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

PROJECT FINAL REPORT

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ABSTRACT

The primary goal of this project was the engineering development of two advanced physical fine coal cleaning processes, column flotation and selective agglomeration, for premium fuel applications. The 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/h process development unit (PDU). Secondary goals were to determine the removal of toxic trace elements during fine coal cleaning and to apply the technology in the near-term to recover fine coal in existing preparation plants. The project began in October 1992 and was completed in September 1997. This is the final report for the project, and it summarizes all of the project accomplishments.

The ash in six common bituminous coals, Taggart, Winifrede, Elkhorn No. 3, Indiana VII, Sunnyside and Hiawatha, could be liberated by fine grinding to allow preparation of clean coal meeting premium fuel specifications (<1-2 lb/MBtu ash and <0.6 lb/MBtu sulfur) by laboratory and bench-scale column flotation or selective agglomeration. Over 2,100 tons of coal were cleaned in the PDU at feed rates between 2,500 and 6,000 lb/h by Microcel™ column flotation and by selective agglomeration using recycled heptane as the bridging liquid. Parametric testing of each process and 72-hr productions runs were completed on each of the three test coals. The following results were achieved after optimization of the operating parameters:

	Flotation			Agglomeration		
	Grind D80, µm	Ash lb/MBtu	Btu Recov, %	Grind D80, µm	Ash lb/MBtu	Btu Recov, %
Taggart	60	1.0	96.9	30	1.1	99.2
Indiana VII	23	2.3	82.0	21	1.9	99.9+
Hiawatha	48	1.9	88.0	42	1.9	98.9

Both cleaning processes were robust and reliable, offering few operating problems during the PDU operations. The steam stripping operation was particularly noteworthy since it recovered about 99 percent of the heptane for reuse. A conceptual plant design and cost estimation study indicated that premium fuel, in the form of coal water slurry, could be produced commercially at a cost of \$2.15/MBtu by flotation and \$2.42/MBtu by selective agglomeration, both well under the goal of less than \$2.50/MBtu.

Depending upon the source coal, advanced physical fine coal cleaning reduced the concentrations of toxic trace elements such as mercury and could be part of a strategy to control hazardous air pollutants.

As a result of extensive in-plant testing for this project, Cyprus Amax installed three 4-meter Microcel™ flotation columns in the Lady Dunn Preparation Plant to recover fine coal that had been lost by the previous mechanical cell flotation plant.

ACKNOWLEDGMENTS

Completion of this project was truly a team effort and the contributions of each member of the team is gratefully acknowledged. Amax Research and Development Center. a subsidiary of Cyprus Amax Minerals Company, was the Prime Contractor for the project. Dr. Mahesh Jha was Project Manager for Amax and Mr. T. J. Feeley, III was Project Manager for the Department of Energy. The team members and the individuals who made important contributions to the project are listed below:

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EXECUTIVE SUMMARY

The U. S. Department of Energy (DOE) awarded cost-sharing contract No. DE-AC22-92PC92208 on September 30, 1992 to an Amax-led team for "Engineering Development of Advanced Physical Fine Coal Cleaning for Premium Fuel Applications." The coal cleaning methods targeted by the program were the advanced column froth flotation and selective agglomeration processes researched and developed under the DOE Acid Rain Control Initiative (ARCI). The program stressed development of processes for preparation of ultra-clean coal water slurry fuel that would be a cost-effective replacement for a portion of the oil and gas fired in utility and industrial boilers.

The replacement of oil and gas can only be realized if retrofit costs are kept to a minimum and retrofit boiler emissions meet national goals for clean air. These concerns established the specifications for the maximum allowable ash and sulfur levels in the fuel and the handling and combustion properties of the coal water slurry fuel (CWF).

PROJECT GOALS AND OBJECTIVES

The ultimate goal of the Department of Energy Coal R&D program is to develop a coal-based fuel that will be a viable alternative for fuel oil or natural gas in future years. The current project had three major objectives to move toward this goal:

- The primary objective was to develop the design base for commercial fine coal cleaning facilities for producing ultra-clean coals which can be converted into coal-water slurry premium fuel. The coal cleaning technologies to be developed were advanced column flotation and selective agglomeration, and the goal was to produce fuel meeting the following specifications --
    - Less than 2 pounds of ash per million Btu (860 grams per gigajoule) and preferably less than 1 pound of ash per million Btu (420 grams per gigajoule).
    - Less than 0.6 pound of sulfur per million Btu (258 grams per gigajoule).
    - Recovery of at least 80 percent of the heating value in the run-of-mine raw coal.
    - Production cost of the CWF to be less than $2.50 per million Btu ($2.37 per gigajoule) including the cost of the coal.
- A second objective was to develop near-term applications of the advanced coal cleaning technologies in new or existing coal preparation plants in order to efficiently process minus 28-mesh fines and convert them into marketable products.
- A third objective was to determine the extent of the removal of toxic trace elements from coal by the advanced column flotation and selective agglomeration technologies.

This report summarizes the work that was done to accomplish the above objectives and the important findings of the study. Topical reports were issued upon completion of important tasks and subtasks, and these contain detailed results of the specific areas investigated during the 5-year project. Chapter 13 contains a list of these reports and also a list of public presentations describing particular aspects of the work.

