designated contractor (Pennsylvania State University) for end-use testing.

Phase IV

Phase IV activities include decommissioning of the PDU, restoration of the host site, and preparation of the final project report. These activities are currently in progress.

PURPOSE OF SUBTASK 8.5 TOPICAL REPORT

The purpose of Subtask 8.5 is to prepare a topical report that summarizes all the work performed on advanced column flotation under Tasks 2, 4, 7, and 8 for premium fuel applications. The work performed for near-term application is covered in Subtask 3.2 Topical Report. This report covers the following broad topics:

- Discussion and summary of all laboratory and bench scale findings.
- Discussion of the PDU Flotation Module design and layout.
- Discussion of the PDU Flotation Module startup and shakedown.
- Discussion of the PDU Flotation Module operation and results.
- Conclusions and recommendations.

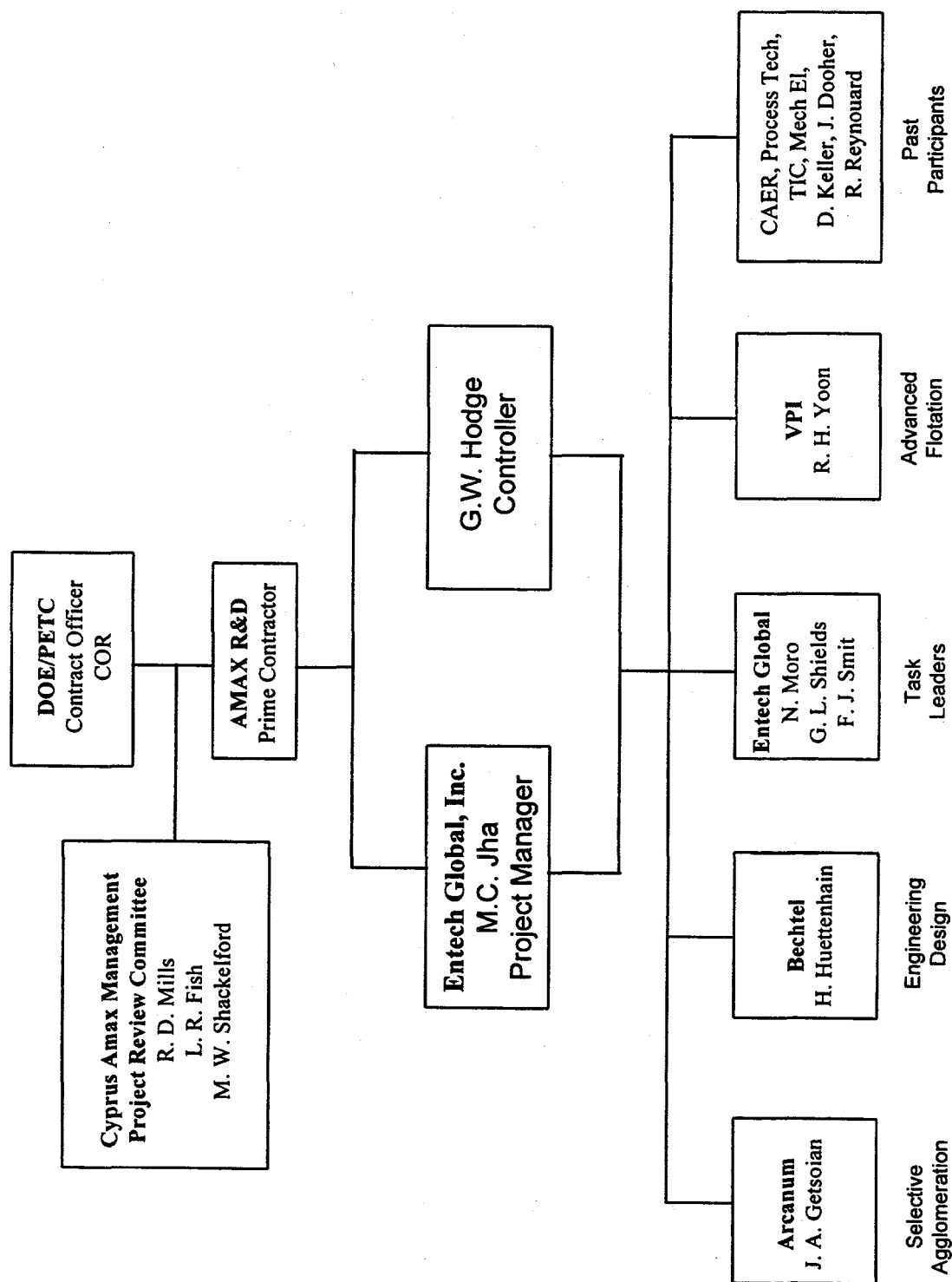


Figure 1. Project Management Organization Chart

Revised April 23, 1997

Table 1. Outline of Work Breakdown Structure

Phase I. Engineering Analysis and Laboratory and Bench-Scale R&D

- Task 1. Project Planning
 - Subtask 1.1. Project Work Plan
 - Subtask 1.2. Project Work Plan Revisions
- Task 2. Coal Selection and Procurement
 - Subtask 2.1. Coal Selection
 - Subtask 2.2. Coal Procurement, Precleaning and Storage
- Task 3. Development of Near-Term Applications
 - Subtask 3.1. Engineering Analyses
 - Subtask 3.2. Engineering Development
 - Subtask 3.3. Dewatering Studies
- Task 4. Engineering Development of Advanced Froth Flotation for Premium Fuels
 - Subtask 4.1. Grinding
 - Subtask 4.2. Process Optimization Research
 - Subtask 4.3. CWF Formulation Studies
 - Subtask 4.4. Bench-Scale Testing and Process Scale-up
 - Subtask 4.5. Conceptual Design of the PDU and Advanced Froth Flotation Module
- Task 5. Detailed Engineering Design of the PDU and Advanced Flotation Module
- Task 6. Selective Agglomeration Laboratory Research and Engineering Development for Premium Fuels
 - Subtask 6.1. Agglomeration Agent Selection
 - Subtask 6.2. Grinding
 - Subtask 6.3. Process Optimization Research
 - Subtask 6.4. CWF Formulation Studies
 - Subtask 6.5. Bench-Scale Testing and Process Scale-up
 - Subtask 6.6. Conceptual Design of the Selective Agglomeration Module
- Task 7. Detailed Engineering Design of the Selective Agglomeration Module

Phase II. PDU and Advanced Column Flotation Module Testing and Evaluation

- Task 8. PDU and Advanced Column Froth Flotation Module
 - Subtask 8.1. Coal Selection and Procurement
 - Subtask 8.2. Construction
 - Subtask 8.3. PDU and Advanced Coal Cleaning Module Shakedown and Test Plan
 - Subtask 8.4. PDU Operation and Clean Coal Production
 - Subtask 8.5. Froth Flotation Topical Report

Phase III. Selective Agglomeration Module Testing and Evaluation

- Task 9. Selective Agglomeration Module
 - Subtask 9.1. Construction
 - Subtask 9.2. Selective Agglomeration Module Shakedown and Test Plan
 - Subtask 9.3. Selective Agglomeration Module Operation and Clean Coal Production
 - Subtask 9.4. Selective Agglomeration Topical Report

Phase IV. PDU Final Disposition

- Task 10. Disposition of the PDU
 - Task 11. Project Final Report
-

Revised April 25, 1995

Subtask	1992							1993							1994												
	O	N	D	J	F	M	A	M	J	J	A	S	O	N	D	J	F	M	A	M	J	J	A	S	O	N	D
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27
1.1 Project Work Plan																											
1.2 Project Work Plan Revisions																											
2.1 Coal Selection																											
2.2 Procurement and Storage																											
3.1 NTA Engineering Analyses																											
3.2 NTA Engineering Development																											
3.3 Dewatering Studies																											
4.1 Grinding																											
4.2 Process Optimization Research																											
4.3 CWF Formulation Studies																											
4.4 AF Bench Testing, Scale-up																											
4.5 AF Conceptual Design PDU																											
5.0 Detailed Design PDU, AF Module																											
6.1 Agglomeration Agent Selection																											
6.2 Grinding																											
6.3 Process Optimization Research																											
6.4 CWF Formulation Studies																											
6.5 Sel. Aggl. Bench Testing, Scale-up																											
6.6 Concpt. Design Sel. Aggl. Module																											
7.0 Detailed Design Sel. Aggl. Module																											
8.1 Coal Procurement																											
8.2 PDU Construction																											
8.3 Shakedown, Test Plan																											
8.4 Operation and Production																											
8.5 AF Topical Report																											
9.1 Construction																											
9.2 Shakedown, Test Plan																											
9.3 Operation and Production																											
9.4 Selective Agglomeration Topical Report																											
10.0 PDU Decommissioning																											
11.0 Project Final Report																											

Revised July 17, 1997

Figure 2. Project Schedule

RESULTS AND DISCUSSION

The purpose of this section is to summarize the work performed on advanced column flotation under Tasks 2, 4, 7, and 8. The sections in this report follow a general chronological order of events as listed below:

1. Laboratory and Bench-Scale Work
 - a) Selection of Test Coals (Subtask 2.1)
 - b) Laboratory Grinding and Mineral Liberation Studies (Subtask 4.1)
 - c) Laboratory Flotation Testing (Subtask 4.2)
 - d) Bench Scale Column Flotation Testing (Subtask 4.4)
 - e) Final Coal Selection and Procurement (Subtask 8.1)
2. Design and Construction of the PDU Flotation Module
 - a) Conceptual Design of PDU Flotation Module (Subtask 4.5)
 - b) Detailed Design of PDU Flotation Module (Task 5)
 - c) Construction of PDU Flotation Module (Subtask 8.2)
3. Process and Plant Description
 - a) Area 100 - Raw Coal Handling
 - b) Area 100 - Grinding and Classification Circuits
 - c) Area 200 - Column Flotation Circuit
 - d) Area 400 - Dewatering Circuit
4. PDU Flotation Module - Operating Personnel / Staffing
5. PDU Flotation Module - Startup and Shakedown
6. PDU Flotation Module Operation and Testwork
 - a) Taggart Coal
 - b) Indiana VII Coal
 - c) Hiawatha Coal

7. Clean Coal Ash Properties
8. Toxic Trace Elements Reduction
9. Microcel™ Scale-up Testwork
10. Lessons Learned
11. Conclusions and Recommendations

LABORATORY AND BENCH SCALE WORK

The selection of test coals as well as discussion of related laboratory and bench-scale testing is discussed in the following sections.

Selection of Test Coals (Subtask 2.1)

Successful accomplishment of project objectives by both froth flotation and selective agglomeration processes is dependent on the selection of suitable source coals. Due to the widely varying quality and economic factors of United States coals, many could not be considered as a feedstock for this project. Accordingly, guidelines were established to evaluate a number of candidate coals and select six coals for use in this project. Overall, five bituminous coals and one low-rank coal were to be selected. All work was conducted under Subtask 2.1. Details of the selection procedure are provided in the Subtask 2.1 report [1] and only a brief summary is presented here. Guidelines included in the contract Statement of Work suggested the following specifications for coal selection:

1. Source Coal Properties

- a) Organic sulfur should be less than 258g/GJ (dry basis). This is approximately 0.88 percent for bituminous coal and 0.75 percent for low-rank coal.
- b) Ash minerals and pyrite must be sufficiently liberated by practical comminution methods.

2. Economic Factors - Coal Acquisition

- a) Selected coal must be obtained from actively mined seams with reserves in excess of 300 million tons.
- b) Sufficient quantities must be available for purchase from the same source to meet the needs of the project.
- c) Market value of the coal should be less than \$1.18 / GJ (\$1.25 / MBtu). This is approximately \$30 / Ton.

3. Economic Factors - Fuel Preparation

a) Because variations in coal quality may affect the preparation of premium CWF, potential coals should have the following characteristics:

- i) Low ash content;
- ii) Low total sulfur content;
- iii) Low organic sulfur content;
- iv) Liberation of ash bearing minerals and pyrite at coarse sizes;
- v) Low inherent moisture;
- vi) High Hardgrove grindability value;
- vii) High hydrophobicity;

In addition to these parameters, geographic diversity was also considered with at least one coal from each US coal mining region (eastern, midwestern, and western). The initial screening of coals from the Keystone Coal Mining Directory and the Amax Database generated a list of 17 candidate coals. These coals are listed in Table 2.

Table 2. - Candidate Coals for Preparation of Premium Fuels

<u>Coal Seam</u>	<u>State</u>	<u>County</u>	<u>Mine</u>	<u>Operator</u>
Upper Freeport	PA	Indiana	Helen	Helen Mining
Stockton / Mercer	WV	Kanawa	130 Mine	Amax - Cannelton
Winifrede	WV	Boone	Sandlick	Amax - Cannelton
Taggart	VA	Wise	Wentz	Westmoreland
Hazard 4A / 5A	KY	Knott	KY Prince	Roaring Creek
Elkhorn No. 3	KY	Pike	Chapperal	Costain
No. 2 Gas	WV	Wyoming	N/A	N/A
No. 2 Gas	WV	Boone	N/A	N/A
Indiana VII	IN	Sullivan	Minnehaha	Amax - Midwest
Illinois No. 5	IL	Wabash	Wabash	Amax - Midwest
Maxwell	CO	Las Animas	Golden Eagle	Basin Resources
O'Conner	UT	Carbon	Skyline	Utah Fuels
Sunnyside	UT	Carbon	Sunnyside	Sunnyside
Wyodak	WY	Campbell	Belle Ayr	Amax - West
Dietz	MT	Big Horn	Spring Creek	Nerco
Rosebud	MT	Rosebud	Rosebud	Western Energy
Lower Smith	WY	Campbell	Eagle Butte	Amax - West

These candidate coals were then subjected to the following evaluations:

- Proximate Analysis - Moisture, Ash, Volatile Matter, and Fixed Carbon;
- Sulfur Forms;
- Heating Value - Btu / lb;
- Equilibrium Moisture;
- Hardgrove Grindability;
- Coarse Coal Liberation - float / sink at SG of 1.6 and 1.9 on 100M x 0 sample;
- Fine Coal Liberation - float / sink at SG of 1.6 and 1.9 on 325M x 0 sample;
- Supplemental Amenability Testing - Flotation and Agglomeration testing on 20 μ m x 0 sample.

A coal selection matrix was then established for ranking each coal according to the previously mentioned parameters and test evaluations. The selection matrix is presented in detail in the Subtask 2.1 Topical Report [1]. They are also included in a published paper [2].

As a result of this evaluation, five bituminous and one low-rank coal was selected for testing in Phase I of the project. Each bituminous coal had the characteristics for successful production of premium fuels. The selected test coals are listed below:

- Taggart Coal - This was the highest ranking coal which also performed very well in amenability testing.
- Sunnyside Coal - This coal compiled a very high score and also performed very well in amenability testing.
- Indiana VII Coal - This coal contains less sulfur than most midwestern coals. Though it scored low, the coal was readily available for testwork since the Minnehaha mine was owned by Amax Coal.
- Winifrede Coal - Winifrede coal is very typical of the coal produced in West Virginia. It was also readily available since the source mine was also owned by Amax Coal.
- Elkhorn No. 3 Coal - This coal, which received a high score, is representative of the coal produced in eastern Kentucky.
- Dietz Coal - Dietz coal was recommended as the single low-rank selection. Though it compiled a low score, it responded better than other low-rank coals to amenability testing.

Each test coal was then subjected to laboratory scale liberation and flotation testwork.

The six test coals selected through Subtask 2.1 were subjected to laboratory mineral liberation and flotation testwork. The liberation testwork was conducted under Subtask 4.1 while the flotation work was completed in Subtask 4.2.

Laboratory Grinding and Mineral Liberation Studies (Subtask 4.1)

The objective of Subtask 4.1 was to study the grinding and mineral liberation characteristics of five of the six test coals. The Dietz coal (subbituminous), was not tested due to the inability to develop a flotation scheme for a subbituminous coal. Overall, the specific goals of this testwork were:

- Determine the grind size required to achieve the needed mineral liberation for premium fuels use.
- Determine the most economical grinding circuit configuration for premium fuels production in the advanced column flotation PDU.
- Determine the grinding circuit operating parameters for the advanced column flotation PDU.
- Prepare ground slurries for laboratory flotation research and optimization.
- Determine capacities of existing Amax R&D grinding equipment.

Detailed information on these objectives can be found in the topical report [3]. Overall the objectives were achieved by parametric laboratory and bench scale testing of various grinding configurations at variable rates (100 kg/hr to 500 kg/hr). Ash-bearing mineral and pyrite liberations were evaluated by washability testing and tree flotation analysis [3].

Twenty tons of each coal was acquired from the respective mine sites. The bituminous coals were commercially cleaned at the mine prior to shipment. The coals were then crushed to a topsize of ½ inch and stored in bulk bags.

The crushed coals were then stage ground in the existing pilot plant's 4-foot (1.2 m) ball mill and 40 liter stirred ball mill. The resulting slurries were then used to determine grinding circuit capacities as well as for mineral / pyrite liberation testwork.

Laboratory froth flotation and agglomeration testing were also performed on the resulting slurries. It was determined from this testwork that the grind sizes shown in Table 3 were sufficient for adequate mineral and pyrite liberation needed for premium fuels production:

Table 3. - Grind Sizes Needed for Mineral Liberation of Test Coals

Test Coal	d ₈₀ - microns
Taggart	45
Indiana VII	20
Sunnyside	45
Winifrede	11
Elkhorn No. 3	45

Overall, a closed-circuit grinding configuration was found to be more effective than an open-circuit configuration at providing greater capacity for a given grind size. The benefit was most evident when grinding to very fine particle sizes, such as those needed for the Indiana VII and Winifrede coals. The size classifier used in the closed-circuit configuration depended on the product size being produced. A solid bowl centrifuge was used for size separations less than 30 microns while a SWECO vibrating screen was used for size separations greater than 30 microns.

The grinding / liberation testwork performed under Subtask 4.1 resulted in grinding power requirements for various coals as shown in Table 4. This data formed the design basis of the PDU grinding circuit.

Table 4. - Estimated Grinding Energy Requirements for Test Coals

Test Coal	HGI	Target d_{80} (μm)	kW / tph	hp / stph
Taggart	52	45	96	116
Indiana VII	55	20	153	185
Sunnyside	54	45	91	110
Winifrede	47	11	341	413
Elkhorn #3	46	45	118	143

Observation of the data in Table 4 indicates that grinding power requirements varied greatly from coal to coal. This difference can be attributed to the varying target sizes (d_{80} values) needed for mineral and pyrite liberation.

The coal slurries produced during Subtask 4.1 were stored in 55 gallon drums for use in laboratory flotation studies performed at Amax R&D, CAER - University of Kentucky, and CCMP - Virginia Tech (Subtask 4.2).

Laboratory Flotation Testwork (Subtask 4.2)

The objective of the laboratory column flotation testwork performed under Subtask 4.2 was to determine the preferred column design and operating conditions for cleaning the six test coals. Detailed information can be found in the topical report [4]. In order to accomplish these objectives in a timely manner, the work was distributed among various project team members. CAER, at the University of Kentucky, investigated equipment design parameters and circuit comparisons with emphasis on the Elkhorn No. 3, Sunnyside, and Winifrede coals. Specifically, they examined the packed column and the effects of column height and aeration configuration on the performance of the Ken-Flote™ column. CCMP, at Virginia Tech, tested the performance of the Microcel™ flotation column with emphasis on the Indiana VII and Winifrede coals. Amax R&D evaluated the relative performance of various flotation reagents and the use of an external air sparger with emphasis on Elkhorn No. 3, Indiana VII, Taggart, Winifrede, and Dietz coals.

Testing revealed that all five bituminous coals responded quite well to laboratory flotation. Residual ash, sulfur, and heating value recovery targets were easily achieved with the Elkhorn No. 3, Taggart, and Sunnyside coals. In fact, testing of the Taggart coal indicated that product ash values of 1 lb ash/MBtu could be attained. Testing of the Indiana VII and Winifrede coals indicated that the product ash and heating value targets may need to be relaxed. The sixth test coal (low rank Dietz coal) did not respond well to laboratory flotation cell testwork, and as a result, was not subjected to column flotation testwork.

Overall, the performance of the Microcel™ and Ken-Flote™ units were very similar with superior performance over the packed column. As a result, it was suggested that both Microcel™ and Ken-Flote™ columns be evaluated at bench scale under Subtask 4.4.

With respect to the overall performance of each test coal in a flotation column, retention time and wash water flow rate appeared to be the most important variables. Though frother dosage was also a very important parameter, the optimal type or quantity could not be defined in this testwork. Collector requirements were very similar to conventional flotation cells with dosages around 1 lb/ton. Solids concentration had an impact on performance with low concentrations improving the flotation of minus 325 mesh coal. Column height, however, was not a critical factor affecting overall performance.

Bench Scale Column Flotation Testing (Subtask 4.4)

Based on the results of laboratory flotation testwork conducted under Subtask 4.2, five of the six original test coals were subjected to bench-scale column flotation testing in Subtask 4.4. The objective of this work was to verify that the performance and operating characteristics observed in laboratory testing can be scaled up to a continuous feed rate of 100 lb/hr. The response of each coal to cleaning in a 1-foot diameter Ken-Flote™ column and Microcel™ column was evaluated. The five coals and associated quality characteristics are shown in Table 5.

Bench Scale Column Flotation Equipment

Two separate flotation columns were evaluated under Subtask 4.4. The Ken-Flote™ and Microcel™ columns were both of steel construction, 12 inches in diameter, and approximately 14 feet tall. Each unit differed, however, in the manner in which air was delivered to the slurry (aeration system).

The Ken-Flote™ column was furnished with a Foam-Jet air sparger fitted with four porous metal plugs. Air and water were forced, under pressure, through the porous sparger openings. The tiny air bubbles generated by the spargers were injected into the base of the column for flotation.

The Microcel™ unit was fitted with a microbubble aeration system consisting of a centrifugal pump and an in-line mixer. Tailings slurry was pumped from the bottom of

the column through the in-line mixer and back into the column at a point located approximately 1-foot higher than the feed port. Compressed air was injected into the slurry upstream of the in-line mixer. The high velocity of the slurry in the mixer sheared the air into tiny bubbles for use in flotation.

A detailed description of both columns is presented in Subtask 4.4 Topical Report [5]

Parametric Testing of Candidate Coals and Column Performance

Each coal was subjected to numerous tests in both flotation columns. Several operating parameters were varied and their effect on performance monitored. The results of the testwork are summarized in Table 6.

Overall, a number of useful observations were made during the testwork.

- Product ash targets were met for all five coals tested in the 12-inch columns.
- There was an obvious trend showing that the unit capacity of the flotation columns increased as the particle size distribution of the feed slurry became coarser.
- Wash water requirements needed for ash rejection were lower for coarse size distributions.
- Reagent requirements for the Microcel™ flotation column were lower than those needed for the Ken-Flote™ unit.
- The Microcel™ unit typically had a greater feed capacity than the Ken-Flote™ unit.
- The Ken-Flote™ column incurred significantly more downtime and, as a result, required more maintenance than the Microcel™ unit.

Based on these findings, the Microcel™ flotation column was chosen for use in the 2 tph PDU production module as well as for near-term application at the Lady Dunn coal preparation plant.

Final Coal Selection and Procurement (Subtask 8.1)

Based on the performance of the six test coal in the laboratory and bench scale testwork, three coals were selected for use in the PDU Flotation Module. These coals and the reasons for their selection are summarized below. The details can be found in Subtask 8.1 Topical Report [6].

Table 5. - Properties of Coals Tested in Bench Scale Column Flotation

Attribute	Taggart		Indiana VII		Sunnyside		Winifrede		Elkhorn No. 3	
	A.R.*	Dry	A.R.*	Dry	A.R.*	Dry	A.R.*	Dry	A.R.*	Dry
Ash - %	2.01	2.07	7.50	9.25	4.78	5.11	7.89	8.42	5.62	6.04
Volatile Matter - %	35.35	36.46	28.14	34.70	34.85	37.29	31.79	33.95	33.45	35.98
Fixed Carbon - %	59.60	61.47	45.45	56.05	53.84	57.60	53.97	57.63	53.90	57.98
Moisture - %	3.05		18.91		6.53		6.35		7.03	
Sulfur - Total %	0.60	0.62	0.40	0.49	0.59	0.63	0.88	0.94	0.80	0.86
Sulfur - Pyritic %	0.05	0.05	0.12	0.15	0.07	0.07	0.14	0.15	0.16	0.17
Sulfur - Sulfate %	0.001	0.001	0.002	0.002	0.002	0.002	0.001	0.001	0.007	0.007
HHV - Btu/lb	14,829	15,296	10,924	13,472	13,378	14,313	12,957	13,836	13,138	14,059
HHV - GJ/t	34.48	35.56	25.40	31.32	31.10	33.28	30.13	32.17	30.55	32.69
Equil. Moisture - %	1.01		9.30		2.52		2.93		3.04	
HGI	52		55		54		47		46	
Density - kg/m ³		1,267		1,387		1,303		1,362		1,330
Sulfur - lb/MBtu	0.41		0.36		0.44		0.68		0.61	
Sulfur - g/GJ	174		156		189		292		263	
ROM HHV Recov %	94.3	94.3	90.5	90.5	90.1	90.1	89.5	89.5	94.6	94.6
ROM Carbon Recov %	89.0	89.0	90.7	90.7	90.0	90.0	90.7	90.7	97.7	97.7

*A.R. - As Received

Table 6. - Bench Scale Column Flotation Test Results

Parameter	Taggart	Elkhorn No. 3	Sunnyside	Indiana VII	Winifrede
FEED					
Feed Ash %	2.10	6.00	5.50	9.50	8.50
Feed Rate - tph/ft ²	0.17	0.18	0.12	0.05	0.03
Feed Slurry - % Solids	10	10	10	6	6
Feed Slurry - gpm/ft ²	6.6	7.2	4.7	3.6	0.9
Feed d ₈₀ - microns	104	104	70	24	12
CLEAN COAL PRODUCT					
Clean Coal Ash - %	1.37	2.75	2.74	2.82	2.91
Clean Coal Ash - lb/MBtu	0.90	1.90	1.90	2.00	2.00
Clean Coal Yield - %	94.2	86.7	92.0	74.0	74.9
Heating Value Recovery - %	95	90.0	95.0	80.0	80.0
Clean Coal Carry Capacity - tph/ft ²	0.16	0.16	0.11	0.04	0.02
Clean Coal Slurry - % Solids	17	16	20	12	6
TAILINGS					
Tailings Ash - %	14.0	27.2	37.2	28.5	25.2
Tailings Yield - %	5.8	13.3	8.0	26.0	25.1
Tailings Slurry - % Solids	0.50	1.19	0.58	0.85	0.46
Tailings Slurry - gpm/ft ²	7.8	8.2	6.6	6.6	5.8
CONDITIONS					
Wash Water - Gallon / Feed Ton	1,800	1,800	2,200	6,500	18,800
Wash Water - gpm/ft ²	4.8	4.8	4.0	4.3	6.3
Wash Water Velocity - ft/min	0.64	0.64	0.54	0.58	0.84
Bias Flow Ratio - %	35	30	56	73	80
Aeration Rate - scfm/ft ²	1.9	1.9	1.9	1.7	1.7
Superficial Air Flow - ft/min	1.9	1.9	1.9	1.7	1.7
Retention Time - minutes	11.5	11.0	13.6	13.6	13.4
Nominal Froth Depth - feet	2.5	2.5	2.5	2.5	2.5
Aerated Slurry Depth - feet	12	12	12	12	12
Frother Dosage - lb/ton	0.25	1.00	0.67	3.00	1.50
Collector Dosage - lb/ton	0.50	1.50	0.23	5.00	3.50

Taggart Coal

Taggart coal was the only test coal that could be cleaned to less than 1 lb ash/MBtu ash and less than 0.6 lb/MBtu sulfur while recovering 80 percent of the heating value (raw coal basis). Because Taggart coal is low in ash and sulfur, fine grinding was not necessary. A d_{80} of 60 microns was adequate. The coal responded very well to column flotation with yields expected to exceed 95 percent. Clean coal slurry prepared from Taggart coal approached 70 percent solids loading by weight. Overall, the \$2.50/MBtu production cost goal should be achieved.

Because of the high yield, excellent quality properties, low expected operating cost, and strategic mining location (eastern industrial belt), Taggart coal should prove to be an excellent feed coal for commercial production of premium fuels.

Indiana VII Coal

Indiana VII coal was recommended for use in the PDU flotation module for reasons quite opposite to that of Taggart. Specifically, Indiana VII was chosen to challenge the capabilities of the PDU flotation and selective agglomeration modules. Though the coal requires fine grinding for adequate mineral and sulfur liberation (d_{80} of 20 microns), testing indicated that the clean coal ash goal of 2.0 lb/MBtu can be achieved.

Besides the technical challenges of the Indiana VII coal, it was also selected for its good flotation and agglomeration properties (typical of a midwestern coal). This coal was also being used in another DOE program at Penn State to develop a coal based fuel to replace fuel oil at a Department of Defense facility in Indiana. In addition, Cyprus Amax Coal Company, a member of the project team, has active operations in this region and owned the Minnehaha mine that produces this coal.

Sunnyside Coal

Sunnyside coal from Utah was selected as the third and final coal for testing in the PDU flotation and agglomeration modules. It is a low-sulfur coal with the ability to easily meet the project quality goals after moderately fine grinding (d_{80} of 45 microns). In addition, a coal from the Utah / Colorado area could represent a dependable fuel source for the western United States. It should be noted that shortly before the PDU operation, Sunnyside coal became unavailable due to mine closure. Another coal (Hiawatha coal) from a nearby mine was identified as a suitable replacement for Sunnyside with similar performance, quality, and geographic considerations.

DESIGN AND CONSTRUCTION OF THE PDU FLOTATION MODULE

The design and construction of the PDU Flotation Module is discussed in the following sections.

Conceptual Design of PDU Flotation Module (Subtask 4.5)

The conceptual design of the PDU Flotation Module was a collaborative effort between Bechtel, Entech, CCMP, and CAER. Flow diagrams and equipment selection are presented in the Bechtel report [8]. Important issues are summarized below.

Selection of Flotation Column - Microcel™

The Microcel™ flotation column was selected for use in the PDU primarily due to its superior performance and ease of operation. Testing indicated that very high yield and quality values could be achieved. In addition, its bubble generation system is designed in a manner which minimizes maintenance and increases bubble generation flexibility.

Selection of Cyclone / Fine Coal Screens for Size Classification

Several discussions were held in regard to the use of a solid bowl centrifuge as a size classifier in the PDU grinding circuit. The advantage of such equipment is the ability to achieve fine size separations. The disadvantage is the high capital, operating, and maintenance costs associated with the unit. Further studies suggested that a cyclone / screen circuit could produce the needed size distribution with a significant savings in capital, operating, and maintenance costs. As a result, the decision was made to include this equipment into the PDU.

Selection of Fine Grinding Mill

Due to the increased liberation requirements of the Indiana VII coal (d_{80} of 20 microns) and the inability of conventional ball mills to achieve this product size analysis, a fine grinding mill was incorporated into the grinding circuit. Several fine grinding mills were evaluated for use in the PDU - a Tower mill, attritor mill, and horizontal bead mill. The horizontal bead mill, supplied by Netzsch, Inc., was selected due to its compact size and prior operating record in the coal processing industry.

Elimination of Rougher / Cleaner Flotation Circuit

Several discussions were held in regard to the use of a rougher / cleaner flotation circuit in the PDU. The advantage of such a circuit is the flexibility to produce various grades of clean coal product. The disadvantage is the high capital cost associated with the added equipment. Testwork indicated that a single stage Microcel unit would perform

as well as multiple stages. Ultimately, the decision was made to use a single Microcel™ flotation column and eliminate the proposed cleaner circuit.

Sizing of Microcel™ Flotation Column

Because the size requirements of a flotation column vary from coal to coal, a decision was made to size the single Microcel™ flotation column based on the test coal with average bench-scale performance characteristics. As a result, a 6-foot diameter unit was chosen based on the bench-scale performance of the Sunnyside coal. The column would be oversized for the Taggart coal and undersized for the Indiana VII coal. Adequate performance for these two coals would be achieved by feed rate variations.

Use of Existing DOE Filters

A decision was made to use a rotary drum filter as well as a pair of Netzsch's plate and frame filters utilized in a previous DOE project. Though the filters were not ideally suited for the steady state production capacities of each PDU test coal, the capital saving to the project was significant.

Selection of High Capacity Thickener

In an effort to reduce capital costs, operating costs, and space requirement, a decision was made to use a high capacity Enviro-Clear thickener in the PDU tailings circuit. The unit, which was installed inside the pilot plant, eliminated the high capital cost associated with a large static thickener (to be located outside) as well as operating and maintenance costs associated with cold weather protection.

Detailed Design of PDU Flotation Module (Task 5.0)

The detailed design of the PDU Flotation Module was performed by Bechtel Corporation of San Francisco, CA with support from Entech Global engineers under Task 5. Details of this work can be found in the 3-volume Design Package submitted to DOE [8].

All structural drawings as well as P&ID's were completed by Bechtel and issued for construction. Electrical drawings were issued by Control Technologies, Inc. and approved by Bechtel Corporation.

Entech Global managed the procurement of all instrumentation as well as new and refurbished capital equipment items used in the PDU Flotation Module. Altogether, 47 pieces of new equipment and 52 pieces of instrumentation / control equipment were purchased. Twelve pieces of existing Amax / DOE equipment were refurbished.

Construction of PDU Flotation Module (Subtask 8.2)

Construction of the PDU Flotation Module was executed under Subtask 8.2. Request for Quotation (RFQ) packages were issued for the PDU construction subcontract during the first quarter of 1995. Entech Global and Bechtel personnel collaborated to decide issues regarding work scope and components of the RFQ. Final copies of the RFQ which included project drawings were sent to the following construction companies:

1. Bateman Engineering, Inc. of Denver, Colorado
2. CLI Corporation of Pittsburgh, Pennsylvania
3. Farnham and Pfile Construction, Inc. of Belle Vernon, Pennsylvania
4. Kilborn International, Inc. of Englewood, Colorado
5. Lincoln Contracting Company of Stoystown, Pennsylvania
6. Lyntech, Inc. of Denver, Colorado
7. TIC of Steamboat Springs, Colorado

A site inspection and meeting for interested bidders was held on February 2, 1995. Only two construction companies attended this meeting. Those in attendance were CLI and TIC. A third company, Vector Services, who had been selected by CLI as a subcontractor approached Amax and requested that they be allowed to submit a bid independently. Their request was approved and a second site inspection and meeting was held on February 16, 1995.

The majority of each meeting was used to clarify questions and issues regarding the RFQ package. Each company submitted a bid estimate for construction of PDU - Phase I, on February 28, 1995. After careful evaluation, the subcontract for the construction of the PDU Flotation Module of the PDU was awarded to TIC - The Industrial Company, of Steamboat Springs, Colorado. An award meeting was conducted on March 15, 1995.

TIC mobilized onto the Amax R&D site March 27, 1995. TIC was responsible for the installation of all process equipment, instrumentation, structural steel, concrete, process piping, power systems, and control systems related to the operation of the PDU Flotation Module. Mechanical and electrical completion of the PDU was achieved on July 31, 1995, and August 31, 1995, respectively.

PROCESS AND PLANT DESCRIPTION

The PDU flotation module, is a pilot scale advanced coal cleaning facility which utilizes column flotation technology to remove unwanted mineral matter and its related impurities, such as sulfur and some toxic trace elements, from run-of-mine (ROM) or washed coals.

Like conventional froth flotation, which has been used for over 80 years, column flotation utilizes air bubbles and flotation reagents to separate the desired hydrophobic

coal particles from the unwanted hydrophilic mineral matter (tailings). The advantages of column flotation over conventional froth flotation, however, is its ability to process finer size particles to produce a cleaner coal while also improving clean coal recovery.

The following sections describe the advanced coal cleaning PDU and flotation module which was divided into several areas for design and operating purposes:

- Area 100 - Raw coal handling;
- Area 100 - Grinding and classification circuit;
- Area 200 - Column flotation circuit;
- Area 400 - Dewatering circuit;

It is important to note that Area 100 is comprised of several different unit operations and will be described in two separate sections. Area 300 is to be used for the selective agglomeration module in place of Area 200 column flotation.

Area 100 - Raw Coal Handling

An integral unit operation to any coal cleaning process is raw coal handling. Without a reliable delivery system, all downstream processes could experience downtime and/or process upsets, which adversely affect yield, quality, and operating costs.

All coals which were to be cleaned in the PDU flotation module are normal commercial products of coal mines (minus 2-inch coal). Taggart and Indiana VII coals were washed but Hiawatha was not. They were delivered in 100 ton rail cars to a coal yard located in north Denver. Here, the coal was unloaded and stored until needed at the PDU site. The coal was then transported by truck to Ralston Development Company located approximately five miles north of the Amax R&D facility. Here the coal was crushed to a top size of 1/2 inch and stored in covered bunkers. When needed, the coal was transported by truck to the PDU site where it was stored in a covered bunker.

A front end loader is used to dump the coal into a receiving hopper. A vibratory feeder located at the bottom of the hopper discharges the coal onto an elevating belt conveyor which transports the material to a 15 ton capacity feed bin. Here the coal is briefly stored prior to processing. A vibrating bin activator, located at the base of the storage bin, minimizes plugging while dumping the material onto a weigh belt feeder which meters the coal to the grinding circuit.

Area 100 - Grinding and Classification Circuits

The grinding and classification circuits are very important to the proper operation of the PDU flotation module. Because most of the undesired mineral matter associated with coal is actually disseminated throughout individual coal particles, the mineral matter must first be released or liberated from the coal before any separation can take place. This liberation is achieved by progressively reducing the particle size of the coal to a

point where the clean particles of coal can be effectively separated from the unwanted mineral matter particles. The PDU flotation module utilizes two ball mills in a series followed by a fine grinding bead mill to achieve this liberation.

Previous test work had revealed that adequate liberation occurs when grinding the coal to the following particle size distributions:

- Taggart coal - $d_{80} = 45$ microns
- Indiana VII coal - $d_{80} = 20$ microns
- Hiawatha coal - $d_{80} = 45$ microns

Figure 3 shows the flow scheme for Area 100. The coal stored in the feed bin is fed at a constant rate from the weigh belt feeder and dumped into a screw conveyor which transports the material to the primary ball mill for grinding. Clarified water is added to the coal prior to its entrance into the ball mill. The primary ball mill is charged with steel balls ranging in size from 3 to 0.5 inch. As the mill rotates, the balls rise along the interior wall to a point where they start to cascade down upon the body of the other balls and coal. This tumbling action reduces the size of the coal particles through a combination of impact and abrasion.

The finely ground coal and water exiting the primary ball is pumped to the secondary ball mill with a progressive cavity pump. The coal particles are further ground in an effort to achieve adequate liberation of the unwanted minerals.

At this point, the size of the particles must be evaluated to ensure that the desired top size / liberation has been obtained. The size evaluation or classification is performed in a series of classifying cyclones. The slurry which exits the secondary ball mill is pumped to these cyclones by a second progressive cavity pump. Two of the cyclones are 3-inch diameter while the remaining four cyclones are 2-inch diameter. The 3-inch diameter units are designed to size the Taggart and Hiawatha coals to 80% passing 45 microns while the 2-inch cyclones are designed to size the Indiana VII coal to 80% passing 20 microns. These finely sized particles exit through the top of the cyclones (vortex finder) while the coarse particles exit through the bottom of the units (apex). Because the optimum solids concentration of the cyclone feed stream is about 20%, additional water can be added, if needed.

The coarse cyclone underflow, which is considered larger than the required top size, must undergo additional size reduction to achieve the desired liberation. This is accomplished by grinding the material in a horizontal fine grinding bead mill. The cyclone underflow is delivered to the fine grinding mill by way of a feed sump and progressive cavity pump. The ground product exiting the mill is then combined with the secondary ball mill product before being sent to the classifying cyclones for a second size evaluation. Because the fine grinding mill is designed to produce a product size consisting of 98% less than 325 mesh (45 microns), its entire product stream should now report to the cyclone overflow.

It should be noted that while cyclones are good size separators, they are not perfect separators. Some fine coal particles, which ideally should report to the cyclone overflow stream, end up in the cyclone underflow. Similarly, some coarse particles, which should report to the cyclone underflow, can be found in the overflow stream. Any misplaced oversize material found in the cyclone overflow stream can cause operational and product quality problems. Because each coal has a defined top size designed to achieve optimum mineral liberation, it is important to ensure that this top size limitation is maintained.

To guarantee that the particle top size constraint is maintained, the cyclone overflow stream is sent to a pair of high frequency fine sizing screens. The Sisetech screens, which are very efficient size separators, assure that all oversize material is removed. The oversized screen overflow product is ultimately sent to one of the three grinding mills for additional liberation. This flow scheme provided the needed flexibility for each of the three test coals. The fine material, which passes through the sizing screens, is now considered fine enough to produce optimum mineral liberation. The screen underflow flows by gravity to the ground product sump from where it is pumped to the flotation circuit by means of a centrifugal pump. The speed of the ground product pump is adjusted automatically to maintain a constant level in the sump.

Area 200 - Column Flotation Circuit

Froth flotation is a complex method of separating finely ground minerals and coal from other undesired minerals. Coal flotation, however, will be the topic of discussion in this section. The upgrading / concentrating occurs by taking advantage of the different physico-chemical surface properties of coal and unwanted mineral matter. Generally, the process involves chemically pre-treating the feed slurry to a point where conditions are favorable for the attachment of the high grade coal particles to small rising air bubbles [9]. The bubbles float to the surface of the slurry where they form a stable froth. The clean coal particles (concentrate) are recovered by removing the froth while the unwanted mineral matter (tailings) is rejected through a discharge pipe located at the bottom of the flotation unit.

In almost all cases, coal flotation requires the addition of two chemical reagents:

- Collector -typically No. 2 fuel oil.
- Frother - typically a synthetic alcohol such as methyl isobutyl carbinol (MIBC).

The collector is used to render the surface of the coal particles hydrophobic (water hating). This improves the contact angle between the coal particle and air bubble making conditions favorable for bubble attachment. Without collectors, some coal particles form only negligible contact with the bubbles which makes flotation difficult.

Since most collectors have a charged (polar) group and a hydrocarbon (non-polar) group, they are considered to be heteropolar. When the collector attaches itself to a

coal particle, the collector molecules are oriented in a fashion where the hydrocarbon groups are pointed outward. The result is the formation of a hydrophobic film on the surface of the coal particle. This film allows the coal particle to readily attach to an air bubble and rise to the surface of the flotation unit [10].

Once the particles / bubbles reach the surface, a frother is used to temporarily stabilize the froth until the particles can be removed. If a frother is not used, the bubbles will burst and release the coal particles back into the slurry. It is important to note that only temporary froth stability is desired. Specifically, the froth should break down once it is withdrawn from the flotation unit. A froth that is too strong, is generally quite dilute in its solids content, difficult to pump, and will most likely affect the performance of downstream dewatering operations adversely.

The type of flotation machine chosen is very important since different units will produce different quality products. The most common type of conventional flotation machine is the sub-aeration unit produced by Wemco and Denver. Though most sub-aeration units are typically self aerating, some are supplied with pressurized air. The incoming air is directed to the base of the unit where specially designed impellers break the air into small bubbles. The impellers then disperse the bubbles into the mixing slurry.

Another type of flotation machine is a column flotation unit. Like conventional froth flotation, which has been used for over 80 years, column flotation utilizes air bubbles and flotation reagents to separate the desired hydrophobic coal particles from the unwanted hydrophilic mineral matter (tailings). The advantages of column flotation over conventional froth flotation, however, is its ability to process finer coal particles to produce a cleaner coal product while also improving clean coal recovery [11]. To understand why column flotation yields results superior to conventional flotation, one must understand how a flotation column functions.

There are currently several philosophies regarding the design of flotation columns. Most differences concern the method of bubble generation. As discussed before, laboratory and bench-scale test work had indicated that superior results are obtained with a Microcel™ flotation column. As a result, this unit was chosen for use in the PDU flotation module.

The Microcel™ flotation column technology was developed by Virginia Polytechnic Institute and State University of Blacksburg, Virginia. The unit, which is basically a long vertical cylinder, varies in size and diameter. The column which is installed in the PDU flotation module is 6 feet in diameter and almost 29 feet tall. Slurry enters the unit below the froth interface at a point approximately 8 feet below the overflow. The slurry moves downward through the column encountering a flow of rising air bubbles. The opposing flow of slurry and air promote efficient bubble contact which improves overall recovery. The rising bubbles and clean coal particles form a thick layer of froth (1 - 5 feet) at the top of the unit. A downward flow of water is introduced into the froth to wash away any unwanted mineral matter (clay particles) entrained in the froth. The net result is improved clean coal quality (ash rejection) and recovery.

A schematic process flow diagram of the PDU flotation circuit is shown in Figure 4. Air bubbles, ranging from 70 to 300 microns in diameter, are generated by shearing pressurized air through externally mounted static mixers. These bubbles usually coalesce and ultimately result in bubble sizes ranging from 500 to 1,000 microns. Frother is metered into a centrifugal pump which recirculates a portion of the slurry through a manifold and into four separate static mixers. Air, which is supplied by compressor, is introduced to the slurry prior to entering the static mixers. The air is then sheared by the blades of the static mixer into small microbubbles. The bubble-rich slurry flows into the flotation column where the bubbles are dispersed and rise to the top of the unit.

The slurry level in the Microcel™ is automatically controlled by increasing or restricting the flow of tailings exiting the unit. A sensitive pressure transducer indicates the level of froth and slurry in the column. If the level is higher or lower than the set point, a pneumatic valve located on the exiting tailings line opens and closes accordingly.

The Microcel™ unit installed in the PDU flotation module was sized to best accommodate the test work on all three coals. The flowsheet representing Area 200, which is shown in Figure 4, was designed to ensure continuous operation while generating meaningful test results. The following paragraphs describe the operation associated with Area 200.

Ground slurry is pumped to the Microcel™ feed sump from Area 100 by means of a variable speed centrifugal pump. Collector is added to the Microcel™ feed sump where the slurry is mixed by a radial agitator. The level of the feed sump is automatically controlled by varying the speed of the Microcel™ feed pump. The material which exits the feed sump is evaluated for proper solids concentration by a nuclear density gauge. If the solids concentration is greater than the desired value (typically 7.50% solids), dilution water is automatically added to the feed sump. Control of the feed solids concentration provides consistent operation and dependable separation results.

A variable speed centrifugal pump is used to generate air bubbles and recirculate a portion of the slurry stream. The separation which takes place produces a clean coal froth and a dilute tailings stream having solids concentrations of approximately 16% and 0.25%, respectively. The clean froth falls into a slurry storage tank from where it is pumped to one of two filter feed sumps. Water sprays on the launder can be used, if necessary, to break the froth and facilitate its transfer. The tailings exit the column to the final tailings sump from where it is pumped to the thickener by means of a level controlling centrifugal pump.

Area 400 - Dewatering Circuit

The dewatering circuit is very important to the continuous operation of the PDU flotation module. Dewatering, or solid-liquid separation, produces dry (relative) filter cake and water (filtrate). The filter cake is the product and the water (filtrate) is recycled and

used over and over again in the process. The dewatering circuit used in the PDU flotation module can be divided into the following two areas:

- Clean coal dewatering.
- Tailings dewatering.

Each product, clean coal and tailings, must be dewatered separately to ensure that no cross contamination occurs. A flowsheet of each area is shown separately in Figures 5 and 6, respectively.