PROJECT APPROACH

The project included laboratory- and bench-scale advanced flotation and selective agglomeration process research and development studies and specific tasks to study the grinding of coals to liberate ash minerals and to develop CWF formulations for the clean coals. Near-term applications were investigated during a separate task. The final tasks were for the design, and construction of an integrated 2-st/h process development unit (PDU) containing advanced flotation and selective agglomeration modules for cleaning test coals. Operation of the PDU provided performance data from parametric tests on three coals followed by a 72-hour production run on each one. Scale-up parameters for the conceptual design of commercial plants for production of premium fuel from coal were also derived from the test program and used for an economic feasibility analysis of each cleaning process.

The project team was headed by a subsidiary of Cyprus Amax Minerals Company, Amax Research & Development Center (Amax R&D), with assistance from Cyprus Amax Coal Company (Midwest and Cannelton Divisions). Entech Global managed the project for Amax R&D and provided research and development services in all project areas at the Amax R&D Center in Golden, Colorado. Bechtel Corporation performed the engineering analysis of potential near-term applications and designed and assisted with the construction of the 2-st/h PDU operated by Entech Global at Amax R&D. Bechtel also studied the economics of commercial premium CWF production.

The Center for Applied Energy Research (CAER) of the University of Kentucky, and Center for Coal and Mineral Processing (CCMP) of the Virginia Polytechnic Institute and State University provided technical assistance with the advanced flotation technologies and Arcanum Corporation provided similar assistance with the selective agglomeration technology. Dr. John P. Dooher of Adelphi University and Dr. Douglas V. Keller, Jr. of Syracuse University were consultants to the project in the areas of CWF formulation and selective agglomeration, respectively. Work on the project began on September 30, 1992 and was completed on September 30, 1997.

ADVANCED FINE COAL CLEANING TECHNOLOGIES

Efficient process flowsheets have evolved for economically preparing the quality of coal most demanded by the existing market place (5 to 15 percent ash). Only rarely can these flowsheets be adjusted to produce clean coal meeting the target premium fuel ash specifications, though, and then only with the loss of a significant amount of the coal.

The logical way to prevent the loss of coal when trying to meet low ash specifications would be to crush and grind the coal before cleaning in order to liberate the mineral matter from the coal. Unfortunately, the technology for cleaning finely ground coal has not been developed to the extent needed for efficient and economical production of low-ash fuel. Coal cleaning technology needed to be advanced in this area.

To fill this need, a number of processes were investigated during the 1972 to 1992 period by the U. S. Department of Energy and by others for physically separating impurities from fine coal. Much of this interest centered on desulfurization, but this work clearly showed that the residual amount of ash in the coal was also reduced to significantly lower levels than the levels achieved by conventional cleaning at coarse mesh sizes. Two of these processes in particular – advanced column flotation and selective agglomeration – were especially attractive and warranted further development as methods for preparing low-ash premium fuel from coal. Each of these two processes had in fact been the focus of successful proof-of-concept development projects* co-sponsored by the Department of Energy under the Acid Rain Control Initiative.

Advanced Column Flotation

Advanced column flotation is an improvement of the froth flotation operation often employed in coal preparation plant for cleaning fine coal slurries. It utilizes a counter-current flow of feed slurry and air bubbles to separate coal particles with hydrophobic (water-repellent) surfaces from mineral refuse particles with hydrophilic (water-wetted) surfaces. The separation is done in a tall column rather than in a series of mechanically agitated cells. Wash water addition to the column above the feed point is a key feature of column flotation when cleaning fine coal. When added in sufficient volume, this wash water creates a net downward flow of water which flushes fine refuse from the cleaning zone between the point where the feed slurry enters the column and where the froth overflows the column. Several types of column flotation have been developed which differ in the way the bubbles are formed. Several of these were studied during the program.

Selective Agglomeration

Selective agglomeration cleaning is also based on the difference between the surface properties of coal and fine refuse particles. Here an oily bridging liquid is mixed with the slurry so that the fine hydrophobic coal particles become coated with the oil. If subjected to intense high shear-rate mixing, the oiled particles adhere to each other in

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***** Southern Company Services, "Engineering Development of Selective Agglomeration," Contract No. DE-AC22-89PC88879, Final Report, April 1993.

ICF Kaiser Engineers, "Engineering Development of Advanced Physical Fine Coal Cleaning Technologies - Froth Flotation," Contract No. DE-AC22-88PC88881, Final Report, March 1995.

clusters or “microagglomerates.” The refuse particles, on the other hand, are not affected by the bridging liquid and remain dispersed in the liquid phase. Continued mixing under less intense low shear-rate conditions allow the microagglomerates to grow in size until they may be separated from the dispersed refuse particles by screening or flotation.

Two types of bridging liquids were considered for this project:

1. Non-volatile oils such as diesel fuel, fuel oil and kerosene which remain with the product coal after the separation.
2. Volatile oils such as pentane, hexane and heptane which can be recovered from the product coal and reused.

This project focused on the use of reusable bridging liquids because of their good performance and potential for lowering operating costs. On the other hand, the use of non-volatile bridging liquids was emphasized for near term applications since the technology for recovering and reusing bridging liquids had not been developed beyond the laboratory stage when the project began.

COAL SELECTION, ACQUISITION AND PROPERTIES

Successful achievement of the ultimate goal of this program will depend upon a plentiful supply of suitable coals for upgrading into premium fuel. Considerable effort was devoted to the selection of six coals for the laboratory and bench-scale testing and, later on, three coals for the PDU testing that would be viable sources for preparation of premium fuel.

The test coals were selected based upon their availability and amenability to the advanced cleaning. At least 32 coals from the states of Pennsylvania, Maryland, West Virginia, Virginia, Ohio, Indiana, Illinois, Kentucky, Alabama, Montana, Wyoming, Colorado, and Utah were given serious consideration. Laboratory amenability tests were made on the most likely candidates before making the final choices.

Five bituminous coals and one subbituminous coal were chosen for detailed laboratory and bench-scale testing. With the possible exception of the subbituminous coal selection, each one was a good prospect for production of premium fuel during the PDU operation since they responded well to the initial amenability testing. The selected coals were as follows:

- Taggart Seam, Wentz Mine, Virginia (2.0% ash, 0.6% sulfur, 3.0% moisture) washed for steam coal and specialty markets, hvA bituminous rank.
- Sunnyside Seam, Sunnyside Mine, Utah (4.8% ash, 0.6% sulfur, 6.5% moisture) washed for metallurgical markets, also hvA bituminous rank.
- Elkhorn No. 3 Seam, Chapperal Mine, Kentucky (5.6% ash, 0.8% sulfur, 7.0% moisture) washed hvA steam coal selected from the very large production from eastern Kentucky.

- Winifrede Seam, Sandlick Mine, West Virginia (7.9% ash, 0.9% sulfur, 6.4% moisture) washed hvA steam coal selected from the large production in West Virginia. It required very fine grinding for liberation of the ash minerals.
- Indiana VII Seam, Minnehaha Mine, Indiana (7.5% ash, 0.4% sulfur, 18.9% moisture) washed hvC steam coal containing less sulfur than most Midwestern coals. It responded better to the amenability testing than other coals from the region, but it also required fine grinding for liberation of the ash minerals.
- Dietz Seam, Spring Creek Mine, Montana (3.9% ash, 0.3% sulfur, 21.5% moisture) subbituminous B coal mined for direct sale to utility customers. It responded better to the laboratory amenability tests than the other low-rank coals that were evaluated.

The selected test coals exhibited a diverse range of attributes. The coals were from seams with at least 500 million tons of reserves, and each coal was from an active mine working the seam. Substantial production was represented from six states in the eastern, central and western parts of the United States. The bituminous coals ranged in rank from high volatile C to high volatile A rank and had ash-free inherent moisture contents between 1.75 and 15.6 percent. The subbituminous selection was representative of the very important Powder River Basin area. Some (such as the Taggart coal) were expected to grade up to premium fuel quality without much difficulty, while others (such as the Winifrede, Indiana VII and Dietz) were expected to challenge the advanced cleaning technologies.

At the conclusion of the bench-scale testing three coals were chosen for use during the PDU process development. The three were Taggart from the Steer Branch Mine in Virginia, Indiana VII from the Kindill No. 3 Mine in Indiana and Hiawatha from the Crandall Creek Mine in Utah. The Hiawatha coal substituted for the coal from the Sunnyside Mine which had closed.

Approximately 2400 tons of coal was purchased from the regular production of the three mines for the PDU testing. Most of it arrived by rail. The coal was trucked to Ralston Development Company as needed for crushing to minus 1/2 inch and transfer to Amax R&D for the PDU operation.

GRINDING/LIBERATION OF TEST COALS

Prior experience has shown that liberation of attached mineral grains from the individual coal particles is a key consideration when cleaning coal by froth flotation and selective agglomeration which depend upon a difference between the surface properties of pure coal and pure mineral matter. A nearly complete difference in surface properties is important because composite mineral particles with relatively small surface exposures of carbonaceous material will adhere to air bubbles and to oil droplets and report with the clean coal product. In many cases, grinding to minus 45 µm (325 mesh) and finer is required in order to achieve the needed liberation.

Because of the importance and the high cost of grinding, considerable attention was given to the subject during the project. In particular, various grinding circuit configurations were evaluated during the Phase I bench-scale studies, and the liberation characteristics for each test coal was quantified so that the required grind size could be identified that would provide clean coal meeting the target premium fuel ash specification during operation of the 2-st/h PDU. Later on, additional grinding equipment testing was done at vendor locations before final selection of the PDU grinding mills.

Bench Scale Grinding Studies

Wet grinding systems were investigated for preparation of feed slurry for the advanced cleaning using available equipment at Amax R&D. Two-stage circuits were considered most appropriate, and the key pieces were a 4-ft x 4-ft dia Marcy overflow ball mill for the primary grinding and a 40-liter Draiswerke stirred ball mill for the finish fine grinding. A 48-inch Sweco screen and a rented 7-inch diameter solid-bowl Bird classifying centrifuge were used for classification. Closed-circuit configurations, where either the screen or the centrifuge oversize was returned to the fine grinding mill, was found to be the most efficient circuit configuration. The inclusion of the centrifuge in the circuit also allowed evaluation of selective grinding technology where the pyrite could be rejected from the recycle material by an additional gravity-separation device.