The clean coal is dewatered in a circuit (Figure 5) which utilizes three filters. A WesTech vacuum drum filter is used as the primary filtration unit while two Netzsch pressure filter presses filter the remaining clean coal product. Clean coal which enters the drum filter feed sump can be stored for a short period of time before being pumped to the units. A centrifugal pump is used for transferring the clean coal slurry to the WesTech filter. The unit, which produces a cake continuously at a rate of approximately 2,000 lb/hr (dry solids), discharges the product into a supersack for storage or disposal. The remaining clean coal product is dewatered by a pair of Netzsch filters. The coal which is stored in the Netzsch filter feed sump can be pumped to either filter by a series of Netzsch piston pumps. The filter cake produced by these units is discharged onto a dedicated conveyor belt and into a supersack for storage or disposal. The filtrate from both units is collected in a common filtrate sump from where a vertical sump pump transfers it to the thickener for treatment (Figure 6).

Tailings from the Microcel™ are sent to an Enviro-Clear thickener for initial dewatering. Cationic and anionic polymers are added to the tailings stream to accelerate the particle settling rate to approximately 12 inch/minute. The thickened solids fall to the bottom of the thickener tank and form a slurry of approximately 20% - 30% solids. The clear water overflows the top of the unit where it is collected in the clarified water sump and pumped back into the process.

The thickener underflow pump transports the thickened tailings to the tailings filter press feed sump. Here, two air operated diaphragm pumps transfer the material under pressure to two Eimco pressure filters. The filter cake is manually discharged from each unit for disposal in supersacks. The tailings filtrate is combined with the clean coal filtrate before being sent to the thickener.

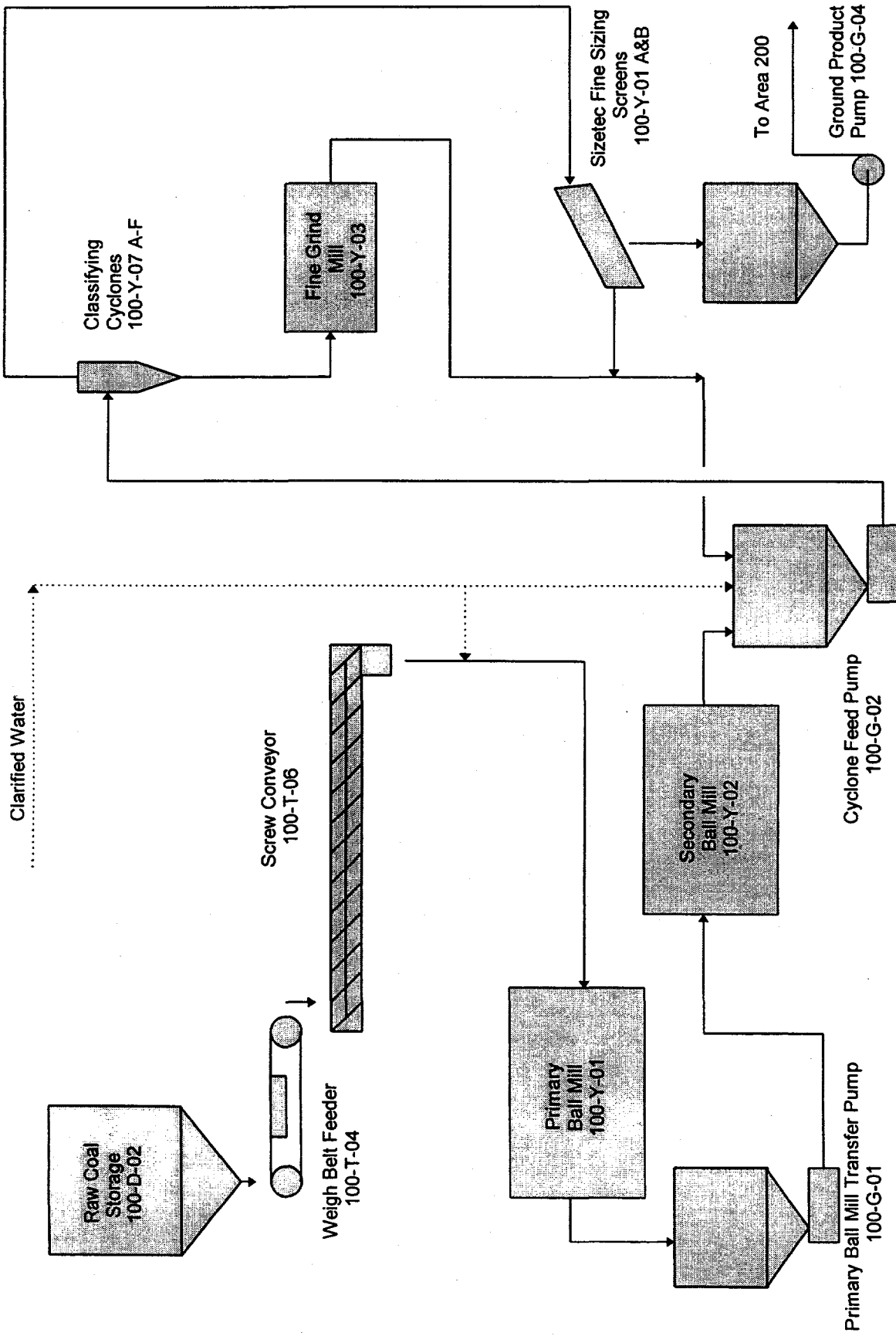


Figure 3. PDU Area 100 - Grinding / Classification Circuit

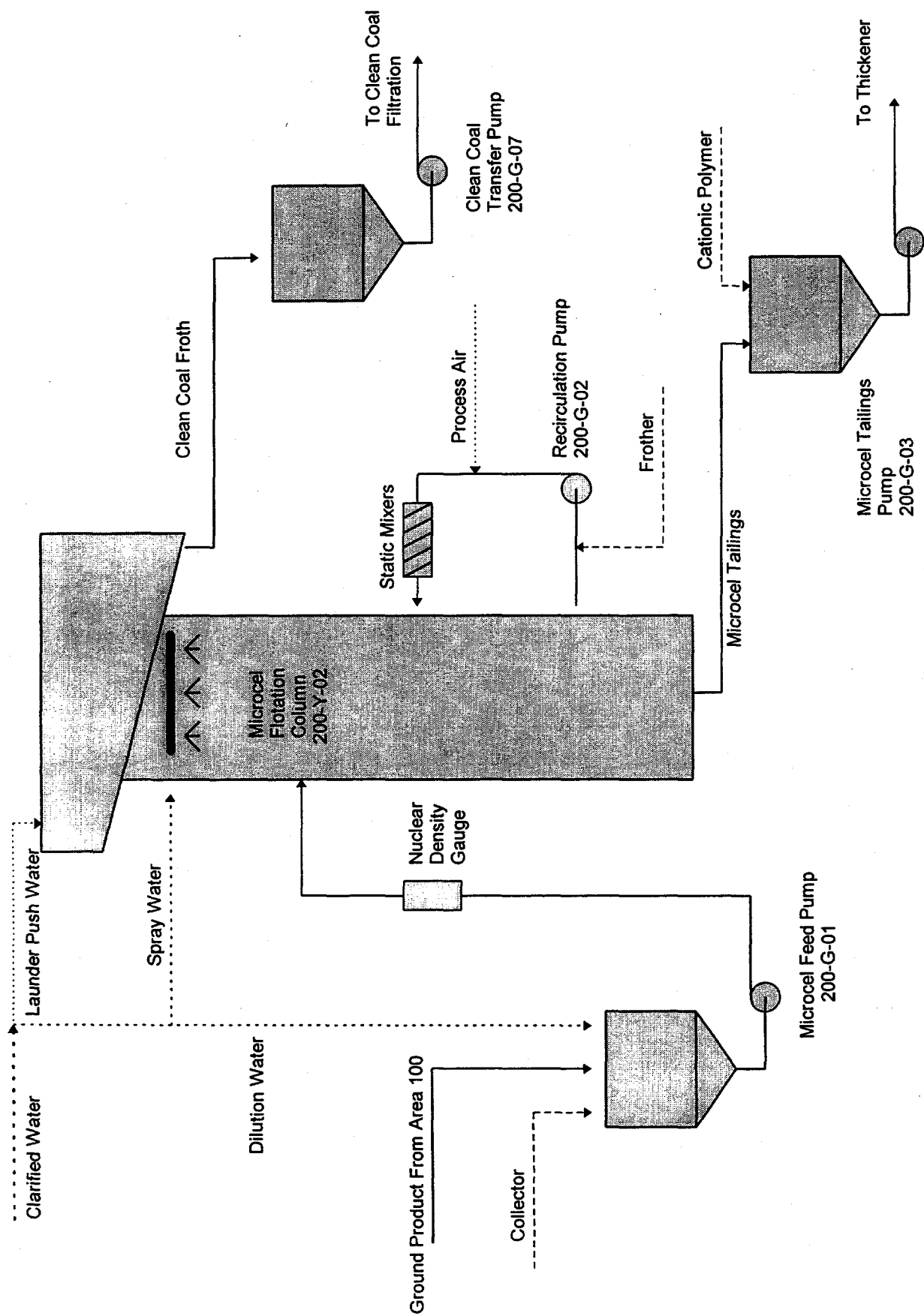


Figure 4. PDU Area 200 - Microcel™ Column Flotation Circuit

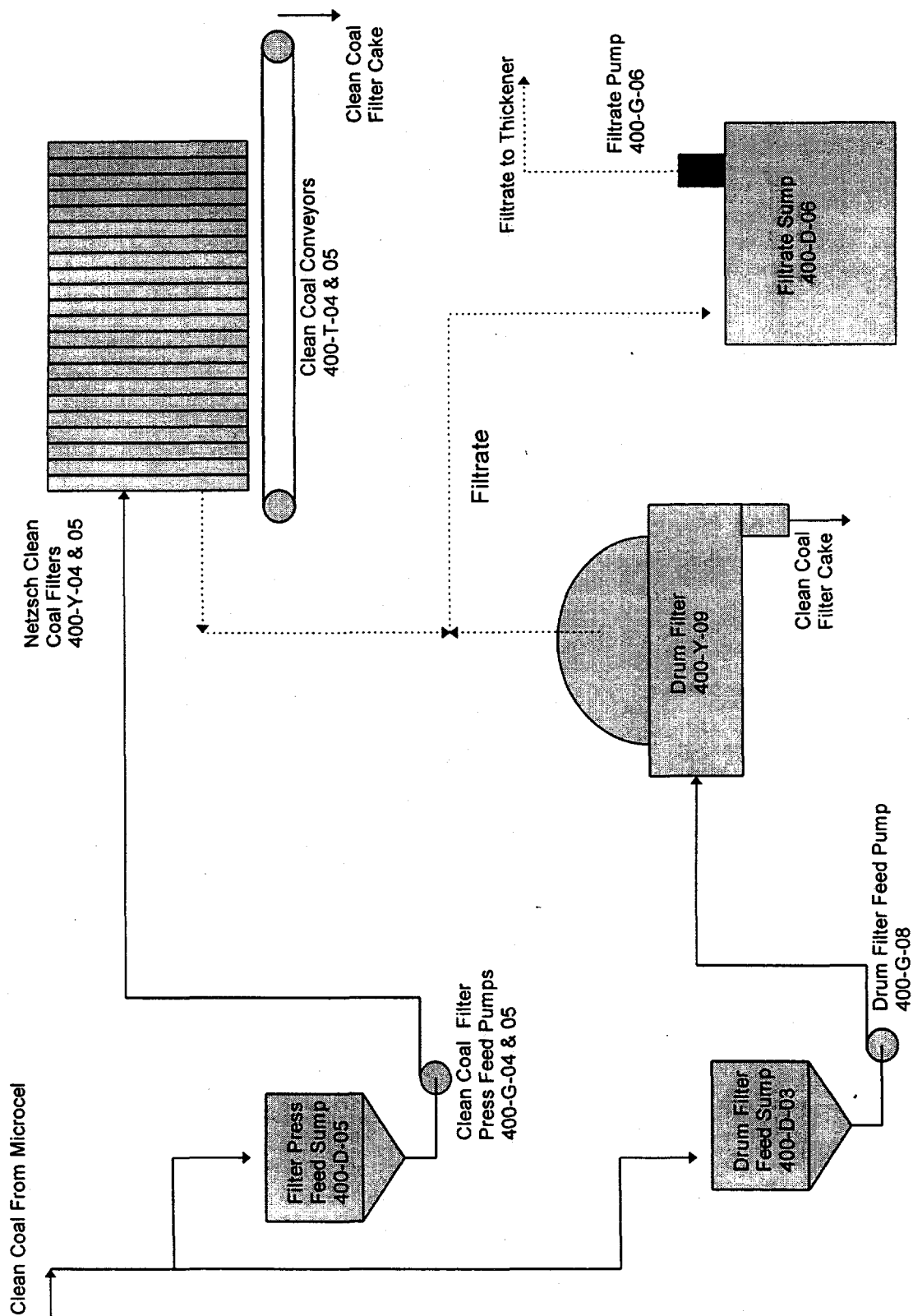


Figure 5. - Area 400 - Clean Coal Dewatering Circuit

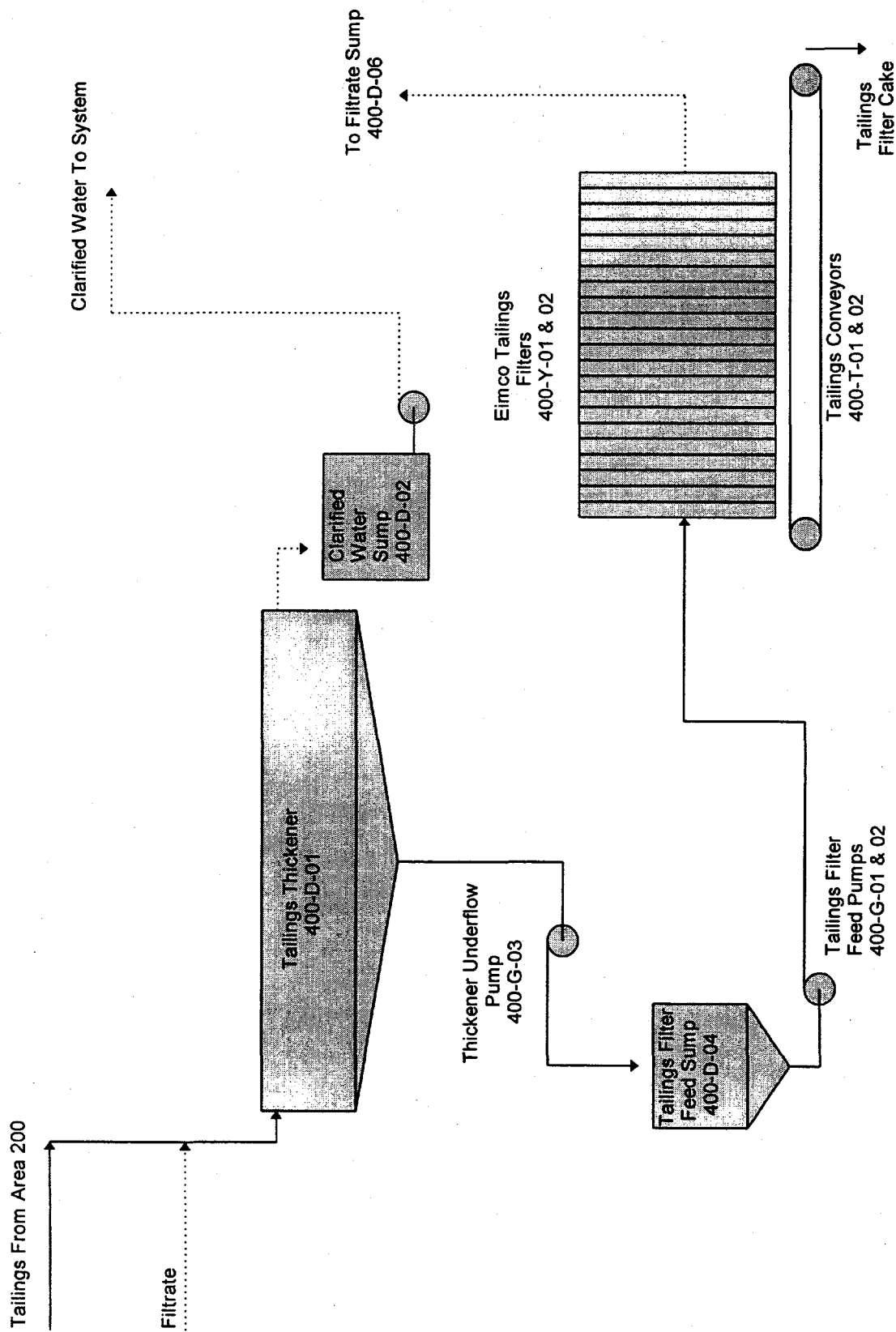


Figure 6. - Area 400 - Tailings Dewatering Circuit

PDU FLOTATION MODULE STARTUP AND SHAKEDOWN

Startup and shakedown of the PDU flotation module was completed during the last quarter of 1995 according to the Test Plan submitted to DOE [12]. Though some minor operating difficulties were encountered, corrective actions resulted in a fully functional PDU flotation module. Physical and mechanical improvements resulted in the elimination of process bottlenecks which allowed the PDU to operate at a design feed rate of 4,200 lb/hr.

PDU Flotation Module - Operating Personnel / Staffing

The PDU flotation module requires many different crafts and skills to properly operate and maintain the equipment found in each unit operation. Because research and development is the main thrust behind this project, technicians who possess strong operating, maintenance, and analytical skills must be utilized. In addition, management must be sure that adequate manpower levels are maintained.

Entech Global, Inc. management established a staffing schedule to ensure that these objectives are realized. A general overview of the required flotation module staffing is shown in Table 7.

Table 7. - PDU Flotation Module Staffing

<u>Staff Position</u>	<u>Primary Responsibility</u>
Operator - Area 100	Monitor / operate coal handling and grinding circuits.
Operator - Area 200	Monitor / operate Microcel™ flotation column. Assist in Area 400.
Operator - Area 400	Monitor / operate dewatering circuit.
Control Room Operator	Monitor PDU status, collect and summarize test data.
Laboratory Technician	Prepare test samples for analyses. Perform sample analyses.
Project Engineer	Supervise operation of PDU flotation module.

Review of the above table indicates that four operating personnel, one laboratory technician, and one project engineer were required for safe and effective operation of the PDU during one shift operation. Additional staff were provided during round-the-clock operations needed for production runs. Details of duties performed by PDU personnel are included in the PDU Test Plan [12].

PDU Flotation Module - Equipment Shakedown

Efforts related to the PDU module shakedown commenced during the third quarter of 1995 and concluded during the fourth quarter of 1995. Any problems, mechanical or electrical, were corrected accordingly by TIC or Entech personnel.

All process equipment items were checked for proper rotation (where applicable) and operated individually. All equipment items, except for the Techweigh scale, passed the shakedown test and were considered ready for operation.

Some additional problems (mostly electrical) were discovered during initial shakedown efforts. As a result, a TIC electrician was retained through the latter part of September, 1995 to complete the associated changes.

Next, all Proportional Integral Derivative (PID) Loops were configured and tested. Specifically, the following loops were evaluated and considered operational:

- Area 100 - Cyclone sump level control
- Area 100 - Ground product sump level control
- Area 200 - Microcel™ feed density control
- Area 200 - Microcel™ feed sump level control
- Area 200 - Microcel™ level control
- Area 200 - Microcel™ tailings level control
- Area 200 - Microcel™ air delivery control
- Area 200 - Microcel™ wash water control
- Area 200 - Microcel™ recirculation flow rate control

PDU Flotation Module Test Plan

The PDU flotation module test plan was completed and submitted on December 14, 1995 to DOE for review and approval [12]. A training session, described in the test plan, was attended by all Entech technicians on December 7, 1995. The session covered startup, operation, and shutdown of the PDU flotation module.

Though parametric testing of the three test coals was initially scheduled for December, 1995, unexpected problems encountered during startup and shakedown of the PDU flotation module pushed the start of test work to January, 1996. This schedule adjustment did not impact the overall project schedule since PDU process improvements allowed more testing to be completed each day. Tables 8, 9, and 10 show the planned test matrix for the Taggart, Indiana VII, and Hiawatha coals, respectively.

Table 8. Parametric Test Matrix - Taggart Coal

Test #	Collector (lb/ton)	Frother (lb/ton)	% Solids	Air Rate (CFM)	Wash H ₂ O (GPM)	Recirculate (GPM)	Feed Rate (lb/hr)
1	0.50	0.75	7.50	55	71	800	4200
2	0.75	0.75	7.50	55	71	800	4200
3	0.25	0.75	7.50	55	71	800	4200
4	0.50	1.00	7.50	55	71	800	4200
5	0.50	0.50	7.50	55	71	800	4200
6	0.50	0.75	10.00	55	71	800	4200
7	0.50	0.75	5.00	55	71	800	4200
8	0.50	0.75	7.50	75	71	800	4200
9	0.50	0.75	7.50	35	71	800	4200
10	0.50	0.75	7.50	55	100	800	4200
11	0.50	0.75	7.50	55	40	800	4200
12	0.50	0.75	7.50	55	71	800	4200
13	0.50	0.75	7.50	55	71	1000	4200
14	0.50	0.75	7.50	55	71	600	4200
15	0.50	0.75	7.50	55	71	800	3200
16	0.50	0.75	7.50	55	54	800	3200
17	0.50	0.75	7.50	55	71	800	5200
18	0.50	0.75	7.50	55	87	800	5200
19	0.50	0.75	7.50	55	71	800	6000
20	0.50	0.75	7.50	55	101	800	6000
21	0.50	0.75	7.50	55	71	800	4200

Table 9. Parametric Test Matrix - Indiana VII Coal

Test #	Collector (lb/ton)	Frother (lb/ton)	% Solids	Air Rate (CFM)	Wash H ₂ O (GPM)	Recirculate (GPM)	Feed Rate (lb/hr)
1	5	2.5	7.50	55	142	800	3200
2	7	2.5	7.50	55	142	800	3200
3	3	2.5	7.50	55	142	800	3200
4	5	3.5	7.50	55	142	800	3200
5	5	1.5	7.50	55	142	800	3200
6	5	2.5	10.00	55	142	800	3200
7	5	2.5	5.00	55	142	800	3200
8	5	2.5	7.50	75	142	800	3200
9	5	2.5	7.50	35	142	800	3200
10	5	2.5	7.50	55	142	800	3200
11	5	2.5	7.50	55	180	800	3200
12	5	2.5	7.50	55	100	800	3200
13	5	2.5	7.50	55	142	1000	3200
14	5	2.5	7.50	55	142	600	3200
15	5	2.5	7.50	55	142	800	2500
16	5	2.5	7.50	55	111	800	2500
17	5	2.5	7.50	55	142	800	3900
18	5	2.5	7.50	55	173	800	3900
19	5	2.5	7.50	55	142	800	3200

Table 10. Parametric Test Matrix - Hiawatha Coal

Test #	Collector (lb/ton)	Frother (lb/ton)	% Solids	Air Rate (CFM)	Wash H ₂ O (GPM)	Recirculate (GPM)	Feed Rate (lb/hr)
1	0.50	0.75	7.50	55	86	800	4300
2	0.75	0.75	7.50	55	86	800	4300
3	0.25	0.75	7.50	55	86	800	4300
4	0.50	1.00	7.50	55	86	800	4300
5	0.50	0.50	7.50	55	86	800	4300
6	0.50	0.75	10.00	55	86	800	4300
7	0.50	0.75	5.00	55	86	800	4300
8	0.50	0.75	7.50	75	86	800	4300
9	0.50	0.75	7.50	35	86	800	4300
10	0.50	0.75	7.50	55	115	800	4300
11	0.50	0.75	7.50	55	55	800	4300
12	0.50	0.75	7.50	55	86	800	4300
13	0.50	0.75	7.50	55	86	1000	4300
14	0.50	0.75	7.50	55	86	600	4300
15	0.50	0.75	7.50	55	86	800	3300
16	0.50	0.75	7.50	55	66	800	3300
17	0.50	0.75	7.50	55	86	800	5300
18	0.50	0.75	7.50	55	106	800	5300
19	0.50	0.75	7.50	55	86	800	6000
20	0.50	0.75	7.50	55	120	800	6000
21	0.50	0.75	7.50	55	86	800	4300

PDU FLOTATION MODULE OPERATION AND TESTWORK

Operation of the PDU Flotation Module commenced in January, 1996 with the Taggart coal and concluded in September, 1996 with the Hiawatha coal. Testing of each coal typically involved the following:

- Testing and optimization of grinding circuit.
- Parametric testing in Microcel™ flotation unit.
- Optimization of Microcel™ flotation unit.
- Extended production run.