Concurrent liberation studies showed that the Taggart, Elkhorn No. 3 and Sunnyside coals did not require exceptionally fine grinding before cleaning. In fact, the optimum PSD appeared to be in the range where ordinary ball milling would suffice. Consequently, additional fine grinding tests were performed using the 4-foot ball mill in place of the stirred ball mill.

The initial assessment of mineral-matter liberation achieved by grinding the test coals was obtained by washability (heavy-liquid) tests on the crushed coals and on the coal in the slurries from the open-circuit grinding tests. Later, selective agglomeration and tree-flotation release analysis tests were also performed on the slurries. Selective agglomeration tests with heptane bridging liquid proved to be the most convenient and most meaningful method for evaluating liberation. The fineness of the grind in terms of the D80 value (size at which 80 percent of the particles would pass) for the test coals were as follows:

	Residual Amount, lb/MBtu		Grind Size
	Ash	Sulfur	D80, µm
Taggart	< 1.0	< 0.4	45
Sunnyside	< 2.0	< 0.5	45
Elkhorn No. 3	< 2.0	< 0.6	45
Indiana VII	< 2.0	< 0.4	20
Winifrede	< 2.0	< 0.6	11
Dietz	< 2.0	< 0.3	20

Based on the results of the bench-scale testing, a two-stage grinding (open-circuit primary grinding and closed-circuit fine grinding) was recommended for the PDU. A stirred ballmill was preferred for the second stage and was required for coals such as Winifrede which needed very fine grinding for liberation. Single-stage closed-circuit ball milling was an option for coals such Taggart, Sunnyside and Elkhorn No. 3.

The centrifuge was an effective classifier for making fine particle size splits but would not be needed in the PDU unless coals such as Winifrede and possibly Indiana VII were being cleaned. There was little incentive to apply the selective grinding technology to any of the test coals because of their low pyrite content.

Grinding circuit energy consumptions were projected from the bench-scale results for use when planning the design and operation of the PDU. The projections were as follows:

	HGI	Target Grind D80, µm	Primary Grinding kWh/st	Fine Grinding kWh/st	Total Energy kWh/st
Taggart	52	45	87		87
Sunnyside	54	45	83		83
Elkhorn No. 3	46	45	107		107
Indiana VII	55	20	27	110	137
Winifrede	47	11	29	280	309
Dietz	41	20	82	103	185

PDU Grinding Circuit

Additional grinding tests were conducted at vendor facilities before final selection of grinding equipment for the PDU. The first of these tests was at Svedala Industries, York PA and subsequent tests were at Union Process, Akron OH and Netzsch, Exton PA. All of these tests were with stirred ball mills for the fine grinding stage. A Netzsch mill was selected for the PDU based upon its efficient grinding, compact size and lower overall installation cost.

At 88 and 76 kWh/st for the Taggart and Hiawatha coals, respectively, grinding performances in the PDU matched the bench-scale projections for these coals but differed for the Indiana VII coal which required only 26 kWh/st in the PDU. Part of the latter difference was because the Indiana VII coal was only ground to D80=67 µm during the PDU production run instead of to D80=20 µm as during the bench-scale work. More importantly though, it appears that the stirred ball mill grinding may not have been operated very efficiently in either the bench-scale circuits or in the PDU circuit.

COLUMN FLOTATION STUDIES

Laboratory, bench-scale and PDU tests were conducted during the program applying column flotation technology to the production of clean coal meeting premium fuel specifications.

Laboratory Process Optimization

The specific objective of the laboratory process optimization studies was to determine the preferred column configurations and operating conditions for cleaning the six test coals.

A number of column flotation systems have been developed for coal flotation. The design and geometry of most of these systems are similar except for the manner of introducing and dispersing air in the slurry (the bubble generating system). Of the better known systems, the Ken-Flote™, GL&V Ontario packed column and the Microcel™ systems have consistently shown superior performance. Accordingly, the performance of these three systems were compared during the laboratory optimization of column flotation, and the laboratory work was divided among the team members to accomplish project objectives.

The laboratory units were 2-, 3- or 4-inch diameter columns between 8 and 26 feet tall. They were set up for continuous feed and continuous discharge of froth and refuse and, depending upon their diameter, could handle between 50 and 500 grams per minute of fine coal. The feed slurries were prepared in the bench-scale grinding circuit at Amax R&D. The main operating variables studied during the parametric testing were feed rate and percent solids, retention time, wash water rate, aeration rate, reagent type and dosage, and column height. The differences in response between several particle size distributions were evaluated for some of the coals.

The five ground bituminous test coals generally responded quite well to the column flotation. Residual ash and Btu recovery specifications were easily met while floating the Elkhorn No. 3, Taggart and Sunnyside coals. In fact, the Taggart coal could be cleaned to less than 1.0 lb/MBtu ash. It appeared that either the residual ash or the Btu recovery specification might need to be relaxed, though, if Indiana VII or Winifrede coals were to be processed into premium fuel by column flotation. The Winifrede coal also needed to be ground finer than the minus 325-mesh grind tested in the laboratory-scale columns.

Each of the aeration configurations separated ash from coal quite well. The packed column required more air, though, and may not have been quite as efficient for rejecting ash. The separations with the Ken-Flote™ Foam-Jet and Microcel™ systems were similar to each other, so there was little reason to choose one over the other at this stage of process development for production of ultra-clean coal. As such it was recommended that the follow-up bench-scale testing include both the Ken-Flote™ and the Microcel™ columns.

Retention time (feed rate) and wash water additions appeared to be the most important variables when operating the flotation columns. Finer particle size distributions required longer retention times and more wash water in proportion to the feed rate. Lower solids concentrations in the column, and in the feed to the column, may be desirable, but within the range tested column height was not a critical factor effecting performance. Collector requirements (diesel fuel in these tests) were similar to mechanical cell flotation (around 1 lb/st). Frother dosages may be a critical operating parameter, but it was not clear from the laboratory work what the proper type or amount should be.

Only laboratory Denver-cell tests were made on the Dietz subbituminous coal. Large quantities of reagents were required before any flotation occurred, and pH adjustment had little impact upon the behavior. However, a combination selective agglomeration and froth flotation showed signs of success but did not appear to be cost-effective. Since there did not appear to be any economically viable scheme for flotation of Dietz subbituminous coal, it was not tested in the column flotation system, and further development of subbituminous coal flotation was dropped from the program.

Bench-Scale Flotation

The primary objective of bench-scale flotation testing was to verify that the performance and operating characteristics observed during the laboratory column flotation can be scaled up to a 100 lb/h system when cleaning the specified test coals. A DOE-owned 12-inch diameter Ken-Flote™ column was procured for the bench-scale testing. On a cross-sectional area basis this column represented a 9- to 16-fold scale-up of the laboratory equipment in use at Amax R&D, CAER and CCMP. The Ken-Flote™ column was supplemented by a 12-inch Microcel™ column purchased from Minerals and Coal Technologies Inc. The two columns were installed in parallel to take advantage of the pumps and data logging facilities included with the Ken-Flote™ system.

Parametric tests were first conducted on the five bituminous test coals using the Ken-Flote™ system. Comparison tests and further rounds of process optimization were subsequently made in the Microcel™ column for the three coals which appeared to be best suited for the PDU operation. The effects of varying feed rate/retention time, wash water addition, aeration and reagent dosage on Btu recovery and ash rejection were quantified during the parametric testing and optimization. Samples from selected parametric tests were analyzed to learn the distribution of the toxic trace elements during advanced flotation.

The Ken-Flote™ and the Microcel™ columns differed in the manner in which the air was introduced into the slurry. The Ken-Flote™ column employed a Foam-Jet sparger fitted with four porous metal plugs which allowed a high-velocity flow of water to pass from an inner tube to the outside carrying with it tiny bubbles of the air which had been forced through the porous metal.

The Microcel™ column was furnished with a microbubble generating system consisting of a centrifugal pump and a Koflo in-line static mixer. The pump drew tailings slurry from the bottom of the column and returned the slurry 12 inches higher up in the column. Compressed air was injected into the pump flow just ahead of the in-line mixer where it was dispersed into microbubbles which flowed into the column along with the recycle tailings slurry.

Parametric Flotation Testing

The initial operation of the bench-scale flotation rig consisted of short parametric tests on each coal using the Ken-Flote™ column. The primary variables considered were feed rate in dry pounds per hour, percent solids in the feed slurry (which sets retention time), aeration rate in cubic feet per minute, and wash water additions in gallons per ton of dry feed coal.

Because of time constraints, the Microcel™ testing focused on the three coals selected for study in the PDU during Phase II. Several rounds of additional optimization testing were conducted on these coals in the Microcel™ unit in order to provide information for designing the PDU.

The flotation response noted for each test coal generally corresponded to the response noted during the laboratory testing. The amount of residual ash in the clean coal was generally found to correlate closer to the bias ratio than to any other operating variable. The bias ratio largely depended upon the amount of wash water added at the top of the column and the amount of water carried over with the froth. The amount of water added at the top should exceed the amount carried over with the froth by a comfortable margin (bias ratio >50 percent) to ensure a positive downward flow of wash water to the tailings.

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. This is consistent with the capacity/particle size relationship generally seen for column flotation. The particle size distribution of the feed slurry also had an impact upon the optimum amount of wash water needed for ash rejection. The coarsely ground slurries (Taggart, Elkhorn No. 3, and Sunnyside) did not need nearly as much wash water as did the finely ground slurries (Indiana VII and Winifrede). Part of the need for extra wash water when floating finely ground slurry can be attributed to the lower solids concentration in the froth when floating finer coal. The drainage of water from the froth was also influenced by the feed and aeration rates, by the reagent schedule and probably by the froth depth.