A discussion of each coal's testing and production runs is provided in the following pages.

Taggart Coal

Parametric testing of the Taggart coal in the PDU Flotation Module commenced in January, 1996 and was concluded during March, 1996. The operation of the PDU with Taggart coal was quite successful with project goals achieved on numerous occasions.

Testing and Optimization of Grinding Circuit - Taggart Coal

The test work was performed to determine the best grinding scenario for optimum liberation of mineral matter. Because the Taggart coal (from the Steer Branch mine) evaluated in the PDU flotation module had a higher ash content than the Taggart coal (from the Wentz mine) previously used in the 12-inch Microcel™ column (4.01% vs. 2.08 %), additional liberation was required. Laboratory testing had shown that adequate liberation is achieved when this new Taggart coal sample is ground to a d_{80} of 50 microns (80 percent passing 50 microns). The challenge faced by the PDU staff was to determine which grinding arrangement would produce a similar size distribution.

Twenty-five tests aimed at optimizing the grinding circuit were conducted. Specifically, the effects of feed rate, cyclone size, screen opening size, and circuit type (open or closed) were evaluated. The results are summarized in Table 11.

With the exception of tests T-17 and T-19, all oversize material from the cyclones and screens was recirculated to the secondary ball mill. The oversize material was recirculated to the primary mill during test T-17 and to the Netzsch mill during test T-19.

An evaluation of the data indicated that the desired clean coal quality of 1 lb ash/MBtu was achieved at a d_{80} of 52 microns during test T-21. The changes made to the grinding circuit prior to the start of test T-21 were:

- Loading of the primary and secondary ball mills was increased from 10,000 lbs each to 13,628 lbs and 14,057 lbs, respectively.
- 140-mesh screen cloth was used in the Sizer screens.
- Influence pattern of the Sizer screen sprays were changed from 35 degrees to 50 degrees.

As a result of this effort, the following grinding arrangement was established for use in all Microcel™ parametric test work utilizing the Taggart coal:

- | | |
|------------------------|---------------------------------|
| • Feed Rate: | 4,200 lb/hr |
| • Primary Water: | 15 gpm |
| • Primary Mill Load: | 13,628 pounds |
| • Secondary Mill Load: | 14,057 pounds |
| • Cyclone Water: | 25 gpm |
| • Cyclones: | 3 inch |
| • Screen Cloth: | 140 mesh |
| • Screen Water: | 36 gpm |
| • Recirculation: | Oversize to Secondary Ball Mill |

Table 11. Parametric Testing of PDU Grinding Circuit - Taggart Coal

Test #	Date	Screen Cloth Size	Cyclone Size	Feed Rate lb/hr	Fuel Oil lb/ton	MIBC lb/ton	% Solids	Air Rate CFM	Wash Water GPM	Particle Size d80	PDU Yield	PDU Energy Recov.	Product Ash lb/MBtu
T-1	1/10/96	70 M	3 inch	4,200	0.50	0.75	7.50	55	71	50	95.60	96.78	1.02
T-2	1/10/96	70 M	3 inch	4,200	0.50	0.75	7.50	55	71	115	96.45	97.26	1.20
T-3	1/11/96	70 M	N/A	4,200	0.50	0.75	7.50	55	71	100	96.25	97.82	1.30
T-4	1/16/96	100 M	N/A	4,200	0.50	0.75	7.50	55	71	85	96.30	97.22	1.70
T-5	1/18/96	100 M	N/A	4,200	0.50	0.50	7.50	55	71	?	95.85	97.90	1.52
T-6	1/23/96	100 M	3 inch	4,200	0.50	0.75	7.50	55	71	76	95.38	97.58	1.09
T-7	1/24/96	100 M	N/A	4,200	0.50	0.75	7.50	55	71	94	96.50	98.59	1.27
T-8	1/24/96	100 M	N/A	4,200	0.50	0.25	7.50	55	71	90	93.21	97.21	1.18
T-9	1/30/96	100 M	3 inch	4,200	0.50	0.75	7.50	55	71	72	93.38	96.59	1.21
T-10	1/30/96	100 M	3 inch	4,200	0.75	0.75	7.50	55	71	74	96.00	98.10	1.18
T-11	2/1/96	100 M	3 inch	4,200	0.25	0.75	N/R	55	71	70	96.18	96.74	1.33
T-12	2/5/96	100 M	3 inch	4,300	0.50	0.75	6.29	55	71	64	95.62	97.00	1.33
T-13	2/6/96	70 M	3 inch	4,300	0.50	0.75	6.69	55	71	78	95.54	97.38	1.17
T-14	2/13/96	70 M	3 inch	4,300	0.50	0.75	8.04	55	71	91	95.46	97.33	1.50
T-15	2/13/96	100 M	3 inch	4,300	0.50	0.75	6.90	55	71	74	97.15	98.39	1.52
T-16	2/14/96	100 M	3 inch	4,300	0.50	0.75	7.31	55	71	68	95.09	97.47	1.22
T-17	2/15/96	100 M	3 inch	4,300	0.50	0.75	4.57	55	71	61	95.38	97.82	1.13
T-17-B	2/19/96	100 M	2 inch	4,300	0.50	0.75	N/R	55	71	80	N/R	N/R	1.16
T-18-A	2/20/96	100 M	3 inch	4,300	0.50	0.75	6.04	55	71	74	94.91	97.52	1.14
T-18-B	2/20/96	100 M	3 inch	4,300	0.50	0.75	5.41	55	71	71	95.17	97.80	1.13
T-18-C	2/20/96	100 M	3 inch	4,300	0.50	0.75	4.91	55	71	65	96.48	98.01	1.85
T-18-D	2/20/96	100 M	3 inch	4,300	0.50	0.75	6.37	55	71	81	94.31	96.46	1.42
T-19	2/21/96	70 M	3 inch	4,200	0.50	0.50	6.95	55	71	36	93.27	96.18	1.16
T-20	2/26/96	140 M	3 inch	4,200	0.50	0.50	7.15	55	71	61	96.69	98.33	1.10
T-21	2/27/96	140 M	3 inch	4,200	0.50	0.50	6.61	55	71	52	95.06	97.14	0.97

Parametric Testing of Microcel™ Flotation Column - Taggart Coal

A test matrix was established to determine the effects of independent variables such as air rate, percent solids, feed rate, wash water, and reagent dosage on response variables such as product ash and yield. The test matrix is shown in Table 12. It should be noted that three proposed tests were removed from the original matrix (Table 8) while one was revised. The three tests removed were those where feed rate was varied while holding the Microcel™ wash water ratio constant. In addition, a midpoint replicate test was revised to determine the effects of an extremely low frother dosage. The reason for these modifications was to conserve Taggart coal for use in the Agglomeration Module.

Like the Taggart coal evaluated in the 12-inch Microcel™ unit, the feedstock used in the PDU flotation module was easily floatable. In fact, the natural floatability of the Taggart coal produced comparable yield and quality values regardless of the change in the input parameters. Noticeable changes in the yield and quality were typically observed only when the input parameters were varied dramatically. The results of the parametric testing are shown in Table 13. This data shows that the overall quality goal of 1 lb ash/MBtu was met or exceeded in four tests. The clean coal yield varied from 58.5 to 96.6% while the energy recovery and product quality varied from 60.1 to 98.0% and 0.77 to 1.23 lb ash/MBtu, respectively.

The results of the parametric testing are also shown in Figures 7 and 8 which indicate that the target clean coal quality of 1 lb ash/MBtu should be optimally achieved at an approximate yield of 95% and an energy recovery of 97%. Optimization test work was performed to confirm this projection.

It is important to note that the grade-yield relationships found for the Taggart coal in the PDU are different than those found during the evaluation of the 12-inch Microcel™. The difference is the result of different feedstock qualities. Taggart coal from the Wentz mine was used in the bench-scale testing. However, due to the closing of the mine, Taggart coal from the Steer Branch mine was used in the PDU operation. Specifically, the ash content of the coal used in the 12-inch Microcel™ was 2.08% while that used in the PDU flotation circuit was 4.01%. The higher ash content normally results in lower yield values at similar product qualities.

It is also important to note that the d_{80} of the Microcel™ feed is directly related to the PDU feed rate. Specifically, the higher the feed rate, the coarser the size distribution (large d_{80} value). As a result, the clean coal quality obtained when varying feed rate is the effect of both feed rate (retention time in the flotation column) and the resulting d_{80} (mineral liberation).

Table 12. PDU Flotation Module Test Matrix - Taggart Coal

Test #	Collector lb/ton	Frother lb/ton	% Solids	Air Rate CFM	Wash Water GPM	Recirculation GPM	Feed Rate lb/hr
T-21	0.50	0.50	7.50	55	71	800	4,200
T-22	0.25	0.50	7.50	55	71	800	4,200
T-23	0.75	0.50	7.50	55	71	800	4,200
T-24	0.50	0.25	7.50	55	71	800	4,200
T-25	0.50	0.50	10.00	55	71	800	4,200
T-26	0.50	0.50	5.00	55	71	800	4,200
T-27	0.50	0.50	7.50	75	71	800	4,200
T-28	0.50	0.50	7.50	35	71	800	4,200
T-29	0.50	0.50	7.50	55	100	800	4,200
T-30	0.50	0.50	7.50	55	40	800	4,200
T-31	0.50	0.50	7.50	55	71	1000	4,200
T-32	0.50	0.50	7.50	55	71	600	4,200
T-33	0.50	0.50	7.50	55	71	800	4,200
T-34	0.50	0.75	7.50	55	71	800	4,200
T-35	0.50	0.10	7.50	35	71	800	4,200
T-36	0.50	0.50	7.50	55	71	800	3,800
T-37	0.50	0.50	7.50	55	71	800	3,200
T-38	0.50	0.50	7.50	55	71	800	5,500

Table 13. Parametric Testing of PDU Flotation Module - Taggart Coal

Test #	Fuel Oil lb / ton	Frother lb/ton	% Solids	Air Rate CFM	Wash GPM	Recirc GPM	Feed lb/hr	Microcel d80	PDU Yield	Energy Recov	Ash lb/MBtu
T-21	0.50	0.50	6.61	55	71	800	4,184	52	95.06	97.15	0.97
T-22	0.25	0.50	7.10	55	71	800	4,188	58	93.52	96.23	1.03
T-23	0.75	0.50	7.17	55	71	800	4,196	52	95.54	97.64	1.01
T-24	0.50	0.25	5.69	55	71	800	4,189	50	88.40	90.75	0.91
T-25	0.50	0.50	6.63	55	71	800	4,200	51	94.36	96.90	0.99
T-26	0.50	0.50	4.54	55	71	800	4,223	51	94.20	96.41	1.05
T-25-B	0.50	0.50	9.85	55	71	800	4,203	53	94.14	97.26	1.16
T-27	0.50	0.50	5.31	75	71	800	4,206	48	93.50	96.15	1.03
T-28	0.50	0.50	6.59	35	71	800	4,200	49	94.94	96.55	1.14
T-29	0.50	0.50	5.65	55	100	800	4,211	51	92.90	95.28	1.07
T-30	0.50	0.50	5.70	55	40	800	4,150	58	96.64	97.97	1.22
T-31	0.50	0.50	6.07	55	71	1,000	4,200	58	92.11	94.85	1.23
T-32	0.50	0.50	6.02	55	71	600	4,190	51	91.12	93.79	1.03
T-33	0.50	0.50	6.74	55	71	800	4,191	50	93.93	96.33	1.14
T-34	0.50	0.50	6.94	35	71	800	4,217	59	87.67	91.01	1.16
T-35	0.50	0.75	6.61	55	71	800	4,193	51	90.36	93.35	1.22
T-36	0.50	0.10	7.40	55	71	800	4,200	53	58.53	60.13	0.77
T-37	0.50	0.50	6.87	55	71	800	3,800	55	93.58	95.69	1.16
T-38	0.50	0.50	5.96	55	71	800	3,192	56	94.83	96.78	1.10
T-39	0.50	0.50	7.05	55	71	800	5,500	63	94.75	96.77	1.16
MAX	0.75	0.75	9.85	75	100	1,000	5,500	63	96.64	97.97	1.23
MIN	0.25	0.10	4.54	35	40	600	3,192	48	58.53	60.13	0.77

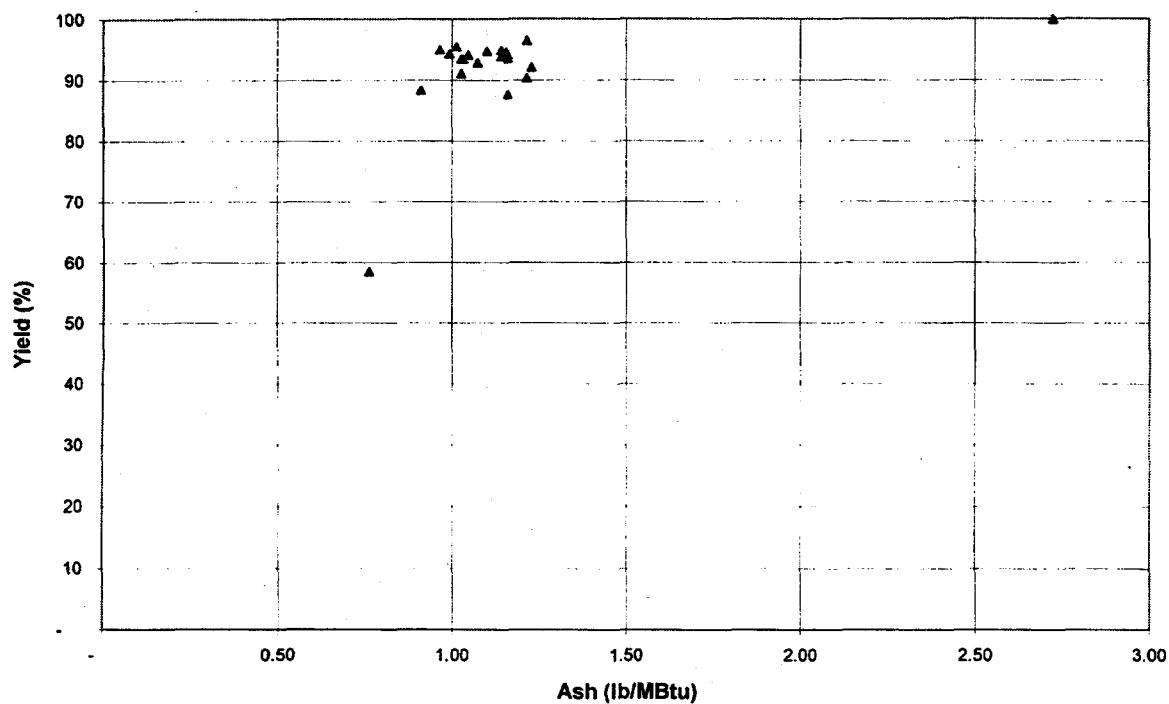


Figure 7. Taggart Coal Parametric Testing - Yield vs. Product Ash

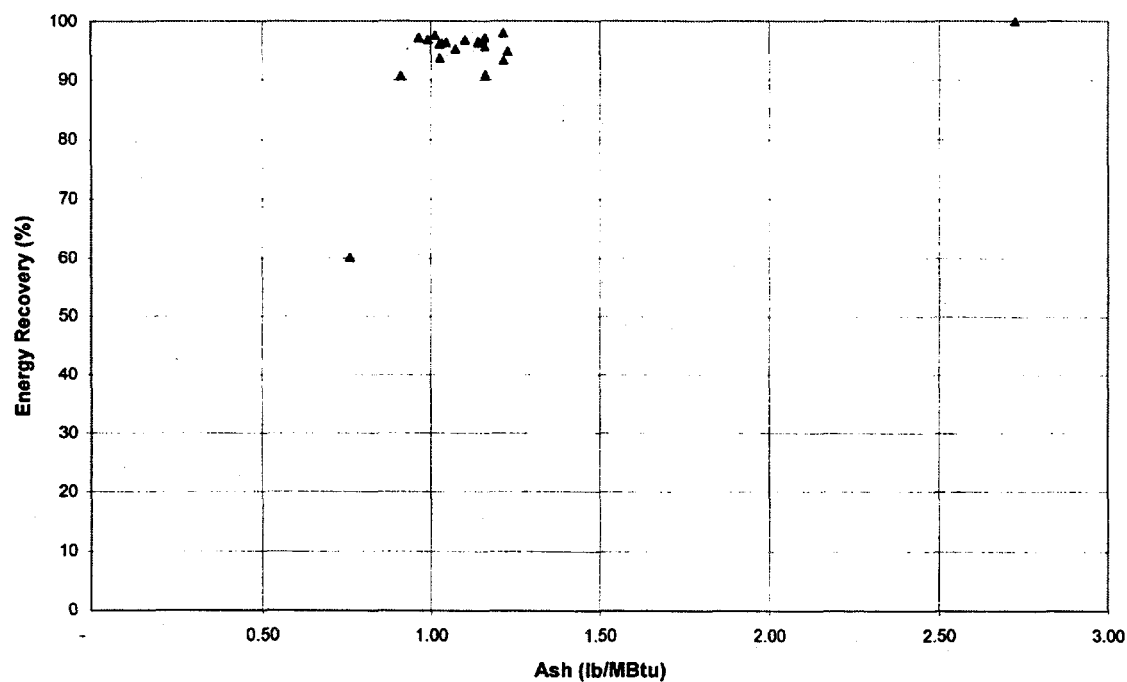


Figure 8. Taggart Coal Parametric Testing - Energy Recovery vs. Product Ash

Development of Regression Equations - Taggart Coal

Data from PDU Flotation Module parametric testing was compiled and multiple regression models (equations) were generated. Forward stepwise regression produced equations which link output variables such as yield and clean coal quality to input variables such as feed rate, wash water rate, air rate, collector addition, and frother addition. The equation term coefficients for the two main response variables are shown in Tables 14 and 15.

The Coefficient of Determination (R^2) and Adjusted Coefficient of Determination (Adjusted R^2) both show that each equation fits the data quite well. In addition, observation of the t-statistic, which indicates the relative importance of each independent variable to the response equation, shows that the most important variable that affects yield and clean coal ash is the frother dosage. In both cases, the frother dosage is directly proportional to these resulting variables. It is important to note that though the yield and clean coal ash are dependent on the feed coal ash content, the ash content itself is not a controllable variable but considered a covariate.

Optimization of Flotation Column - Taggart Coal

A total of seven tests were performed to determine the optimum Microcel™ setpoints needed to achieve the process development goals of 1 lb ash/MBtu and over 80% energy recovery. Equations were first developed to estimate the effects of tested input variables on Microcel™ outputs such as yield and quality. The equations were developed by evaluating the input and output variables of parametric tests T21 through T39 by multiple linear regression. The resulting equations were then used to determine optimum Microcel™ setpoints. A unique function found in the Microsoft Excel software package called "Solver" was used to determine the proposed optimal setpoints for test work. The results of the optimization test work are summarized in Table 16.

An evaluation of the data shows that the overall quality goal of 1 lb ash/MBtu was achieved in two tests during optimization (tests TO-6 and TO-7). The clean coal yield varied from 87.5% to 88.3% while the product ash varied from 0.88 lb ash/MBtu to 0.97 lb ash/MBtu. It is important, however, to note that the aforementioned results correspond to a d_{80} of approximately 60 microns. Parametric testing, on the other hand, indicated improved yield and energy recovery when the d_{80} was close to 50 microns. As a result, the yield and product ash values of 94.4% and 0.99 lb ash/MBtu achieved during parametric test T-25 should be considered optimum.

Table 14. Regression Analysis of Taggart Yield (%)

<u>Input Variable</u>	<u>Equation Coefficient</u>	<u>t-Statistic</u>
Intercept	130.66143	27.10
% Solids	-1.66891	-2.14
(Feed Ash) ²	-0.45968	-15.95
(% Solids) ²	0.16377	3.05
(Frother lb/ton) ^{1/2}	-28.15453	-9.16
1 / (Frother lb/ton)	-5.97012	-34.14
1 / Recirculation Rate (gpm)	-3503.73683	-3.67
ln (Air Rate CFM)	2.55721	3.59
Coefficient of Determination (R ²)	0.999	
Adjusted R ²	0.997	

Table 15. Regression Analysis of Taggart Product Ash (lb/MBtu)

<u>Input Variable</u>	<u>Equation Coefficient</u>	<u>t-Statistic</u>
Intercept	173.13462	2.51
% Solids	0.16140	3.73
d80	-1.18726	-2.82
(Feed Ash) ²	-0.06340	-1.57
(Frother lb/ton) ²	0.92194	7.07
(Wash Water GPM) ²	2.91293 E-05	1.59
(Recirculation GPM) ²	6.67988 E-07	1.30
(d80) ²	0.01114	2.84
1 / (Feed Ash %)	-8.20673	-1.52
1 / (Collector lb/ton)	-0.09205	-2.95
1 / (% Solids)	5.52978	3.10
1 / (Air Rate CFM)	-111.34457	-1.43
1 / (Wash Water GPM)	26.86557	1.35
1 / (Feed Rate lb/hr)	-47,416.95503	-2.24
ln (Air Rate CFM)	-2.67400	-1.74
ln (Recirculation GPM)	-0.64466	-1.03
ln (Feed Rate lb/hr)	-13.42548	-2.34
Coefficient of Determination (R ²)	0.978	
Adjusted R ²	0.861	

Table 16. PDU Flotation Optimization Testing - Taggart Coal

Test	Fuel Oil lb/ton	Frother lb/ton	% Sol	Air CFM	Wash GPM	Recirc GPM	Feed lb/hr	Column d80	PDU Yield	Energy Recov	Sulfur lb/MBtu	Ash lb/MBtu
TO-1	0.26	0.42	6.51	75	77	852	4,200	58	95.53	97.43	0.45	1.18
TO-2	0.26	0.42	5.58	75	77	852	4,200	58	95.26	97.31	0.46	1.16
TO-3	0.26	0.31	6.16	55	77	800	4,200	52	92.69	95.21	0.46	1.05
TO-4	0.26	0.25	7.72	75	77	990	4,200	66	92.34	95.23	0.47	1.24
TO-5	0.53	0.25	6.24	75	77	990	4,200	66	95.39	97.71	0.47	1.32
TO-6	0.26	0.20	7.14	55	77	800	4,200	59	86.63	88.26	N/R	0.88
TO-7	0.26	0.25	7.70	55	77	800	4,200	60	85.46	87.53	N/R	0.97
T-25	0.50	0.50	6.63	55	71	800	4,200	51	94.36	96.90	0.44	0.99
MAX	0.53	0.50	7.72	75	77	990	4,200	66	95.53	97.71	0.47	1.32
MIN	0.26	0.20	5.58	55	77	800	4,200	51	85.46	87.53	0.45	0.88

Table 17. Extended Production Run - Taggart Coal

Date	Time	Total Hours	Feed lb/hr	Feed Solids	Feed Ash %	Tails Ash %	Product Moist %	Particle d80 (μ)	PDU Yield	Btu Rec %	Sulfur lb/MBtu	Ash lb/MBtu
3/25/96	1:00 P	1.50	3,824	7.95	3.46	31.18	32.94	N/A	94.51	96.37	0.46	1.23
3/25/96	7:00 P	7.50	3,824	8.01	3.46	38.49	33.80	N/A	95.35	96.47	0.47	1.17
3/26/96	1:00 A	13.50	3,824	7.79	3.41	42.89	33.17	N/A	95.99	97.67	0.47	1.17
3/26/96	7:00 A	19.50	3,824	7.70	3.48	43.21	34.48	N/A	96.01	97.42	0.47	1.22
3/26/96	1:00 P	25.50	3,824	7.64	3.61	40.85	32.78	68	95.63	97.00	0.47	1.28
3/26/96	7:00 P	31.50	3,824	7.67	3.65	35.96	32.77	72	94.72	96.04	0.47	1.24
3/27/96	1:00 A	37.50	3,824	7.77	3.78	35.30	31.65	71	94.20	96.08	0.47	1.23
3/27/96	7:00 A	43.50	3,824	7.29	3.57	38.10	32.01	73	95.20	96.74	0.47	1.22
3/27/96	1:00 P	49.50	3,824	7.20	3.75	36.40	32.08	N/A	94.61	96.73	0.48	1.26
3/27/96	7:00 P	55.50	3,824	6.86	3.80	40.93	32.09	N/A	94.99	96.71	0.47	1.23
3/28/96	1:00 A	61.50	3,824	6.82	3.76	44.23	31.19	N/A	95.36	97.39	0.47	1.19
3/28/96	7:00 A	67.50	3,824	7.35	3.60	45.61	31.30	N/A	95.89	97.59	0.47	1.20
3/28/96	11:30 A	72.00	3,824	6.96	3.44	42.29	33.90	N/A	96.07	97.57	0.46	1.23
AVG			3,824	7.46	3.60	39.65	32.63	71	95.27	96.91	0.47	1.22
MAX				8.01	3.80	45.61	34.48	73	96.07	97.67	0.48	1.28
MIN				6.82	3.41	31.18	31.19	68	94.20	96.04	0.46	1.17
SDEV				0.41	0.14	4.17	1.02	2	0.63	0.58	0.01	0.03

An extended production run of the PDU Flotation Module was successfully completed during the week of March 25, 1996. The effort commenced Monday, March 25 at 11:30 AM and concluded 72 hours later on Thursday, March 28 at 11:30 AM. Aside from a failed belt splice, uninterrupted operation was achieved showing excellent reliability of the operation. The PDU Flotation Module was operated at the following parameters during the production run:

• Test No.:	T-40
• Coal:	Taggart
• Nominal Feed Rate:	3,800 lb/hr
• Sizer Screen Cloth:	100 mesh
• Grinding Circuit:	Closed / 3" Cyclone / Screen / Sec Mill
• Primary Water:	15 GPM
• Cyclone Water:	20 - 25 gpm
• Ground Product H ₂ O:	0 GPM
• Collector:	0.50 lb/ton (9 cc/min)
• Frother:	0.50 lb/ton (9 cc/min)
• % Solids Setpoint:	7.50
• Microcel™ Dilution:	0 gpm
• Air Rate:	55 CFM
• Microcel™ Level SP:	55 inches
• Spray Water:	71 gpm
• Launder Water:	0 gpm
• Microcel™ Recirculation:	800 GPM

The results of the production run are presented in Table 17. The PDU was operated at a feed rate of approximately 3,800 lb/hr due to the filter capacity limitations previously encountered during parametric testing. Had the feed rate been greater than 3,800 lb/hr, the PDU would have shut down prematurely due to a lack of clean coal slurry storage capacity. Overall, 275,340 pounds of coal (137.67 tons) were processed in the PDU Flotation Module while 220 bags of clean coal filter cake were produced.