It appears from a comparison of optimized operating conditions that reagent requirements were less with the Microcel™ system than with the Ken-Flote™ system. Wash water requirements were less for one coal, greater for another coal and about the same for the third coal. The Microcel™ column appeared to have a greater capacity than the Ken-Flote™ column for flotation of Taggart and Indiana VII coals and somewhat less capacity for flotation of the Sunnyside coal. Microcel™ Btu recovery was significantly better for Sunnyside coal, but in other respects, and for the other two coals, the separation performances of the two systems were close to being the same. Scale-up of the Microcel™ column was considered more straight forward and reliable; therefore the Microcel™ column was selected for use in the PDU. The following projections were made for the design and operation of the PDU:

	Taggart	Elkhorn No. 3	Sunnyside	Indiana VII	Winifrede
Feed Coal:					
Ash, %	2.10	6.00	5.50	9.50	8.50
Grind, D80 µm	104	104	70	24	12
Clean Coal:					
Ash, %	1.37	2.75	2.74	2.82	2.91
Btu recovery, %	95.0	90.0	95.0	80.0	80.0
Froth Carrying Capacity, st/h per sq ft	0.16	0.16	0.11	0.04	0.02
Fine Refuse:					
Ash, %	14.0	27.2	37.2	28.5	25.2
Wash Water Requirement, gallon/short ton feed coal	1800	1800	2200	6500	18800
Superficial Wash Water Flow, ft/minute	0.64	0.64	0.54	0.58	0.84
Bias Flow Ratio, %	35	30	56	73	80
Aeration Rate, scfm/sq ft	1.9	1.9	1.9	1.7	1.7
Superficial Air Flow, ft/minute	1.9	1.9	1.9	1.7	1.7
Retention Time, minutes	11.5	11.0	13.6	13.6	13.4
Frother, lb/short ton	0.25	1.00	0.67	3.00	1.50
Diesel Fuel, lb/short ton	0.50	1.50	0.23	5.00	3.50

The five test coals were all relatively low in sulfur and each one had been washed at the mine to reject mineral matter. As a result, the flotation feed samples did not contain much pyritic sulfur, the highest of the five original head samples being the 0.17 percent pyrite sulfur (0.12 lb/MBtu) found in the Elkhorn No. 3 washed coal. Only minor reductions in sulfur contents were accomplished since most of the sulfur in these coals was in the organic form. Pyritic sulfur was rejected from the Sunnyside, Indiana VII, and Winifrede coals, but the amount rejected had little impact upon the final concentration of sulfur in the respective clean coals.

PDU Operations

The conceptual design of the PDU and Flotation Module was a collaborative effort between Bechtel, Entech, CCMP, and CAER and the detailed design of the PDU and Flotation Module was performed by Bechtel Corporation with support from Entech Global engineers. The plant was designed to fit inside an existing building at the Amax R&D Center in Golden, Colorado and utilize existing support services at that location.

Process and Plant Description

The advanced coal cleaning PDU and Flotation Module was divided into areas for design and operating purposes:

- Area 100 - Raw coal handling
- Area 100 - Grinding and classification
- Area 200 - Column flotation
- Area 400 - Clean coal dewatering

- Area 400 - Fine refuse (tailings) dewatering

Equipment for the PDU and Flotation Module was sized to allow production of 2 st/h of Sunnyside clean coal. It was expected that Taggart would be processed at the same rate but to produce 1 lb/MBtu ash coal. The capacity of the PDU would be less for Indiana VII coal. All areas of the PDU were operated from a central control room utilizing an integrated graphic data-logging and control system furnished by Honeywell. Installation of the Area 300 Selective Agglomeration Module was deferred until after completion of the column flotation testing since operation of the agglomeration module required modification of the grinding circuit and the clarified water system.

Area 100 - Raw Coal Handling Crushed coal was trucked as needed from Ralston Development Company to a covered storage pile at Amax R&D. A front-end loader was used to move the coal from the pile to a 15-ton receiving hopper for metering into the coal grinding circuit.

Area 100 - Grinding and Classification The PDU utilized two 5-ft dia x 10 ft mills in series for the initial grinding. These mills were charged with steel balls. The two ball mills were followed by a fine Netzsch grinding mill to achieve final liberation of the ash minerals. The fine grinding mill was charged with glass beads.

Clarified recycle water was added to the ball mills along with the coal to form a slurry in mills. The slurry exiting the secondary ball mill was pumped to a cluster of 2- and 3-inch diameter classifying cyclones, and the cyclone overflow was sent to high frequency fine sizing screens to remove any oversize material before flotation. The screen oversize material and the cyclone spigot product were ordinarily pumped to the Netzsch mill for further grinding and returned to the cyclone feed pump.

Area 200 - Column Flotation Area 200 contained a Microcel™ flotation column purchased from Control International. The column was 6 feet in diameter and almost 29 feet tall. Slurry entered the unit below the froth interface at a point approximately 8 feet below the overflow. A downward flow of wash water was distributed into the froth zone to wash out entrained mineral matter and clay particles. The air bubbles were generated by shearing pressurized air that had been injected into tailings slurry pumped through four externally mounted static in-line mixers. The slurry level in the column was controlled by increasing or restricting the flow of tailings exiting the unit using the signal from a pressure transducer to locate the position of the froth/slurry interface.