Disposal of Clean Coal Product - Taggart Coal

Communications during the first quarter of 1996 with DOE/PETC indicated that Penn State University (PSU) was interested in procuring the Taggart clean coal produced during the extended production run of the PDU Flotation Module. Though

approximately 200 tons of clean coal filter cake was slated for transport to PSU's Coal Utilization Laboratory, only 50 bags were ultimately shipped. Difficulties in finding coal storage at Penn State resulted in a reduced delivery. The delivery of Taggart coal was made August 13, 1996. The remaining clean coal product was either shipped to Western Aggregates in Golden, CO for use as a fuel or disposed at a local landfill.

Indiana VII Coal

Parametric testing of the Indiana VII coal in the PDU Flotation Module commenced in April, 1996 and was concluded during July, 1996. Though the product ash goal of 2 lb ash/MBtu was difficult to achieve, the operation of the PDU with Indiana VII coal was considered quite successful.

Laboratory Release Analysis - Indiana VII Coal

To better define the theoretical grade-yield curves associated with different feed size distributions, release analysis test work was performed on the Indiana VII coal. Two Microcel™ feed slurries, one having a d_{80} of 22 microns and a second with d_{80} of 19 microns, were evaluated. The results are shown in Figure 9. It is seen that the product goal of 2 lb ash/MBtu can be achieved at yields of 74% and 83% for feed slurries having a d_{80} of 22 and 19 microns respectively. The increase in yield can be attributed to enhanced liberation of carbon and mineral matter at the finer grind.

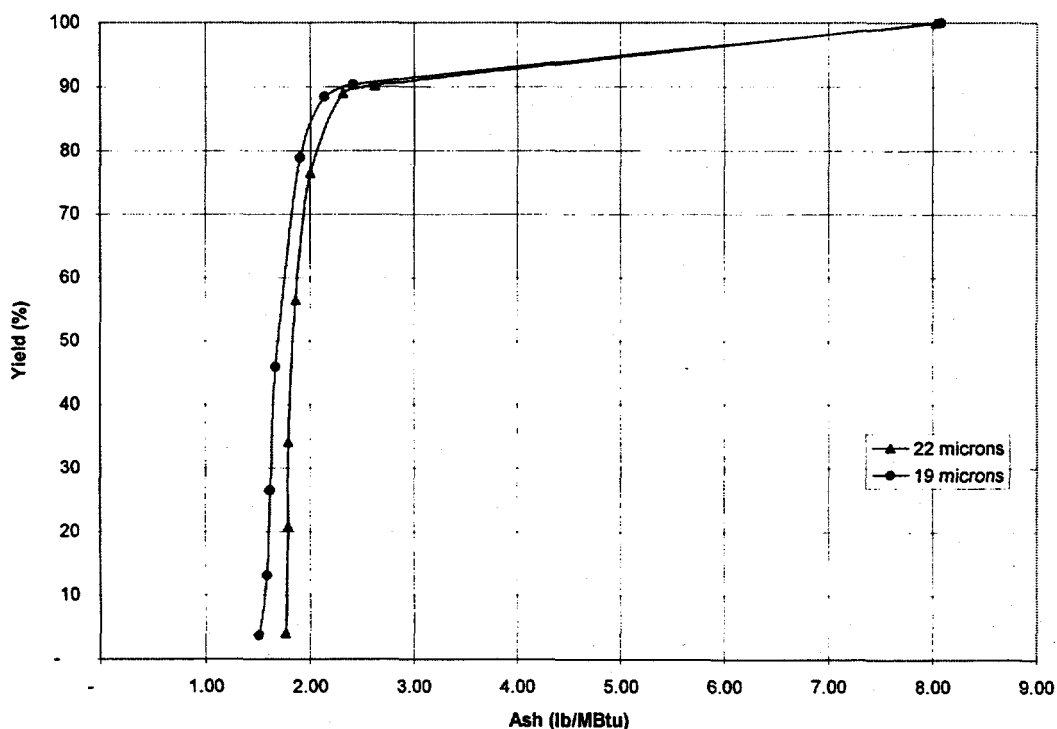


Figure 9. Laboratory Release Analysis - Indiana VII Coal

It is important to note that though the additional yield associated with a d_{80} grind of 19 microns may be desirable, the production of a similar size consist in the PDU flotation module would result in a dramatic reduction in filtering capacity as well as an increase in product moisture. As a result, all PDU test work was performed at a d_{80} of 22 microns.

Testing and Optimization of Grinding Circuit - Indiana VII Coal

Overall, the testing indicated that the required particle size distribution (d_{80} of 22 microns) could only be achieved at the following conditions:

- Feed Rate: 3,200 lb/hr
- Grinding Circuit: 2" Cyclones, Screens, Netzsch Mill
- Primary Water: 8 gpm
- Cyclone Water: 12 gpm
- Screen Cloth: 270 mesh
- Screen Water: 142 gpm

Modification of Area 100 Grinding Circuit

The fine liberation requirements of the Indiana VII coal led to several unexpected operating problems in the PDU grinding circuit. Specifically, degradation and loss of grinding media from the ball mills resulted in screen blinding, cyclone plugging, increased d_{80} values, and unexpected downtime. As a result, parametric testing of the Indiana VII coal was temporarily suspended and efforts redirected to troubleshooting and correcting these problems. Consultations with Mineral Resource Development Inc., a firm specializing in grinding and size reduction, revealed that the speed of each mill was too fast, the ball size distribution was too coarse, the ball charge was too heavy, and the mill solids concentration was too dilute. Their recommendations, which were implemented by Entech Global personnel, are listed as follows:

- **Reduce Mill Speed** - The critical speed of each mill was calculated to be approximately 35 RPM. The actual speed of each mill, however, was measured at 85% of critical or 30 RPM. Because optimum grinding efficiency occurs at a mill speed of 65% of critical, the speed of each mill was reduced to 24 RPM.
- **Reduce Ball Size Distribution** - The size distribution of balls in each mill was found to be too large for the 1/2 inch coal being fed to the circuit. As a result, the ball topsize in the primary mill was reduced from 3 inches to 2 inches while that of the secondary mill was reduced from 2-1/2 inches to 1-1/2 inches. The new overall distribution of balls in each mill is as shown in Table 18.

Table 18. Distribution of Balls in Primary and Secondary Mills

<u>Ball Size</u>	<u>Primary Mill Distribution</u>	<u>Secondary Mill Distribution</u>
2 inch	33.3 %	--
1.5 inch	33.3 %	33.3 %
1 inch	33.3 %	33.3 %
0.5 inch		33.3 %
Total	100.0 %	100.0 %

- **Reduce Overall Ball Charge** - The total weight of balls in each mill was too high for the amount of coal being processed. As such, balls were impacting each other, contributing to media degradation. The total ball weight of each mill was reduced from 13,600 and 14,000 pounds in the primary and secondary mills, respectively, to 8,100 pounds in each mill.
- **Increase Solids Concentration** - Previous test work performed in the PDU on the Taggart coal revealed that the concentration of coal solids in each mill was typically around 20%. The low solids concentration is the result of excess push water being used at the primary mill inlet. Ideally, the solids concentration in each mill should be somewhere between 40% and 50%. Entech operating personnel found that the mill solids concentration can be increased by slowly reducing the primary mill push water to its operating minimum quantity. Therefore, the ratio of push water to coal feed tonnage was reduced from over 7 GPM per t/hr of coal to 5 GPM per t/hr of coal. This reduction in push water increased the mill solids concentration to over 38%.

Implementation of each of the above recommendations eliminated all operational problems previously encountered in the grinding circuit. Not only were screen blinding and cyclone plugging problems eliminated, the d_{80} of the Microcel™ feed particle size distribution consistently ranged from 20 to 23 microns.

Parametric Testing of Microcel™ Flotation Column - Indiana VII Coal

A test matrix was established to determine the effects of independent variables such as air rate, % solids, feed rate, wash water, and reagent dosage on response variables such as product ash and yield. The test matrix is shown in Table 19. Due to the fine liberation requirements of the Indiana VII coal, several unexpected operating problems were encountered in the PDU grinding circuit. Degradation and loss of grinding media from the ball mills resulted in screen blinding, cyclone plugging, increased d_{80} values, and unexpected downtime. As a result, the parametric testing of the Indiana VII coal was temporarily suspended after 13 tests. Testing resumed after all problems associated with the grinding circuit were corrected. Unfortunately, due to inadequate liberation of mineral matter, data from only 19 tests could be considered valid.

Table 19. PDU Flotation Module Test Matrix - Indiana VII Coal

Test	Collector lb/ton	Frother lb/ton	% Solids	Air Rate CFM	Wash Water GPM	Recirculation GPM	Feed Rate lb/hr
I-1	5.0	2.50	7.50	55	142	800	3,200
I-2	3.0	2.50	7.50	55	142	800	3,200
I-3	7.0	2.50	7.50	55	142	800	3,200
I-4	5.0	3.50	7.50	55	142	800	3,200
I-5	5.0	1.50	7.50	55	142	800	3,200
I-6	5.0	2.50	10.00	55	142	800	3,200
I-7	5.0	2.50	5.00	55	142	800	3,200
I-8	5.0	2.50	7.50	75	142	800	3,200
I-9	5.0	2.50	7.50	35	142	800	3,200
I-10	5.0	2.50	7.50	55	180	800	3,200
I-11	5.0	2.50	7.50	55	100	800	3,200
I-12	5.0	2.50	7.50	55	142	1000	3,200
I-13	5.0	2.50	7.50	55	142	600	3,200
I-14	5.0	2.50	7.50	55	142	800	2,500
I-15	5.0	2.50	7.50	55	111	800	2,500
I-16	5.0	2.50	7.50	35	142	800	3,900
I-17	5.0	2.50	7.50	55	173	800	3,900
I-18	5.0	2.50	7.50	55	142	800	3,200

The results of all parametric test work are shown in Table 20.

Table 20. Parametric Testing of PDU Flotation Module - Indiana VII Coal

Test	Fuel Oil lb / ton	Frother lb / ton	% Sol	Air CFM	Wash GPM	Recirc GPM	Feed lb / hr	Column d80	PDU Yield	Energy Recov	Sulfur lb/MBtu	Ash lb/MBtu
I-7	4.97	2.53	8.99	55	142	800	3,200	22	87.8	94.7	0.42	3.04
I-14	4.97	0.73	5.70	55	142	800	3,200	21	11.9	13.2	N/A	1.81
I-15	4.97	1.13	6.29	55	142	800	3,200	22	57.2	62.3	N/A	1.95
I-16	5.04	0.94	6.57	55	111	800	2,500	18	19.8	21.7	N/A	1.93
I-17	4.97	2.53	7.30	55	142	800	3,200	22	86.9	94.3	N/A	2.54
I-18	2.97	2.53	7.11	55	142	800	3,200	20	86.0	92.9	N/A	2.29
I-19	6.97	2.53	7.04	55	142	800	3,200	23	87.5	94.6	N/A	2.50
I-20	4.97	1.53	7.10	55	142	800	3,200	23	53.5	58.6	N/A	1.89
I-21	4.97	2.99	7.21	55	142	800	3,200	22	86.9	94.4	N/A	2.39
I-22	4.97	2.53	4.14	55	142	800	3,200	23	65.9	71.9	N/A	1.90
I-23	4.97	2.53	7.40	75	142	800	3,200	22	88.6	94.8	N/A	3.25
I-24	4.97	2.53	7.52	35	142	800	3,200	22	78.4	85.0	N/A	2.49
I-25	Abort	Abort	Abort	Abort	Abort	Abort	Abort	Abort	Abort	Abort	Abort	Abort
I-26	4.97	2.53	7.09	55	100	800	3,200	22	87.8	94.5	N/A	2.54
I-27	4.97	2.53	7.28	55	142	990	3,200	21	89.7	96.4	N/A	2.57
I-28	4.97	2.53	7.25	55	142	600	3,200	22	65.8	71.9	N/A	2.07
I-29	5.04	2.47	5.89	55	142	800	2,500	21	79.5	86.3	N/A	2.43
I-30	5.04	2.47	4.67	55	111	800	2,500	19	83.7	91.3	N/A	2.59
I-31	4.98	2.51	7.94	55	142	800	3,900	27	84.1	91.6	N/A	2.65
I-32	4.98	2.51	7.79	55	173	800	3,900	25	83.1	88.8	N/A	3.01
MAX	6.97	2.53	8.99	75	173	990	3,900	27	89.7	96.4	0.42	3.25
MIN	2.97	0.73	4.14	35	100	600	2,500	18	12.0	13.2	0.42	1.81

Observation of the data shows that the overall quality goal of 2.00 lb ash/MBtu was achieved on five occasions (parametric tests I-14, I-15, I-16, I-20, and I-22).

Unfortunately, the overall PDU yield and energy recovery suffered significantly during these tests. Test I-25 was aborted due to insufficient wash water availability.

Overall, the clean coal yield varied from 12.0% to 89.7% while the energy recovery and product ash varied from 13.2% to 96.4% and 1.81 lb ash/MBtu to 3.25 lb ash/MBtu, respectively. Though the Microcel™ feed d_{80} varied from 18 to 27 microns, the large d_{80} values found in Tests I-31 and I-32 are the result of lower retention time in the ball mills (high feed rates). The results of these parametric tests are compared to the release analysis (d_{80} of 22 microns) in Figures 10 and 11. Observation of the graphs in these figures reveals that the desired clean coal quality goal of 2.0 lb ash/MBtu can be achieved at a theoretical yield and energy recovery of 73% and 81%, respectively.

Development of Regression Equations - Indiana VII Coal

Data from PDU Flotation Module parametric testing was compiled and multiple regression models (equations) were generated. Forward stepwise regression produced equations which link output variables such as yield and clean coal quality to input variables such as feed rate, wash water rate, air rate, collector addition, and frother addition. The equation term coefficients for the two main response variables are listed in Tables 21 and 22.

The Coefficient of Determination (R^2) and Adjusted Coefficient of Determination (Adjusted R^2) both show that each equation fits the data quite well. In addition, observation of the t-statistic, which indicates the relative importance of each independent variable to the response equation, shows that the most important variable that affects yield and product ash is frother dosage and air rate, respectively. It is also important to note that though the yield and product ash are also dependent on the feed coal ash content, the ash content itself is not a controllable variable and thus considered a covariate.

Table 21. Regression Analysis of Indiana VII Yield (%)

<u>Input Variable</u>	<u>Coefficient</u>	<u>t-Statistic</u>
Intercept	8,269.0	11.52
Collector lb/ton	7.3089	12.69
% Solids	-1.1019	-2.23
1 / Feed Rate lb/hr	-226370	-8.48
1 / (Frother lb/ton)	-69.336	-52.46
1 / (Air Rate CFM)	-8566.4	-9.56
ln (Air Rate CFM)	-159.60	-8.73
(Recirculation GPM) ^{1/2}	-18.088	-10.87
1 / Recirculation GPM	-229961	-13.60
(Feed Ash %) ²	-15.034	-8.18
1 / Feed Ash %	-32216	-7.99
(d80) ²	-1.52448	-20.10
1 / d80	-23211	-20.87
Coefficient of Determination (R ²)	0.989	
Adjusted R ²	0.987	

Table 22. Regression Analysis of Indiana VII Clean Coal Ash (lb/MBtu)

<u>Input Variable</u>	<u>Coefficient</u>	<u>t-Statistic</u>
Intercept	11.963	5.44
1 / Feed Rate lb/hr	2505	2.23
(Collector lb/ton) ²	0.006314	2.57
(Frother lb/ton) ²	-0.10371	-1.69
1 / (Frother lb/ton)	2.1090	2.43
ln (Frother lb/ton)	2.4188	2.64
(% Solids) ²	0.04262	5.93
ln (% Solids)	-2.4218	-3.91
(Air Rate CFM) ²	0.00058226	9.65
ln (Air Rate CFM)	-2.3263	-6.58
ln (Wash Water GPM)	-0.3158	-1.88
1 / (Recirculation GPM)	-729.5	-4.87
(Feed Ash %) ²	-0.002247	-0.46
(d80) ²	-0.000163	-0.26
Coefficient of Determination (R ²)	0.905	
Adjusted R ²	0.889	

Optimization of Flotation Column - Indiana VII Coal

Four tests were performed to determine the optimum Microcel™ setpoints needed to achieve the process development goals of 2.00 lb ash/MBtu and 80 percent energy recovery with maximum yield. Equations were first developed to estimate the effects of tested input variables on Microcel™ outputs such as yield and quality. The equations were developed by evaluating the input and output variables of parametric tests I-14 through I-32 as well as I-7 by multiple linear regression. The resulting equations were then used to determine optimum Microcel™ setpoints. A unique function found in the Microsoft Excel software package called "Solver" was used to determine the proposed optimal setpoints for testwork. The results of the optimization testwork are shown in Table 23.

Table 23. Optimization Testing of PDU Flotation Module - Indiana VII Coal

Test #	Fuel Oil lb / ton	Frother lb / ton	% Sol	Air CFM	Wash GPM	Recirc GPM	Feed lb / hr	Column d80	PDU Yield	Energy Recov	Sulfur lb/MBtu	Ash lb/MBtu
I-O-1	4.97	1.60	N/A	55	142	800	3,200	23	79.0	85.8	N/A	2.34
I-O-2	4.97	1.73	7.61	55	142	800	3,200	24	79.4	85.9	N/A	2.63
I-O-3	4.97	1.93	7.52	55	142	800	3,200	22	84.9	91.5	N/A	2.48
I-O-4	4.97	1.26	7.55	55	142	800	3,200	22	65.8	71.3	N/A	2.36
MAX	4.97	1.93	7.61	55	142	800	3,200	24	84.9	91.5	N/A	2.63
MIN	4.97	1.26	7.52	55	142	800	3,200	22	65.8	71.3	N/A	2.34

It can be seen from the table that the product quality goal of 2 lb ash/MBtu was not achieved. It is suspected that a buildup of frother in the clarified water circuit resulted in higher recovery of unwanted middlings material which in turn increased the clean coal yield and ash content. The frother buildup was visible as white foam on the clarified water tank surface and also at screen sprays.

The hypothesis is partially supported by comparing the results of two tests that were conducted at similar conditions. Except for a small difference in the metered frother dosage, Test 1-20 and optimization Test 1-0-1 were performed at similar conditions. Though similar performance values would be expected, the yield of optimization Test 1-0-1 was over 25 points higher than 1-20 while the product quality was 0.45 lb ash/MBtu higher.

Extended Production Run of PDU Flotation Module - Indiana VII Coal

An extended production run of the PDU Flotation Module was successfully completed during the month of July, 1996. The effort commenced Monday, July 22 and concluded the night of Wednesday, July 24. Like the Taggart production run, a failed belt splice was the only operational difficulty. Due to the extremely poor filtering characteristics of this coal, 16 hours each day was dedicated to operation of the PDU while the remaining 8 hours were used for filtering accumulated clean coal slurry. Though the operating parameters were changed during the first day, the resulting setpoints are listed below:

• Coal:	Indiana VII
• Nominal Feed Rate:	3,200 lb/hr
• Sizing Screen Cloth	270 mesh
• Grinding Circuit:	2" Cyclones / Screens / Netzsch Mill
• Primary Water:	8 GPM
• Cyclone Water:	12 GPM
• Ground Product H ₂ O:	40 GPM
• Collector:	5.00 lb/ton (72 cc/min)
• Frother:	1.375 lb/ton (21 cc/min)
• % Solids Setpoint:	5.50
• Microcel™ Dilution H ₂ O:	10 GPM
• Air Rate:	49 CFM
• Microcel™ Level SP:	55 inches
• Spray Water:	142 GPM
• Microcel™ Recirculation:	800 GPM
• Operating Hours:	48 hours
• Pounds Processed:	154,170
• Tons Processed:	77

A breakdown of the production run by time interval is shown in Table 24. Observation of the data shows that the clean coal ash quality, yield, and energy recovery increased during the second half of the production run. This increase is most likely attributable to the steady growth of the d_{80} values (reduced liberation of mineral matter) coupled with an apparent buildup of frother in the clarified water system.

Disposal of Clean Coal Product - Indiana VII Coal

Communications with DOE/PETC indicated that Penn State University (PSU) was interested in procuring the Indiana VII clean coal produced during the extended production run of the PDU Flotation Module. Due to its poor shipping characteristics, only one 2,000 pound super sack of Indiana VII was transported to Penn State. The remaining clean coal product was disposed at a local landfill.

Table 24. Extended Production Run - Indiana VII Coal

<u>Date</u>	<u>From</u>	<u>Until</u>	<u>Cumulative Hours</u>	<u>Feed lb/hr</u>	<u>Feed Solids</u>	<u>Feed Ash%</u>	<u>Tails Ash%</u>	<u>Prod d80</u>	<u>PDU Yield</u>	<u>Btu Rec %</u>	<u>lb Ash / MBtu</u>
7/22/96	07:00	11:00	4	3,200	N/S	N/S	N/S	N/S	N/S	N/S	N/S
7/22/96	11:00	15:00	8	3,200	6.96	10.80	41.94	25	80.6	88.0	2.38
7/22/96	15:00	19:00	12	3,200	6.96	9.95	28.26	17	72.5	78.6	2.17
7/22/96	19:00	23:00	16	3,200	7.34	10.89	25.65	18	65.1	71.4	2.13
7/23/96	07:00	11:00	20	3,200	N/S	N/S	N/S	N/S	N/S	N/S	N/S
7/23/96	11:00	15:00	24	3,200	4.40	10.67	33.06	N/S	74.6	81.6	2.21
7/23/96	15:00	19:00	28	3,200	4.52	10.34	28.41	19	71.1	77.5	2.17
7/23/96	19:00	23:00	32	3,200	4.61	10.74	27.81	18	69.0	75.6	2.22
7/24/96	07:00	11:00	36	3,200	4.39	10.58	33.46	28	77.5	83.8	2.87
7/24/96	11:00	15:00	40	3,200	4.38	10.41	37.55	26	79.0	86.0	2.32
7/24/96	15:00	19:00	44	3,200	4.47	10.66	41.98	28	80.9	88.2	2.36
7/24/96	19:00	23:00	48	3,200	4.42	10.60	42.72	27	81.7	88.9	2.46
AVG					5.25	10.56	34.08	23	75.2	82.0	2.33
MAX					7.34	10.89	42.72	28	81.7	88.9	2.87
MIN					4.38	9.95	25.65	17	65.1	71.4	2.13

Hiawatha Coal

Parametric testing of the Hiawatha coal in the PDU Flotation Module commenced in August, 1996 and was concluded during September, 1996. The operation of the PDU with Hiawatha coal was very successful with project goals achieved on numerous occasions.

Laboratory Release Analysis - Hiawatha Coal

Because the Hiawatha coal was not evaluated in the 12-inch Microcel™ during Subtask 4.4 (Hiawatha replaced Sunnyside), no data was available for indicating expected performance. One significant difference between these two coals was ash content, which was about 5.1% (dry basis) for the Sunnyside coal and ranged from 7.5% to 8.5% for the Hiawatha coal. (Sunnyside coal was washed at the mine but Hiawatha coal was not). As a result, to better define the theoretical grade-yield curves associated with different feed size distributions, laboratory release analysis test work was performed on the Hiawatha coal. Two Microcel™ feed slurries, one having a size distribution with d₈₀ of 54 microns and a second with d₈₀ of 49 microns, were evaluated. The results of the flotation test work are shown in Figures 12, 13, 14, and 15.

An analysis of the data shows that though the quality goal of 2.00 lb ash/MBtu can be achieved at both particle size distributions, there is a subtle change in the flotation characteristics when the d₈₀ is reduced from 54 microns to 49 microns. At a product quality value of 2.00 lb ash/MBtu, the yield improves from about 92% to 93% while the energy recovery rises from 98% to about 99%. However, at a product quality of 1.90 lb ash/MBtu, the yield increases from 89% to 92% while the energy recovery improves from about 94% to 98%. Overall, it can be concluded that grinding the Hiawatha coal to a d₈₀ of 49 microns typically improves the yield and energy recovery when operating near the "knee" of each curve.

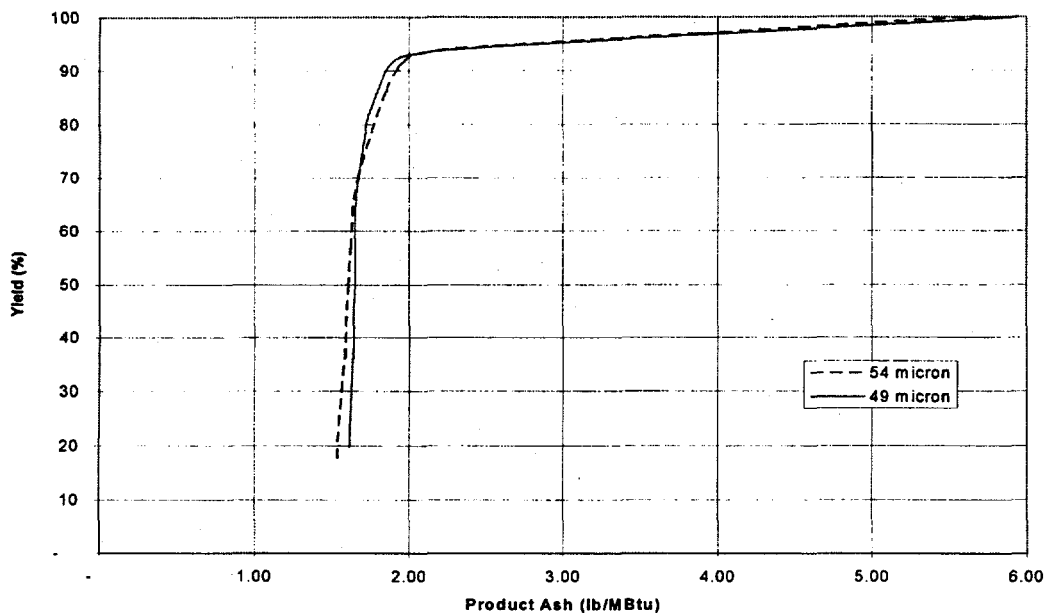


Figure 12. Hiawatha Release Analysis - Yield vs. Ash

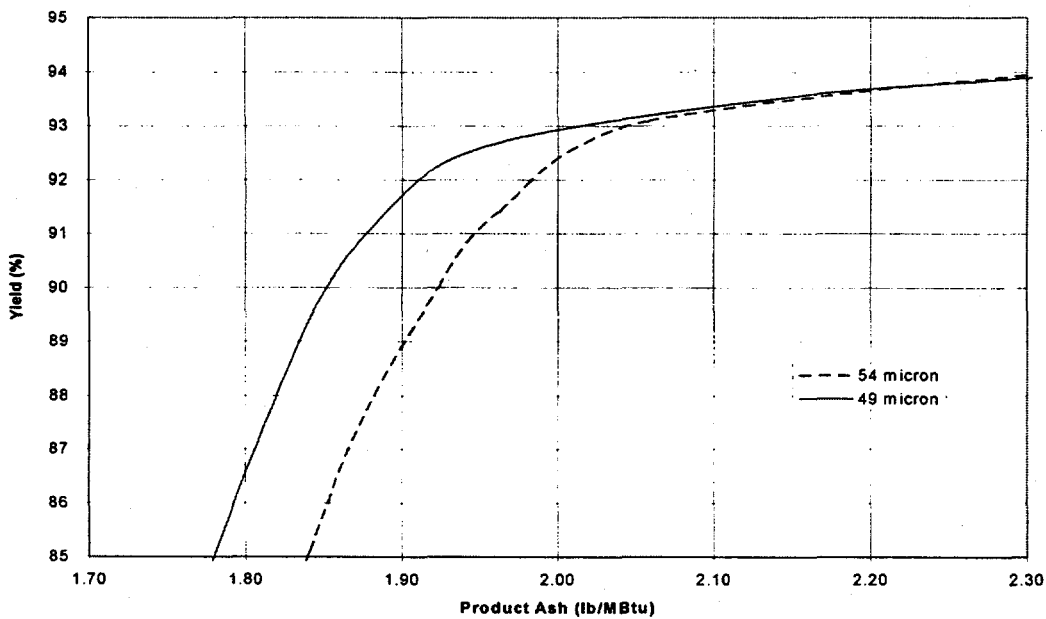


Figure 13. Hiawatha Release Analysis - Yield vs. Ash (Enlarged)

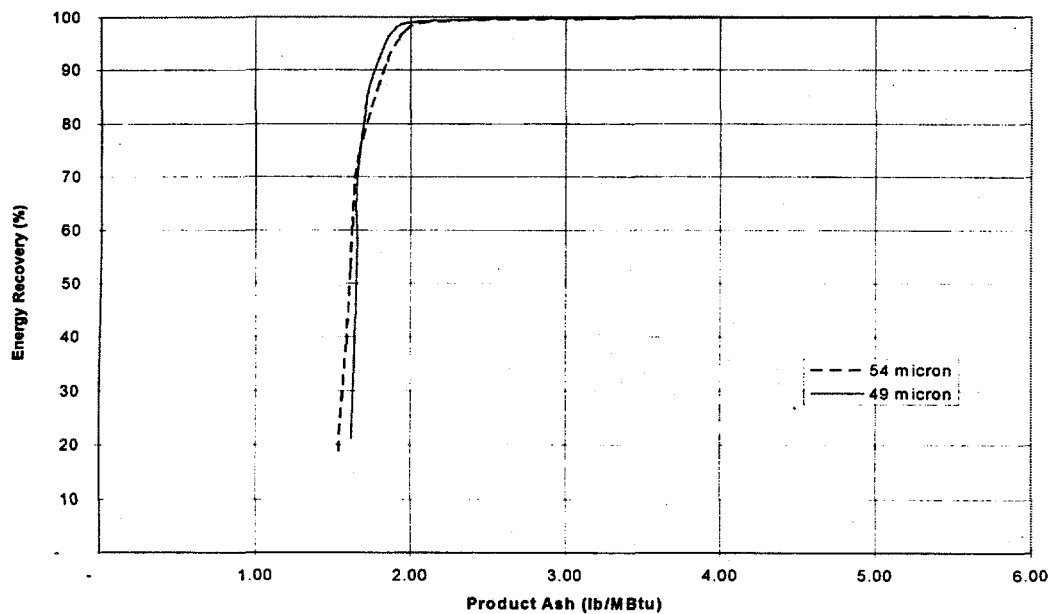


Figure 14. Hiawatha Release Analysis - Energy Recovery vs. Ash

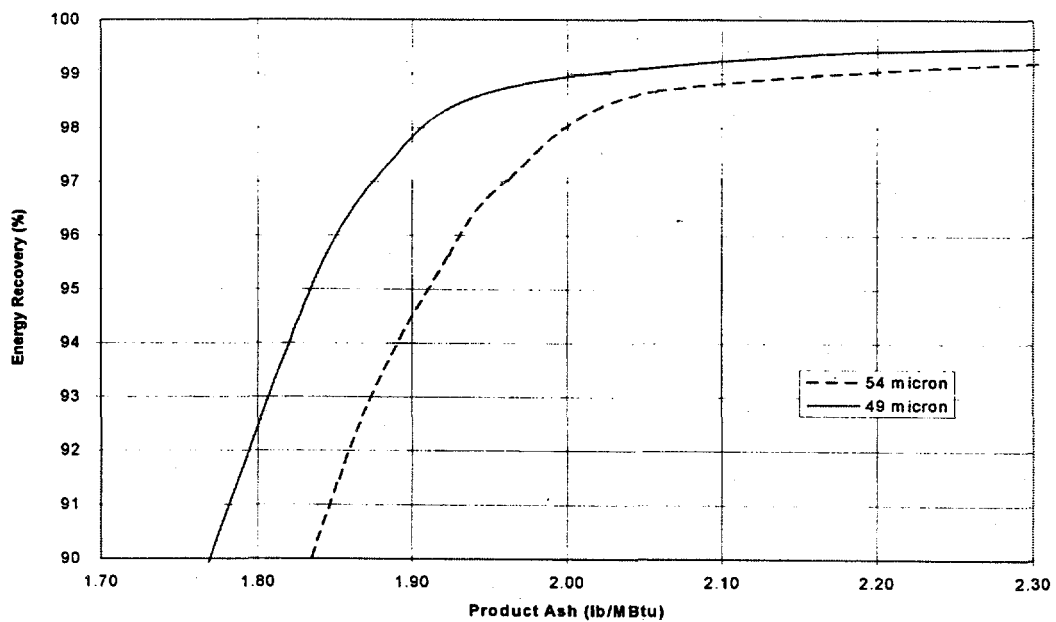


Figure 15. Hiawatha Release Analysis - Energy Recovery vs. Ash (Enlarged)

Testing and Optimization of Grinding Circuit - Hiawatha Coal

The testing was performed to determine the optimum grinding circuit arrangement needed to achieve adequate mineral liberation for the Hiawatha coal. Because the Hiawatha coal was not evaluated in the 12-inch Microcel™ during Subtask 4.4 (Hiawatha replaced Sunnyside), no data was available for indicating expected performance.

Release analysis of the Hiawatha coal suggested that the product ash quality goal of 2.00 lb/MBtu can be achieved with size distributions having d_{80} values of 54 and 49 microns, respectively. As a result, the two objectives of the grinding circuit testwork were:

- Determine optimum conditions to achieve a size distribution with d_{80} of 54 microns;
- Determine optimum conditions to achieve a size distribution with d_{80} of 49 microns;

Nineteen (19) tests, aimed at optimizing the grinding circuit, were conducted. The results of the testwork, which utilized approximately 125 tons of coal, are presented in Table 25.

Observation of the data shows that the grinding circuit was evaluated under four (4) general conditions:

- Mill loads at 8,100 lbs, 100 mesh screens, 4-inch cyclone and 3-inch cyclones;
- Mill loads at 8,100 lbs, 100 mesh screens, 3-inch cyclones only;
- Mill loads at 8,350 lbs, 145 mesh screens, 3-inch cyclones only;
- Mill loads at 9,100 lbs, 145 mesh screens, 2-inch cyclones only.

Condensing the tabular data to reflect these four conditions gave the results presented in Table 26. An evaluation of the data shows that each of the four conditions produced a ground product with varying d_{80} values. Maximum d_{80} values were obtained during tests H-3 through H-14 while a minimum d_{80} value was achieved during test H-19. Because the release analysis indicates improved yield and energy recovery at a d_{80} of 49 microns, and to ensure adequate liberation, the grinding conditions used in test H-19 was used for all parametric testwork of the Microcel™ flotation column.

Table 25. Parametric Testing of Grinding Circuit - Hiawatha Coal

Test #	Date	Feed Rate lb/hr	Primary Ball Load - lbs	Sec Ball Load - lbs	Screen Cloth Size	4-inch Cyclone	3-inch Cyclone	2-inch Cyclone	Wet Screen d80	Microtrac d80
H-1	8/5/96	4,300	8,100	8,100	100M	X	X			54
H-2	8/7/96	4,300	8,100	8,100	100M	X	X			58
H-3	8/8/96	4,300	8,100	8,100	100M		X			61
H-4	8/8/96	4,300	8,100	8,100	100M		X			59
H-5	8/12/96	4,300	8,100	8,100	100M		X			60
H-6	8/12/96	4,300	8,100	8,100	100M		X			62
H-7	8/13/96	4,300	8,100	8,100	100M		X			57
H-8	8/13/96	4,300	8,100	8,100	100M		X			59
H-9	8/13/96	4,300	8,100	8,100	100M		X			61
H-10	8/14/96	4,300	8,100	8,100	100M		X			57
H-11	8/14/96	4,300	8,100	8,100	100M		X			56
H-12	8/14/96	4,300	8,100	8,100	100M		X			59
H-13	8/21/96	4,300	8,100	8,100	100M		X		53	58
H-14	8/22/96	4,300	8,100	8,100	100M		X			
H-15	8/26/96	4,300	8,350	8,350	145M		X		54	
H-16	8/26/96	4,300	8,350	8,350	145M		X		53	
H-17	8/26/96	4,300	8,350	8,350	145M		X		52	
H-18	8/27/96	4,300	8,350	8,350	145M		X		51	
H-19	8/28/96	4,300	9,100	9,083	145M			X	45	
Avg									51	58
Max									54	62
Min									45	54

Table 26. Parametric Testing of Hiawatha Grinding Circuit - Condensed

Test #	Feed Rate	Prim Ball Load	Sec Ball Load	Screen Cloth Size	4-inch Cyclone	3-inch Cyclone	2-inch Cyclone	Average d80	Maximum d80	Minimum d80
H1 - H2	4,300	8,100	8,100	100 M	X	X		56	58	54
H3 - H14	4,300	8,100	8,100	100 M		X		59	62	56
H15 - H18	4,300	8,350	8,350	145 M		X		52	54	51
H19	4,300	9,100	9,083	145 M			X	45	45	45

Parametric Testing of Microcel™ Flotation Column - Hiawatha Coal

Parametric testing of Hiawatha coal in the PDU flotation column was concluded in September, 1996. A total of 20 tests were conducted which utilized approximately 124 tons of feed coal. The results of these tests are presented in Table 27.

An inspection of the data shows that the overall quality goal of 2.00 lb ash/MBtu was achieved on two occasions (parametric tests H-19 and H-20). Unfortunately, the overall PDU yield and energy recovery suffered significantly during one of these tests.

Overall, the clean coal yield varied from 12.3% to 94.0% while the energy recovery and product ash varied from 13.8% to 98.7% and 1.43 lb ash/MBtu to 2.87 lb ash/MBtu, respectively. The Microcel™ feed d_{80} values averaged 50 microns with a standard deviation of 3 microns. The results of these parametric tests are compared to the release analysis (d_{80} of 49 microns) in Figures 16 and 17.

Development of Regression Equations - Hiawatha Coal

Data from PDU Flotation Module parametric testing was compiled and multiple regression models (equations) were generated. Like the Taggart and Indiana VII coals, forward stepwise regression produced equations which link output variables such as yield and clean coal quality to input variables such as feed rate, wash water rate, air rate, collector addition, and frother addition. The equation term coefficients for the two main response variables are shown in Tables 28 and 29.

The Coefficient of Determination (R^2) and Adjusted Coefficient of Determination (Adjusted R^2) both show that each equation fits the data quite well. In addition, observation of the t-statistic, which indicates the relative importance of each independent variable to the response equation, shows that the most important controllable variables that affect clean coal ash are Recirculation Rate (affects bubble size) and Wash Water. However, the variables that have the most significant impact on yield are Feed Ash % and Frother Dosage.

It is important to note that though the yield and product ash are dependent on the feed coal ash content, the ash content itself is not a controllable variable and thus considered a covariate.

Table 27. Parametric Testing of PDU Flotation Module - Hiawatha Coal

Test #	Feed lb/hr	Fuel Oil lb / ton	Frother lb / ton	% Solids	Air CFM	Wash GPM	Recirc GPM	Column d80	PDU Yield	Energy Recov	Ash lb/MBtu
H-19	4,304	0.25	0.30	4.27	55	140	800	45	12.3	13.8	1.43
H-20	4,304	0.50	0.30	4.50	55	140	800	49	74.5	80.2	1.69
H-21	4,304	0.50	0.50	6.38	55	86	800	49	89.8	96.5	2.40
H-22	4,306	0.25	0.75	3.78	55	86	800	52	90.8	97.2	2.40
H-23	4,306	0.75	0.75	6.60	55	86	800	52	91.5	98.0	2.51
H-24	4,306	0.50	1.00	6.67	55	86	800	49	90.3	97.6	2.31
H-25	4,302	0.50	0.75	9.99	55	86	800	54	91.1	97.8	2.74
H-26	4,302	0.50	0.75	4.44	55	86	800	47	91.8	97.6	2.30
H-27	4,308	0.50	0.75	7.17	75	86	800	50	92.7	98.4	2.63
H-28	4,305	0.50	0.75	7.50	30	86	800	52	90.6	96.7	2.54
H-29	4,295	0.50	0.75	7.42	55	140	800	53	89.0	96.5	2.17
H-30	4,295	0.50	0.75	7.04	55	55	800	47	94.0	98.7	2.87
H-31	4,305	0.50	0.75	7.71	55	86	990	54	90.7	97.4	2.37
H-32	4,305	0.50	0.75	7.35	55	86	600	48	89.8	95.3	2.14
H-33	4,308	0.50	0.35	7.07	55	86	800	51	90.3	96.3	2.16
H-34	4,308	0.50	0.40	7.15	55	86	800	46	90.6	96.8	2.30
H-35	4,308	0.50	0.45	7.43	55	86	800	45	90.5	96.6	2.38
H-36	5,973	0.50	0.75	8.63	55	86	800	61	92.5	97.2	2.41
H-37	5,333	0.50	0.75	6.14	55	86	800	56	90.4	96.6	2.35
H-38	3,307	0.50	0.75	6.02	55	86	800	49	91.8	97.5	2.39
MAX	5,973	0.75	1.00	9.99	75	140	990	61	94.0	98.7	2.87
MIN	3,307	0.25	0.30	4.27	30	55	600	45	12.3	13.8	1.43

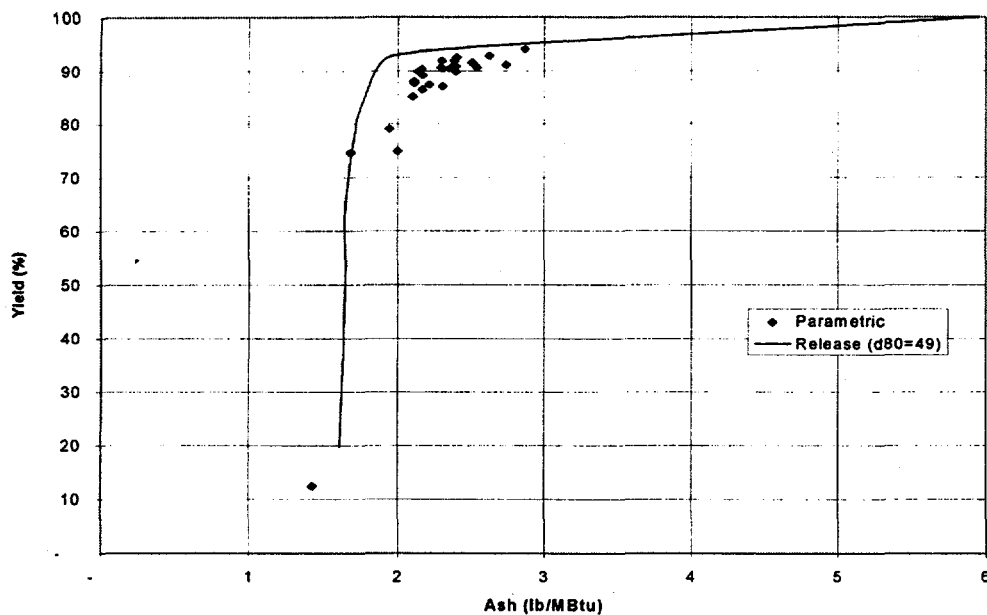


Figure 16. Parametric Testing of Hiawatha Coal - Yield vs. Ash

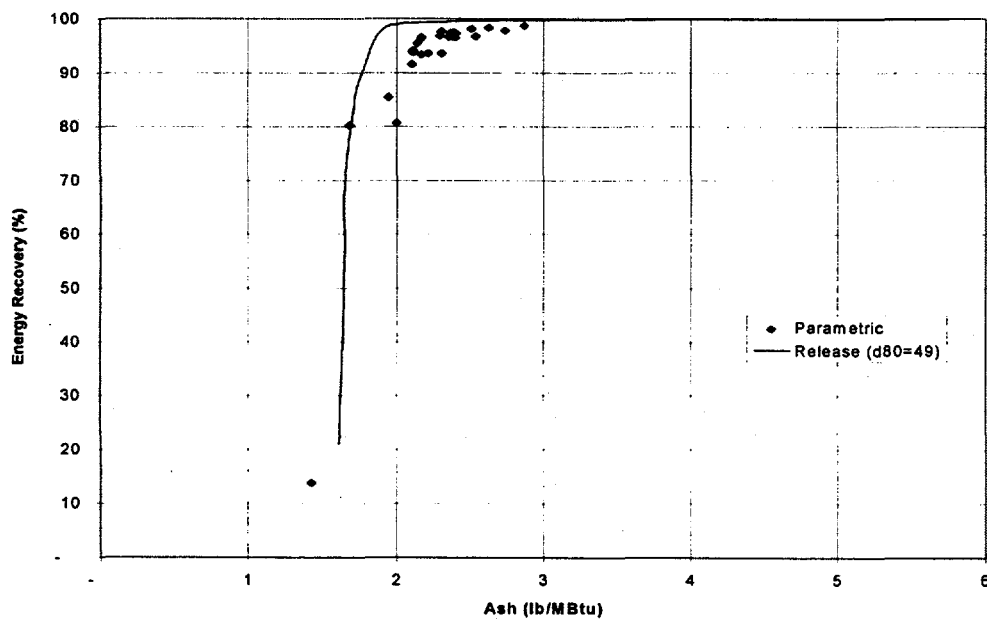


Figure 17. Parametric Testing of Hiawatha Coal - Energy Recovery vs. Ash

Optimization of PDU Flotation Column - Hiawatha Coal

A total of eight tests were performed to determine the optimum Microcel™ setpoints needed to achieve the process development goals of 2.00 lb ash/MBtu and 80 percent energy recovery with maximum yield. Equations were first developed to estimate the effects of test input variables on Microcel™ outputs such as yield and quality. The equations were developed by evaluating the input and output variables of parametric tests H-19 through H-38 by multiple linear regression. The resulting equations were then used to determine optimum Microcel™ setpoints. A unique function found in the Microsoft Excel software package called “Solver” was used to determine the proposed optimal setpoints for testwork. The results of the optimization testwork are shown in Table 30.