Area 400 - Clean Coal and Tailings Dewatering Three filters were used to dewater the clean coal. A Westech vacuum drum filter was used as the primary filtration unit while two Netzsch presses filtered the remaining clean coal slurry. The filter cakes were collected in bulk bags for storage or disposal. Filtrates were transferred to an Enviro-Clear thickener where they were clarified for reuse.

Tailings from the Microcel™ column were also sent to the Enviro-Clear thickener for initial dewatering. Flocculants were added to the tailings stream to accelerate sedimentation. The clarified water overflowing the top of the unit was pumped back into the process. The thickened solids formed a slurry of 20 to 30 percent solids which was filtered in two Schriver plate-and-frame presses for land-fill disposal in bulk bags.

Flotation Module Operation and Test Work

Beginning with Taggart coal, the basic strategy was to conduct a planned series of 19 parametric tests, follow-up with 4 or more additional tests to identify optimum operating conditions, and complete the series with a 72-hour round-the-clock production run before moving on to the next coal. The process variables included in the parametric test matrix included MIBC and fuel oil additions, percent solids in the feed slurry, aeration rate, wash water addition, recirculation rate to the aerator, and the feed rate of the coal. Additional tests were made to tune the operation of the grinding circuit for each coal in order to produce the desired particle size distribution and liberation for the flotation separation.

The test work began during January 1996 with Taggart coal and was finished with Hiawatha coal during September 1996. Over 1,000 tons of coal were processed through the PDU during operation of the Flotation Module.

Flotation of Taggart Coal

Twenty-five tests were conducted aimed at tuning the ball mill circuit for grinding the Taggart coal before flotation. It was found that the desired clean coal quality of 1 lb ash/MBtu could be achieved in the PDU at D80=52 µm by using both ball mills, 100-mesh screens and the 3-inch cyclones.

The ground Taggart coal readily floated. In fact, the natural flotability of the coal produced comparable yield and quality values regardless of changes in the operating parameters. Noticeable changes in the yield and quality usually were observed only when the input parameters were varied dramatically. Overall, the quality goal of 1 lb ash/MBtu was met or exceeded in four of the parametric tests. The clean coal yield varied from 58.5 to 96.6 percent while the Btu recovery and residual ash varied from 60.1 to 98.0 percent and 0.77 to 1.23 lb/MBtu, respectively.

Operating conditions were optimized by applying stepwise regression analyses procedures to the parametric testing results. Frother dosage was the most important variable affecting yield and residual ash in the clean coal. Seven optimization tests were performed and the residual ash goal was achieved in two tests. However, the best results were obtained during an earlier parametric test at a feed rate of 4,200 lb/h and a grind of D80=51 µm. A residual ash of 0.99 lb/MBtu at 96.7 percent Btu recovery had been achieved during that test.

Pennsylvania State University requested 50 tons of the clean coal for future combustion testing. Because they planned to prepare CWF from the clean coal, they preferred a coarser particle size distribution than optimum for rejecting ash from Taggart coal. For this reason, the production run was conducted at a coarser grind (D80=71 µm) than preferred by the parametric and optimization testing. Aside from a failed belt splice, uninterrupted operation was achieved showing excellent reliability of the operation during the 72 hours. Overall, 137.7 tons were processed during the run for a weight yield of 95.3 percent, Btu recovery of 96.9 percent, and a clean coal residual ash of 1.22 lb/MBtu.

Flotation of Indiana VII Coal

Flotation of the Indiana VII coal in the PDU commenced during April 1996 and concluded during July. Though the residual ash goal of 2 lb ash/MBtu was difficult to achieve, the operation was considered to be quite successful. All of the PDU test work was performed at a grind of D80=20-24 µm. Some modification of the Area 100 Grinding and Classification circuit was necessary in order to produce such a fine grind. The desired clean coal quality of 2 lb ash/MBtu could be achieved in the PDU by using both ball mills and the fine grinding mill, 270 mesh screens and the 2-inch cyclones.

The residual ash goal of 2.0 lb/MBtu was achieved on five occasions during the parametric testing. Unfortunately, product yield and Btu recovery suffered significantly during these particular tests. Overall, the clean coal yield varied from 12.0 to 89.7 percent while the Btu recovery and residual ash contents varied from 13.2 to 96.4 percent and 1.81 to 3.25 lb/MBtu, respectively. The most important variables effecting performance were fuel oil dosage, particle size, and those variables which effected the bubble size coming from the aerator (aeration rate, frother dosage and recirculation rate). Aeration and recirculation rates and percent solids had strong effects upon the amount of residual ash in the clean coal.

Like the Taggart production run, a failed belt splice was the only operational difficulty during the Indiana VII production run. 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, 77 tons were processed during the run for a weight yield of 75.2 percent, Btu recovery of 82.0 percent, and a clean coal residual ash of 2.33 lb/MBtu. The nominal feed rate was 3,200 pounds per hour.