Observation of the data shows that the product quality goal of 2.00 lb ash/MBtu was achieved on two occasions (H-OP-44 and H-OP-46). Because test H-OP-46 used a lower frother dosage, the setpoints used during this test were used for the extended production run.

Extended Production Run of PDU Flotation Module - Hiawatha Coal

The extended production run of the PDU Flotation Module on Hiawatha coal was successfully completed during the week of September 23, 1996. The PDU operated 72 continuous hours without interruption. Though the operating parameters were changed during the first day, the resulting setpoints are listed below:

- | | |
|--|--------------------------------------|
| • Coal | Hiawatha |
| • Nominal Feed Rate: | 4,300 lb/hr |
| • Sizer Screen Cloth | 145 mesh |
| • Grinding Circuit: | Closed / 2" Cyclones / Sizer Screens |
| • Primary Water: | 8 GPM |
| • Cyclone Water: | 20 GPM |
| • Ground Product H ₂ O: | 20 GPM |
| • Collector: | 0.45 lb/ton (9 cc/min) |
| • Frother: | 0.35 lb/ton (7 cc/min) |
| • % Solids Setpoint: | 5.24 |
| • Microcel™ Dilution H ₂ O: | 20 GPM |
| • Air Rate: | 51 CFM |
| • Microcel™ Level SP: | 55 inches |
| • Spray Water: | 140 GPM |

- Microcel™ Recirculation: 810 GPM
- Operating Hours: 72 hours
- Pounds Processed: 310,270
- Tons Processed: 155
- Clean Filter Cake Bags: 131

A breakdown of the production run by time interval is shown in Table 31

Except for % Solids and d_{80} , the average values shown are weighted averages based on feed rate and yield. Values listed as AVG #1 do not include the first four hours of operation while AVG #2 values do not include the first eight hours of operation. Based on the fact that some parameters were varied during the first eight hours of the production run, the resulting values obtained during this time period should be omitted. As a result, the Hiawatha production run produced a clean coal product with the following average values:

- Ash: 1.89 lb/MBtu
- Sulfur: 0.44 lb/MBtu
- Yield: 81.8 %
- Energy Recovery: 88.0 %

Disposal of Clean Coal Product - Hiawatha Coal

Communications with DOE/PETC indicated that Penn State University (PSU) was interested in procuring the Hiawatha clean coal produced during the extended production run of the PDU Flotation Module. Overall, 131 bags (supersacks) were produced during the Hiawatha production run. Of this amount, 44 bags were shipped to Penn State with the remainder disposed at a local landfill.

CLEAN COAL ASH PROPERTIES

Hazen Research Inc., Golden, CO, determined the ash chemistry and fusion properties of feed and clean coal samples from the extended PDU production runs utilizing the Taggart, Indiana VII, and Hiawatha coals. It was found that the PDU Microcel™ flotation consistently increased the base/acid ratio of the ash and decreased the silica/alumina ratio. The overall results were substantial declines in the reducing atmosphere fusion temperatures of the ash in the Taggart and Indiana VII coals, and a smaller decline in the fusion temperatures of the ash in the Hiawatha coal. The softening (spherical) temperatures are compared in Figure 18 to illustrate the difference caused by the cleaning. The complete set of fusion temperatures are listed in Table 32.

Table 28. Regression Analysis of Hiawatha Clean Coal Ash (lb/MBtu)

<u>Input Variable</u>	<u>Coefficient</u>	<u>t-Statistic</u>
Intercept	80.106	21.63
% Solids	0.075829	17.26
(Frother lb/ton) ²	-1.8928	-10.28
(Air Rate CFM) ²	0.00002260	4.24
(Wash Water GPM) ^{1/2}	-0.144977	-21.27
1 / (Collector lb/ton)	-0.062601	-7.43
1 / (Frother lb/ton)	0.7819	5.53
1 / (Recirculation GPM)	-7896.6	-21.50
1 / (Feed Ash %)	1.8427	2.27
ln (Frother lb / ton)	3.0606	7.91
ln (Recirculation GPM)	-9.8943	-20.32
Coefficient of Determination (R ²)	0.991	
Adjusted R ²	0.989	

Table 29. Regression Analysis of Hiawatha Coal - Yield (%)

<u>Input Variable</u>	<u>Coefficient</u>	<u>t-Statistic</u>
Intercept	-442.10	-10.40
Feed Ash %	152.092	20.45
(Feed Ash %) ²	-8.8689	-22.10
(Air Rate CFM) ^{1/2}	-8.694	-4.13
1 / (Collector lb/ton)	-1.8587	-3.41
1 / (Frother lb/ton)	-5.9086	-12.78
1 / (% Solids)	-75.07	-7.26
1 / (Air Rate CFM)	-1485.3	-4.82
Coefficient of Determination (R ²)	0.987	
Adjusted R ²	0.985	

Table 30. Optimization Testing of PDU Flotation Module - Hiawatha Coal

<u>Test</u>	<u>Feed lb / hr</u>	<u>Fuel Oil lb / ton</u>	<u>Frother lb / ton</u>	<u>% Solids</u>	<u>Air CFM</u>	<u>Wash GPM</u>	<u>Recirc GPM</u>	<u>Column d80</u>	<u>PDU Yield%</u>	<u>Energy Recov%</u>	<u>Ash lb/MBtu</u>
H-OP-39	4,280	0.45	0.35	4.50	51	117	810	50	87.3	93.5	2.21
H-OP-40	4,280	0.45	0.40	4.37	51	117	810	52	88.0	94.0	2.11
H-OP-41	4,301	0.45	0.45	5.04	51	117	810	56	87.6	93.8	2.11
H-OP-42	4,301	0.45	0.45	5.09	51	140	810	51	86.4	93.4	2.17
H-OP-43	4,301	0.45	0.40	5.09	51	140	810	50	87.0	93.6	2.31
H-OP-44	4,306	0.45	0.30	5.37	51	140	810	52	78.9	85.4	1.95
H-OP-45	4,306	0.45	0.35	5.20	51	140	810	52	85.1	91.6	2.11
H-OP-46	4,292	0.45	0.25	5.01	51	140	810	49	74.8	80.6	2.00
MAX	4,306	0.45	0.45	5.37	51	140	810	56	88.0	94.0	2.31
MIN	4,280	0.45	0.25	4.37	51	117	810	49	74.8	80.6	1.95

Table 31. Extended Production Run - Hiawatha Coal

<u>Date</u>	<u>From</u>	<u>Until</u>	<u>Feed lb/hr</u>	<u>% Solid</u>	<u>Feed Ash %</u>	<u>Clean Ash %</u>	<u>Tails Ash %</u>	<u>Feed d80</u>	<u>PDU Yield</u>	<u>Energy Recov</u>	<u>Sulfur lb/MBtu</u>	<u>ASH lb/MBtu</u>
9/23/96	7 AM	11 AM	4,309	N/S	N/S	N/S	N/S	N/S	N/S	N/S	N/S	N/S
9/23/96	11 AM	3 PM	4,309	4.78	8.16	2.44	16.25	48	58.6	62.8	0.44	1.70
9/23/96	3 PM	7 PM	4,309	4.67	8.39	2.58	25.68	51	74.9	80.4	0.44	1.80
9/23/96	7 PM	11 PM	4,309	4.73	8.57	2.92	43.89	52	86.2	92.4	0.45	2.05
9/23/96	11 PM	3 AM	4,309	5.68	8.09	2.28	20.68	46	68.4	73.5	0.45	1.59
9/24/96	3 AM	7 AM	4,309	5.76	8.32	2.41	26.15	50	75.1	80.7	0.43	1.68
9/24/96	7 AM	11 AM	4,309	5.87	10.56	2.63	35.99	47	76.2	84.1	0.45	1.84
9/24/96	11 AM	3 PM	4,309	5.16	8.82	2.87	38.53	47	83.3	89.7	0.44	2.01
9/24/96	3 PM	7 PM	4,309	5.22	7.82	3.08	36.57	50	85.9	91.0	0.45	2.16
9/24/96	7 PM	11 PM	4,309	5.47	8.88	2.69	39.31	46	83.1	89.7	0.44	1.88
9/24/96	11 PM	3 AM	4,309	5.29	7.68	2.65	37.17	48	85.4	90.8	0.44	1.85
9/25/96	3 AM	7 AM	4,309	5.31	8.71	2.78	44.21	50	85.7	92.2	0.45	1.95
9/25/96	7 AM	11 AM	4,309	5.61	9.34	2.75	46.22	46	84.8	92.0	0.45	1.92
9/25/96	11 AM	3 PM	4,309	5.07	8.93	2.75	45.53	50	85.6	92.3	0.43	1.92
9/25/96	3 PM	7 PM	4,309	5.26	8.80	2.71	42.71	50	84.8	91.4	0.43	1.90
9/25/96	7 PM	11 PM	4,309	5.34	9.10	2.57	33.98	48	79.2	85.8	0.44	1.79
9/25/96	11 PM	3 AM	4,309	5.24	8.27	2.66	40.06	44	85.0	91.1	0.45	1.86
9/26/96	3 AM	7 AM	4,309	5.12	9.04	2.75	43.65	51	84.6	91.4	0.45	1.92
AVG #1				5.27	8.68	2.69	33.22	48	80.4	86.5	0.44	1.88
AVG #2				5.30	8.71	2.70	35.63	48	81.8	88.0	0.44	1.89
MAX				5.87	10.56	3.08	46.22	52	86.2	92.4	0.45	2.16
MIN				4.67	7.68	2.28	16.25	44	58.6	62.8	0.43	1.59

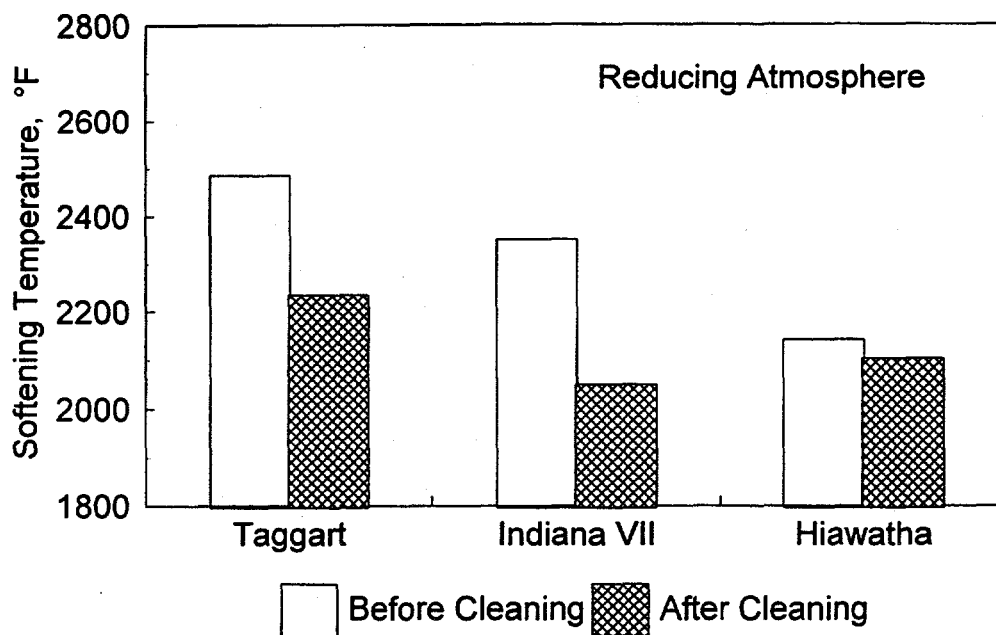


Figure 18. Softening Temperatures of Ash in Test Coals

Table 32. Ash Fusion Temperatures (°F) Before and After Cleaning

	Taggart Coal (Test T-40)		Indiana VII Coal (Test I-EX)		Hiawatha Coal (Test H-EX)	
	Before Cleaning	After Cleaning	Before Cleaning	After Cleaning	Before Cleaning	After Cleaning
Oxidizing Atmosphere:						
Initial	2570	2550	2365	2350	2210	2350
Softening	2630	2562	2420	2385	2255	2385
Hemispherical	2650	2575	2482	2390	2310	2390
Fluid	2702	2590	2512	2420	2500	2420
Reducing Atmosphere:						
Initial	2305	2130	2315	2025	2110	2050
Softening	2485	2235	2350	2050	2141	2102
Hemispherical	2575	2435	2375	2055	2274	2117
Fluid	2642	2513	2400	2060	2482	2135

The ash compositions of the coals are presented in Table 33 along with slag viscosity calculations and assessments of the slagging and fouling characteristics of the ash. The calculated viscosities agree with the fusion temperature measurements. Except for titania, and iron oxide in the case of the Taggart coal, the concentrations of the ash constituents were significantly reduced on a heating value (lb/MBtu) basis, by advanced flotation cleaning in the PDU.

Table 33. Ash Chemistry of Test Coals Cleaned by Column Flotation

Ash Constituent, %:	Taggart Coal			Indiana VII Coal			Hiawatha Coal		
	Before Cleaning	After Cleaning	Reduction Percent*	Before Cleaning	After Cleaning	Reduction Percent*	Before Cleaning	After Cleaning	Reduction Percent*
SiO ₂	49.43	41.43	59	54.00	46.44	76	51.32	40.28	77
Al ₂ O ₃	27.43	27.72	50	21.53	23.04	70	13.61	20.05	56
TiO ₂	1.10	1.65	26	0.99	3.09	13	0.79	2.02	24
Fe ₂ O ₃	9.50	17.92	7	6.58	11.92	49	5.44	9.36	49
CaO	1.82	1.95	47	3.43	3.71	70	11.00	9.22	75
MgO	0.88	0.61	66	0.72	1.03	60	1.37	1.20	74
Na ₂ O	1.35	0.77	72	0.98	1.21	66	1.91	3.90	39
K ₂ O	2.86	2.17	63	2.65	3.43	64	0.73	0.71	71
P ₂ O ₅	0.28	0.29	49	0.14	0.23	54	0.34	0.75	34
SO ₃	1.23	0.99	60	4.24	1.68	89	9.30	11.10	64
Ash Viscosity Calculations:									
Base Content, %	17.33	24.98		15.80	22.69		23.73	28.12	
Acid Content, %	82.67	75.14		84.20	77.31		76.27	71.88	
Dolomite Ratio	16.45	10.93		28.90	22.25		60.49	42.72	
Base/Acid Ratio	0.21	0.33		0.19	0.29		0.31	0.39	
Silica/Alumina Ratio	1.81	1.49		2.51	2.02		3.77	2.01	
T(cv), °F	2775	2635		2575	2255		2474	2317	
T250 Temp, °F	2695	2495		>2800	2550		2531	2416	
Equiv. Silica, %	80.30	66.92		83.42	73.60		74.24	67.07	
Viscosity at 2600 °F, P	878	92		>1000	270		300	94	
Ash Type	High Rank	High Rank		High Rank	High Rank		Lignite	Lignite	
Slagging/Fouling Characteristics:									
Slagging Type	--	--		--	--		Medium	Medium	
Fouling Type	Medium	Medium		Low	Medium		Low	Medium	

* Percentage reduction calculated on a heating value (lb/MBtu) basis.

TOXIC TRACE ELEMENTS REDUCTION

Samples of the crushed feed coal, ground feed coal, clean coal, and fine refuse from the extended PDU production runs utilizing the Taggart, Indiana VII, and Hiawatha coals were submitted to Huffman Laboratories, Golden, CO, for determination of the concentrations of twelve toxic trace elements. The toxic trace elements were antimony, arsenic, beryllium, cadmium, chromium, cobalt, lead, manganese, mercury, nickel, selenium, and chlorine. The perchloric acid dissolution/atomic absorption, total halides, and cold-vapor spectroscopy methods used to analyze these samples were the same as the methods used to analyze the samples from the bench-scale testing [13] [14]. The analytical results for the clean coals, as-received test coals, and run-of mine (ROM) coals are presented in Table 34.

The variations in trace element concentrations from coal to coal seen for these samples were similar to the variations seen in the bench-scale testing samples. As shown in Table 34, there were substantial reductions, over 25% on a heating value basis, in the residual concentrations of arsenic, lead, and chlorine for all three as-received test coals. The reduction in the concentrations of beryllium, cadmium, chromium, cobalt, manganese, mercury, nickel, and selenium varied from coal to coal. Flotation did not appear to reduce the heating value basis concentration of antimony in any of the coals.

Reduction in trace-element concentrations from the amounts in the parent Taggart and Indiana VII ROM coals was greater on a heating value basis than the reduction from the amounts in the as-received test coals. The residual concentrations of arsenic, beryllium, cadmium, chromium, cobalt, lead, manganese, mercury, and selenium in the two clean coals were especially lower than their concentrations in the two ROM coals. Mixed results were seen for nickel and chlorine, and the concentration of antimony did not appear to have been reduced at all by the combination of preparation plant cleaning and column flotation. Except for chlorine, the rejection of the toxic trace elements from the Taggart and Indiana VII ROM coals was greater than their rejection from the Hiawatha test coal even though the Hiawatha coal had not been washed prior to flotation in the PDU.

Table 34. Toxic Trace Elements in Coals

	Analyses, % or PPM			Reduction (Heating Value Basis)*, %	
	Clean Coal	As-Recv'd Test Coal	ROM Coal	From As-Recv'd Test Coal	From ROM Coal
Taggart Coal:					
Ash, %	1.83	3.64	34.70	51	97
S(tot), %	0.72	0.75	0.46	6	0
S(pyr), %	0.08	0.10	0.02	22	neg
Sb, PPM	0.60	0.49	0.17	neg	neg
As, PPM	2.14	3.50	2.47	40	45
Be, PPM	2.2	2.3	2.0	87	29
Cd, PPM	< 0.1	0.1	0.1	6	> 36
Cr, PPM	9	7.1	30	neg	81
Co, PPM	9	9.5	12	7	52
Pb, PPM	3.5	5	38	32	94
Mn, PPM	9	13	110	32	95
Hg, PPM	0.02	0.02	0.03	2	57
Ni, PPM	11	12	11	10	36
Se, PPM	1.02	1.25	1.39	20	53
Cl, PPM	75	269	177	73	73
Indiana VII Coal:					
Ash, %	3.23	10.46	38.10	72	95
S(tot), %	0.59	0.86	0.77	37	54
S(pyr), %	0.13	0.39	0.51	69	84
Sb, PPM	2.81	1.92	1.2	neg	neg
As, PPM	1.91	2.56	4.1	31	72
Be, PPM	2.8	2.6	2.3	1	27
Cd, PPM	< 0.1	0.3	0.1	> 70	> 38
Cr, PPM	13	13	22	9	63
Co, PPM	8.1	8.1	11	8	55
Pb, PPM	9	13	14	37	60
Mn, PPM	17	42	150	62	93
Hg, PPM	0.02	0.02	0.02	9	38
Ni, PPM	38	34	30	neg	23
Se, PPM	0.41	0.50	0.78	24	68
Cl, PPM	133	312	38	61	neg
Hiawatha Coal**:					
Ash, %	2.70	8.57		71	
S(tot), %	0.63	0.69		16	
S(pyr), %	0.11	0.19		46	
Sb, PPM	0.09	0.07		neg	
As, PPM	0.39	0.64		43	
Be, PPM	0.3	0.3		7	
Cd, PPM	0.1	< 0.1		neg	
Cr, PPM	4.4	5.2		22	
Co, PPM	0.6	0.4		neg	
Pb, PPM	< 2	< 2			
Mn, PPM	4	6		38	
Hg, PPM	0.02	0.02		7	
Ni, PPM	1	< 1		neg	
Se, PPM	0.81	0.84		11	
Cl, PPM	234	310		30	

* neg = negative number

** Hiawatha ROM coal will be the same as the as-received test coal

MICROCEL™ SCALE-UP TESTWORK

To better understand and determine the similitude between the 12-inch Microcel™ unit and the 6-foot Microcel™ unit, comparative tests were conducted on the 12-inch unit at conditions similar to those used in production runs and parametric testing. The results are discussed below.

Scale-up of Microcel™ Flotation Column - Taggart Coal

Because the Taggart coal that was used in the PDU Flotation Module has a higher ash content than that used during the parametric testing of the bench-scale 12-inch column (4% versus 2%), the similitude for scale-up would be inaccurate. As a result, six tests were performed on the Taggart coal in the 12-inch Microcel™ unit. The parameters of the six tests correspond directly to those used in optimization tests TO-2 through TO-7. The results are provided in Tables 35 and 36.

Analysis of the data shows that the clean coal quality goal of 1 lb ash/MBtu was achieved during all 12-inch Microcel™ tests. However, when compared to the identical tests conducted in the 6-foot Microcel™, different results were obtained. This difference is depicted more clearly in Figure 19.

Though both units produce results that fall on the same grade-yield curve, Figure 19 clearly illustrates that the 6-foot Microcel™ produces clean coal at a higher yield and ash than the 12-inch unit. Discussions with Mr. Dennis Phillips of Virginia Tech revealed that the discrepancy is most likely attributable to the frother addition point.

The addition of frother into the suction side of the Microcel™ recirculation pump (6-foot column) is typically more efficient than adding frother in the slurry feed mixing tank (1-foot column). Specifically, when frother is added to the pump suction it is closest to the point where it is needed most - the air injection point. The result is a smaller bubble size for a constant frother addition rate. The smaller bubbles, which have greater surface area than larger bubbles, are now capable of carrying more clean coal particles to the overflow.

As a result, the discrepancy in performance between the two columns is not unexpected. The addition of frother to the 12-inch column slurry mix tank resulted in large bubbles (about 2 mm) with low carrying capacity and high selectivity. The result was a low-yield, high-quality product. However, the addition of frother to the recirculation suction line of 6-foot column resulted in smaller bubbles (1 mm) with high carrying capacity and low selectivity. The result was a high-yield, low-quality product.

Table 35. Scale Up Testing of 1-Foot Microcel™ Column - Taggart Coal

Test	Fuel Oil lb / ton	Frother lb / ton	% Solids	Air CFM	Wash GPM	Feed lb / hr	% Yield	Energy Recov (%)	Ash lb/MBtu
T12-01	0.26	0.40	6.93	2.08	2.44	120	91.15	94.18	1.00
T12-02	0.26	0.33	6.89	1.53	2.35	120	72.29	74.76	0.82
T12-03	0.26	0.24	6.73	2.08	2.10	117	84.78	87.78	0.98
T12-04	0.48	0.23	7.00	2.08	2.10	122	90.96	93.89	0.99
T12-05	0.20	0.17	7.83	1.53	2.16	137	74.43	76.68	0.85
T12-06	0.23	0.22	7.55	1.53	2.16	132	76.76	78.98	0.84
MAX	0.48	0.40	7.83	2.08	2.44	137	91.15	94.18	1.00
MIN	0.20	0.17	6.73	1.53	2.10	117	72.29	74.76	0.82

Table 36. Performance of 1-foot and 6-foot Microcel™ - Taggart Coal

Parameter	Series #1		Series #2		Series #3		Series #4		Series #5		Series #6	
	12 in	6 ft	12 in	6 ft	12 in	6 ft	12 in	6 ft	12 in	6 ft	12 in	6 ft
Feed, lb/hr	120	4,200	120	4,200	117	4,200	122	4,200	137	4,200	132	4,200
Solids Conc., %	6.93	5.81	6.89	6.16	6.73	7.72	7.00	6.24	7.83	7.14	7.55	7.70
Fuel Oil, lb/ton	0.26	0.26	0.26	0.26	0.26	0.26	0.48	0.53	0.20	0.26	0.23	0.26
Frother, lb/ton	0.40	0.41	0.33	0.30	0.24	0.25	0.23	0.25	0.17	0.20	0.22	0.25
Yield, %	91.15	95.26	72.29	92.69	84.78	92.34	90.96	95.39	74.43	86.63	76.76	85.46
Feed Ash, %	4.45	4.27	4.26	4.27	4.60	5.26	4.35	4.73	3.97	3.63	3.84	3.86
CC Ash, %	1.51	1.74	1.24	1.59	1.48	1.86	1.50	1.98	1.28	1.33	1.27	1.46
CC Ash, lb/MBtu	1.00	1.16	0.82	1.05	0.98	1.24	0.99	1.32	0.85	0.88	0.84	0.97
Tails Ash, %	34.73	42.23	12.14	34.68	21.98	37.89	33.03	48.82	11.80	13.97	12.33	16.38
Product, lb/hr/ft ²	139	142	111	138	127	137	141	142	130	129	129	127
Air Vel, ft/min	2.65	2.65	1.95	1.95	2.65	2.65	2.65	2.65	1.95	1.95	1.95	1.95
H ₂ O Vel, ft/min	0.42	0.36	0.40	0.36	0.36	0.36	0.36	0.36	0.37	0.36	0.37	0.36

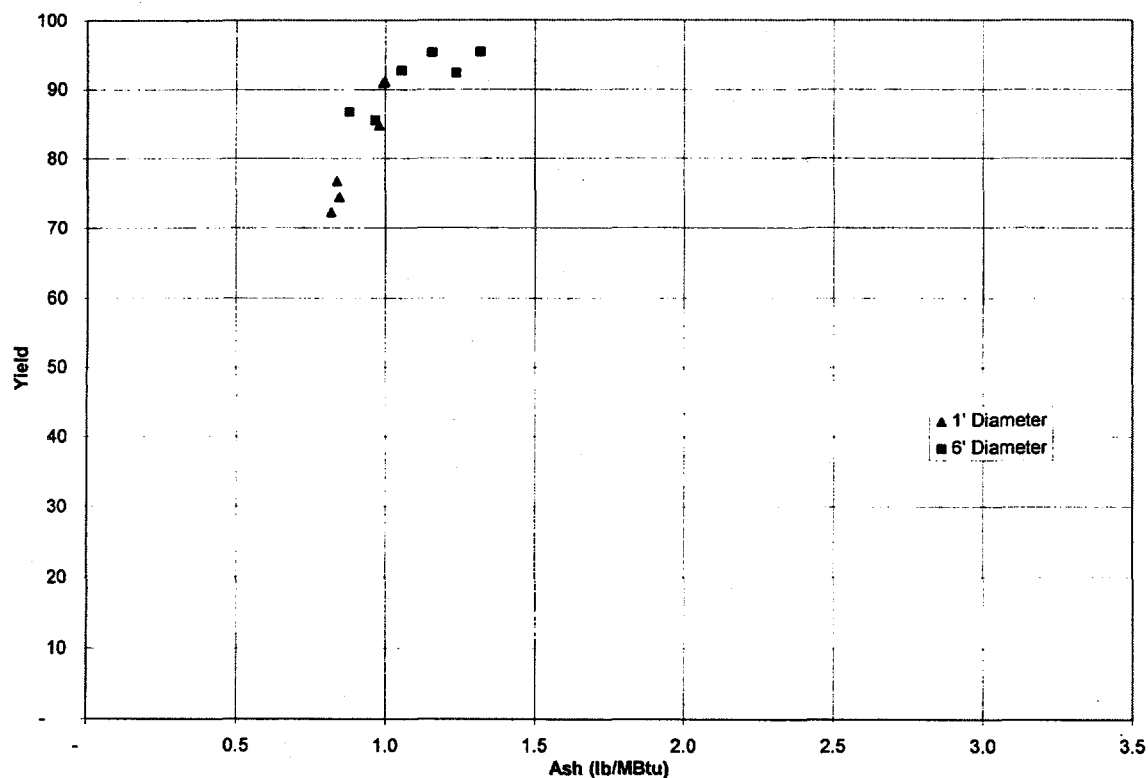


Figure 19. Comparison of 1-Foot and 6-Foot Microcel™ Columns - Yield vs. Ash - Taggart Coal

Table 37. Comparison of 12-inch & 6-foot Microcel™ Performance - Indiana VII Coal

Parameter	6-Foot Unit (Optimization)	12-Inch Unit
Feed Rate (lb/hr)	3,200	85.4
Feed Ash (%)	10.18	10.08
Feed Solids Concentration (%)	7.55	6.07
Collector Rate (lb/ton)	4.97	5.18
Frother Rate (lb/ton)	1.26	1.30
Cross Sectional Area (ft ²)	28.27	0.79
Air Rate (CFM)	55	1.53
Air Superficial Velocity (ft/min)	1.95	1.95
Wash Water Rate (GPM)	142	3.95
Wash Water Velocity (ft/min)	0.67	0.67
Tails Ash (%)	23.45	26.89
Tails Solids Concentration (%)	0.78	1.18
Carrying Capacity (lb/hr/ft ²)	74	86
Clean Ash (%)	3.27	2.98
Clean Ash (lb/MBtu)	2.36	2.15
Clean Solids Concentration (%)	12.45	12.30
Yield (%)	65.8	70.3
Energy Recovery (%)	71.3	76.4

Scale-up of Microcel™ Flotation Column - Indiana VII Coal

The 12-inch Microcel™ flotation column was operated at conditions similar to those used during some parametric testing and optimization runs. Though both Microcel™ units produced results that fall on the same grade-yield curve, the data indicates that the 6-foot unit produced clean coal at a higher yield, energy recovery, and ash content than the 12-inch unit. A comparison of the conditions and results for both units is shown in Table 37, while graphical analysis is presented in Figure 20.

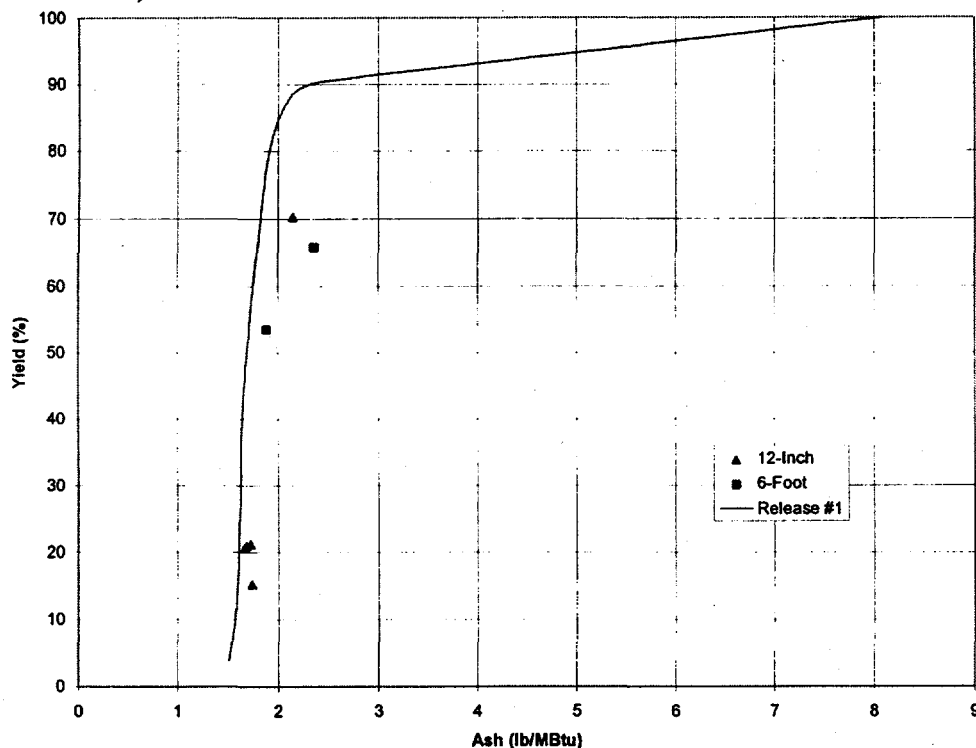


Figure 20. Comparison of 1-Foot and 6-Foot Microcel™ Columns - Yield vs. Ash - Indiana VII Coal

Scale-up of Microcel™ Flotation Column - Hiawatha Coal

The 12-inch Microcel™ flotation column was operated at conditions similar to those used during the extended PDU production run. Though both Microcel™ units produced results that fall on the same grade-yield curve, the data indicated that the 6-foot unit produced clean coal at a higher yield, energy recovery, and ash content than the 12-inch unit. A comparison of the conditions and results for both units is shown in Table 38, while graphical analysis is presented in Figure 21.

Table 38. Comparison of 12-inch & 6-foot Microcel™ Performance - Hiawatha Coal

<u>Parameter</u>	<u>6-Foot Unit (Extended Run)</u>	<u>12-Inch Unit</u>
Feed Rate (lb/hr)	4,309	139
Feed Ash (%)	8.68	8.77
Feed Solids Concentration (%)	5.27	6.13
Collector Rate (lb/ton)	0.46	0.48
Frother Rate (lb/ton)	0.30	0.26
Cross Sectional Area (ft ²)	28.27	0.79
Air Rate (CFM)	51	1.42
Air Superficial Velocity (ft/min)	1.80	1.81
Wash Water Rate (GPM)	140	3.75
Wash Water Velocity (ft/min)	0.66	0.64
Tails Ash (%)	36.27	21.09
Tails Solids Concentration (%)	0.43	1.16
Carrying Capacity (lb/hr/ft ²)	125	116
Clean Ash (%)	2.68	2.32
Clean Ash (lb/MBtu)	1.89	1.62
Clean Solids Concentration (%)	23.32	23.23
Retention Time (min)	12.44	9.03
Bias Factor	0.81	0.56
In-Line Mixer Velocity (ft/sec)	8.82	6.50
Yield (%)	81.8	65.6
Energy Recovery (%)	88.0	67.3

It is thought that the difference in performance may be attributed to varying recirculating velocities and flotation cell geometry as discussed below:

- Bubbles generated in the 12-inch column (2 mm) were considerably larger than those in the 6-foot column (1 mm). Large bubbles typically result in a lower carrying capacity (low yield) and more selectivity (higher quality) than smaller bubbles. Investigations into this difference revealed that the recirculation velocity through the 12-inch unit's in-line mixer was lower than that of the 6-foot unit. Specifically, the 12-inch unit operated with a recirculation velocity of 6.5 ft/sec while the 6-foot unit generally operated with a recirculation velocity of 8.8 ft/sec. Because bubble size is inversely proportional to recirculation velocity, the lower velocity in the 12-inch column most likely resulted in larger bubbles and varying performance.
- The retention time of the 12-inch column is only 73% of the 6-foot column (9.0 minutes versus 12.4 minutes). This low retention time may have resulted in the rejection of desired middlings to the tailings stream. Had the retention time been longer (geometry of 12-inch column similar to 6-foot column) the middlings

might have reported to the clean coal stream increasing the yield and product ash.

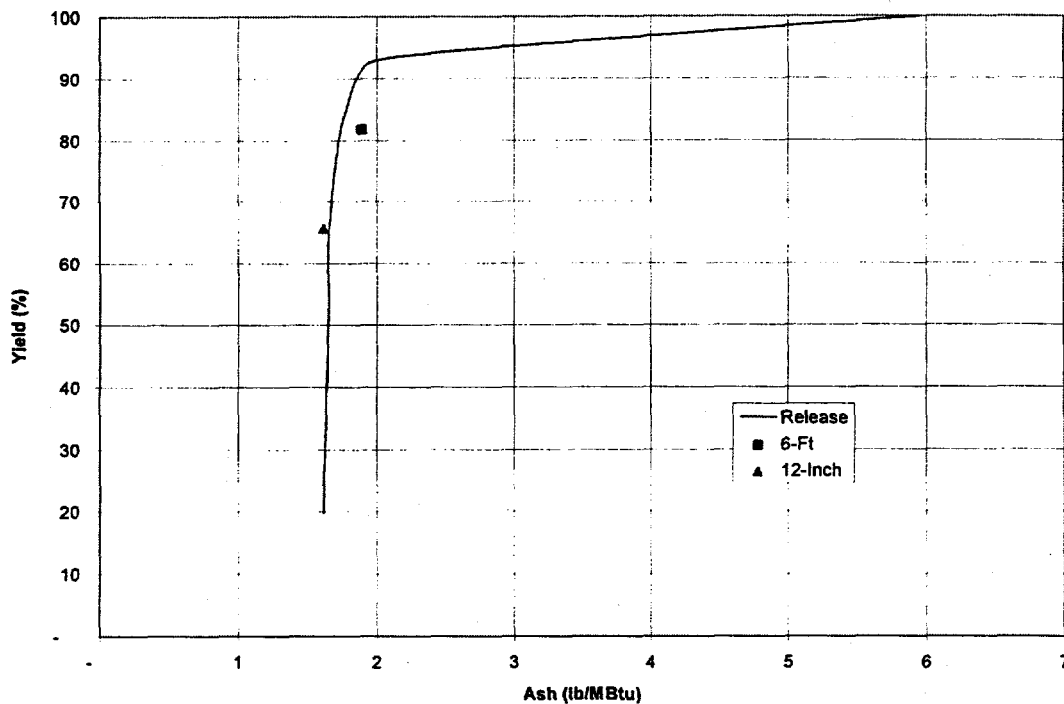


Figure 21. Comparison of 1-Foot and 6-Foot Microcel™ Columns - Yield vs. Ash - Yield vs. Ash Hiawatha Coal

LESSONS LEARNED

Based on the testwork and operation of the PDU Flotation Module, the following general lessons were learned:

- Feed coal should be stored in a silo for protection from the elements. Coal left uncovered may result in material handling problems due to freezing or sticking at transfer points. These problems were particularly noticeable with the Indiana VII coal.
- Sumps should be designed with enough capacity that small changes in volume do not product large fluctuations in level readings. Specifically, sumps and tanks should favor additional width with less height. PDU sumps showed widely fluctuating levels due to small width dimensions.
- Sumps should also have enough capacity to absorb process upsets without causing downtime.

- Proposed ball mill charges should be reviewed for proper loading and ball size. PDU ball mills were improperly charged (initially) which resulted in inefficient grinding and premature ball wear.
- Ball mill circuits should be designed for feed streams of 40% - 50% solids (by weight). PDU circuits initially operated at about 25% solids which compounded the problems of inefficient grinding and premature ball wear.
- Ball mills should only treat the size fraction that need grinding. Grinding the entire feed stream, as in the PDU, increases mill size requirements as well as operating costs. Size classification equipment, such as screens or cyclones, should be used as needed.
- Ball mill discharge magnets should be included in future designs for removal of grinding media degradation. Fine iron from the ball mill grinding media clogged pumps and cyclone apexes in the PDU.
- Baffles should be used in all agitated tanks. Many PDU tanks were initially designed without baffles. This resulted in vortexing, pump cavitation, and inaccurate sump level readings. The problem was corrected with the addition of baffles to the tanks.
- Nuclear density gauges should monitor only pipes that are free of air bubbles. The PDU density gauge provided radical and inaccurate fluctuations in density due to entrained air bubbles. The solution to this problem is the simple deaeration of slurry before entering the nuclear gauge. This can be accomplished with a deaeration tank upstream of the feed pump.
- Microcel™ flotation column wash water lines should be plumbed with the option of using fresh water. Though this option may not be used with high frequency, it allows the plant to operate at times when clarified water may cause contamination of the clean coal product.
- The Microcel™ flotation column should be designed with a fill water port (6 - 8 inches in diameter) for fast filling resulting in minimal downtime.
- Clean coal product sumps should be located immediately adjacent to the Microcel™ flotation column with large vertical pipe feeds. Sumps initially used in the PDU were located over 30 feet from the Microcel™ unit and were considerably undersized. The problems caused by this oversight included pipe plugging and unwanted downtime.
- Microcel™ column interface levels should be monitored with a ball float. This monitoring method proved to be the most reliable in such a harsh environment. Continuous data acquisition should be considered with such a unit.
- A variable speed pump proved to be invaluable to the proper operation of the Microcel™ column. The ability to adjust pump speed provides tremendous flexibility in varying bubble size and grade / yield.
- Dewatering equipment should be designed specifically for the intended use. The filters utilized in the project were used in a prior DOE effort, and as such,

were not a perfect match for the PDU application. The result was low filtering capacity and unscheduled downtime.

CONCLUSIONS AND RECOMMENDATIONS

The work completed in this study has provided considerable insight to the scale-up, design, operation, and performance of flotation columns (and related unit operations) as well as the need for further research in this area. A summary of relevant conclusions and recommendations follows.

CONCLUSIONS

Success of Program

The work and results related to this project should be considered entirely successful. The 2 tph flotation module was operated from January, 1996 through September, 1996 processing over one thousand tons of the Taggart, Indiana VII, and Hiawatha coals. Parametric testing was performed on each test coal followed by optimization testwork and a round-the-clock production run. A substantial amount of each coal's clean product was transported to Penn State University for combustion testing. Overall, the Taggart coal was cleaned to 1 lb ash/MBtu while the Indiana VII and Hiawatha coals were cleaned to 2 lb ash/MBtu. Not only were the project goals achieved, the process equipment performed extremely well in terms of reliability, control, and repeatability of results. A commercial plant cost study performed by Bechtel, estimated the cost of production for premium quality coal water slurry fuel to be \$2.15/MBtu which met the overall project goal.

Operation and Performance of Microcel™ Flotation Column

The operation and performance of the Microcel™ flotation column was very successful. Not only was the unit simple for the technicians to operate and maintain, it was easily capable of producing premium quality fuel. Overall, the unit could reach steady state within 20 minutes and maintain production levels with little variance. The bubble generation system proved to be extremely reliable with no unplanned downtime. The wash water system also performed reliably with only a small amount of maintenance needed to clean the discharge orifices. Extended production runs indicated that the Microcel™ flotation column is a dependable and cost effective means of cleaning coal to high quality levels.

Important Process Variables

Testing of the three coals in the PDU flotation module indicated that several process variables were important to proper operation. The most important variables and their effect on each coal's performance in the Microcel™, are discussed below:

Frother Dosage - This variable was found to have the most significant impact on yield for all three coals. It was also found to be very closely linked to product ash for the Taggart coal.

Air Rate - This variable was found to have the greatest impact on product ash for the Indiana VII coal. Specifically, increases in air rate increased the product ash.

Wash Water - The ability of this parameter to remove entrained mineral matter from clean coal froth resulted in a very large impact on product ash for the Hiawatha coal.

Recirculation Rate - The Hiawatha coal product ash was also greatly influenced by the recirculation rate. Variations in this parameter resulted in highly selective bubble size fluctuations.

Test Coal Grind Size

Testing performed in this program has indicated the size distribution needed for adequate mineral liberation in each coal. Taggart and Hiawatha coals have very similar liberation requirements while those for the Indiana VII coal are much more aggressive. Each test coal and corresponding d_{80} value is provided in Table 39.

Table 39. Effect of Grind Size for Test Coals

Coal	d80 Value	Ash - lb/MBtu	Sulfur - lb/MBtu	Yield %	Energy Recovery %
Taggart	51	0.99	0.46	94.4	96.9
Indiana VII	22	2.07	0.42	65.8	71.9
Hiawatha	51	1.89	0.44	81.8	88.0

It should be noted that the theoretical yield (67%) and energy recovery (73%) for Indiana VII coal could not be achieved due to excess frother in the closed water loop. Additional testing may allow these targets to be achieved.

Scale-up Criteria

Testing has determined that many criteria affect the scale-up similitude between the 1-foot and 6-foot columns. For all test coals, similar operating conditions in each column produced varying results. The 1-foot column consistently produced clean coal products with lower ash and yield values than the 6-foot unit. The difference in operation was attributed to differences in recirculation line velocity (which affects bubble size) and retention time. The 1-foot column produced bubbles that were visibly larger and more selective than the 6-foot unit. In addition, the retention time of the 1-foot column, though coal dependent, was significantly smaller than that of the 6-foot column.

It was found, however, that the clean coal carrying capacity values of the 1-foot and 6-foot units were similar. These values are shown in Table 40.

Table 40. Microcel™ Scale-up Criteria - Clean Coal Carrying Capacity (lb/hr/ft²) for 1-Foot and 6-Foot Units

Microcel™ Size	Taggart Coal	Indiana VII Coal	Hiawatha Coal
1-Foot	129	74	116
6-Foot	127	86	125

Ash Property Changes

It was found that the PDU Microcel™ flotation consistently increased the base/acid ratio of the ash and decreased the silica/alumina ratio. The overall results were substantial declines in the reducing atmosphere fusion temperatures of the ash in the Taggart and Indiana VII coals, and a smaller decline in the fusion temperatures of the ash in the Hiawatha coal. Except for titania, and iron oxide in the case of the Taggart coal, the concentrations of the ash constituents were significantly reduced on a heating value (lb/MBtu) basis, by advanced flotation cleaning in the PDU.

Toxic Trace Element Removal

There were substantial reductions, over 25% on a heating value basis, in the residual concentrations of arsenic, lead, and chlorine for all three as-received test coals. The reduction in the concentrations of beryllium, cadmium, chromium, cobalt, manganese, mercury, nickel, and selenium varied from coal to coal. Flotation did not appear to reduce the heating value basis concentration of antimony in any of the coals.

Reduction in trace-element concentrations from the amounts in the parent Taggart and Indiana VII ROM coals was greater on a heating value basis than the reduction from the amounts in the as-received test coals. The residual concentrations of arsenic, beryllium, cadmium, chromium, cobalt, lead, manganese, mercury, and selenium in the two clean coals were especially lower than their concentrations in the two ROM coals. Mixed results were seen for nickel and chlorine, and the concentration of antimony did not appear to have been reduced at all by the combination of preparation plant cleaning and column flotation. Except for chlorine, the rejection of the toxic trace elements from the Taggart and Indiana VII ROM coals was greater than their rejection from the Hiawatha test coal even though the Hiawatha coal had not been washed prior to flotation in the PDU.

RECOMMENDATIONS

Recommendations for Design of Commercial Plant

The design of any commercial column flotation application should be based on sound scale-up data. Designers should use as large a flotation column as possible with the ability to vary the retention time through column height. A six-foot unit, if placed in operation at PETC, would prove very useful for such an endeavor. However, a three foot unit should be considered secondary with a 1-foot column as a final choice. In addition, it is imperative that the test column be equipped with a variable speed pump for bubble size control. The inability of the 12-inch and 6-foot columns to produce similar results was attributed to variances in bubble size and retention time.

Designers should also give consideration to maintenance requirements of the flotation column - specifically the bubble generation system. The simple design of the Microcel™ column provides almost worry-free operation with little maintenance. As a result, Microcel™ flotation columns are currently recommended for commercial applications.

In addition, design engineers should be mindful of the process control scheme developed for the flotation column. Because many different parameters affect the performance of the column (frother dosage, collector dosage, air rate, wash water rate, recirculation rate), careful control of these parameters is necessary for consistent product yield and quality. As a result, instrumentation and control equipment are vital and highly recommended.

The operation and performance of the Microcel™ flotation column is very predictable when feedstock characteristics are known and operating conditions are continually monitored and controlled by a computerized control and data acquisition system (CDAS). Though the resources that are needed to generate this performance data may be tedious and somewhat expensive, the resulting data is invaluable. Not only can the information be used to predict column performance, if used by a trained professional, it can be used to optimize performance for maximum economic benefit and return on investment (ROI). This optimization step was proven during this testwork and is recommended for future commercial and near-term applications.

Recommendations for Future R&D Work

Each year, hundreds of thousands of recoverable tons of fine coal are lost to refuse disposal. This may be the result of poor flotation cell / column performance in an existing preparation plant or even the lack of an economical fine coal cleaning process itself. If methods for optimizing existing flotation columns could be developed, considerable economic benefits would be realized by United States coal producers. Optimization of column flotation has practical applications with real-world benefits. The ability to optimize the performance of a commercially installed flotation column would result in tremendous economic benefits to domestic mining companies. As a result of

this study, it is recommended that future research in the areas of near-term flotation column performance optimization be explored.

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