Flotation of Hiawatha Coal

The operation of the PDU with Hiawatha coal was very successful with project goals achieved on numerous occasions. Overall, the clean coal yield varied during the parametric testing from 12.3 percent to 94.0 percent while Btu recovery and residual ash contents varied from 13.8 percent to 98.7 percent and 1.43 to 2.87 lb/MBtu, respectively. The most important operating variables affecting residual ash were the recirculation rate and wash water usage. However, the operating variable that had the most significant impact on yield was the frother dosage.

A 72-hour extended production run on Hiawatha coal was successfully completed using set points derived from the optimization testing. There were no interruptions during the run. Overall, 155 tons of coal were processed with a weight yield of 81.8 percent, Btu recovery of 88.0 percent, and a clean coal residual ash content of 1.89 lb/MBtu. Forty-four bulk bags of the clean coal were shipped to Penn State for future combustion testing.

Microcelä Scale-up Test Work

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. Test work performed on all three test coals ground in the PDU indicated that the 12-inch Microcel™ unit consistently produced clean coal products with better quality (lower ash) but at lower yields. The differences in performance was attributed to differences in bubble size and retention time in the column. However, the carrying capacities of the two sizes of the Microcel™ column were comparable on a cross-sectional area basis when floating comparable coal slurries ground in the PDU:

	Carrying Capacity, lb/hour/square foot		
Column	Taggart	Indiana VII	Hiawatha
12-inch	129	74	116
6-foot	127	86	125

Lessons Learned from Operation of Flotation Module

Based on the test work and operation of the PDU and Flotation Module, a number of lessons were learned which should be considered when planning similar endeavors in the future. The most important of these lessons dealt with the grinding circuit, the froth handling system and the operation of the bubble generator.

Conclusions of PDU Flotation

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 method for cleaning coal to high quality levels.

The capacity of the column depended upon the properties of the coal being processed and particularly upon the grind size required for liberation of the ash minerals. Frother dosage, aeration rate, wash water addition rate, and the recirculation rate though the in-line aerator were found to be significant operating variables impacting performance of the system. Performance goals could generally be met by adjusting these operating variables to reach product yield and quality specifications. Flotation performance scaled up from a 12-inch column to a 6-foot column in a consistent manner.

SELECTIVE AGGLOMERATION STUDIES

Normal pentane (n-pentane, C_5H_{12}) and n-heptane (C_7H_{16}) were employed as bridging liquids for most of the work. A number of light and heavier hydrocarbons were considered before making the selections. Usage cost was an important reason for favoring light volatile hydrocarbons such as pentane and heptane over heavier hydrocarbons such as fuel oil and kerosene since the light hydrocarbons can be conveniently stripped from the product with steam and reused.

Laboratory Process Optimization

The laboratory process optimization research was performed to determine the operating conditions required to best meet the target premium fuel coal quality and Btu recovery specifications. An important objective was to compare the performance of pentane and heptane so that one of the two might be selected for use during the bench-scale testing to develop design parameters for the PDU. A further objective was to compare the performance of an innovative unitized reactor design combining high- and low-shear mixing in one vessel with the usual plant design employing a series of separate vessels for each shear regime.

The key factors of interest during laboratory-scale selective agglomeration were the efficiency of the separation (recovery of the heating value in the coal and rejection of the ash) and the time required for formation of the agglomerates (phase inversion time). Inversion time depended upon a number of factors: coal and oil concentrations, particle size, oil properties, mixing conditions, and most importantly, upon how rapidly the bridging liquid (oil) spread across the coal particles.

The five bituminous coals responded quite well to laboratory scale selective agglomeration with pentane and heptane bridging liquids. Target residual ash and Btu recovery specifications were easily met for the Taggart, Elkhorn No. 3, Sunnyside, Indiana VII and Winifrede coals ground to the fineness projected from the liberation studies. The target sulfur specification was met when cleaning the Taggart, Sunnyside and Indiana VII coals. The subbituminous Dietz coal did not respond as well and did not meet the target residual ash specification consistently. Heating value recoveries were consistently over 93 percent with heptane and pentane bridging liquids and commonly exceeded 98 percent from the Taggart, Indiana VII, Sunnyside and Elkhorn No. 3 coals.

The heptane to coal and pentane to coal weight ratios were approximately the same for good agglomeration with the two hydrocarbons. The more finely ground slurries, such as the Winifrede slurry, required more bridging liquid. Indiana VII coal also required about 16 lb/t asphalt along with the bridging liquid in order to activate agglomeration.

Agglomeration proceeded satisfactorily in both of the two continuous systems (unitized and two-stage) under a variety of operating conditions (percent solids, impeller speeds, feed rates, etc) until the feed rate was increased to the point where Btu recovery fell sharply and agglomeration ceased. The capacity of the units were at their maximum and expenditures for mixing energy were at their least when feed rates were just below the level where the recovery fell sharply. Changes in operating conditions had little impact upon the amount of residual ash left in the agglomerated clean coal.

The separation performances of the unitized reactor and the two-stage system were similar. Since the unitized reactor system did not offer any power-saving advantages, the two-stage system was recommended for the bench-scale testing because its development was further along and more scale-up information was available for it. Minimum high-shear mixing energy consumptions in the two-stage system were in the 11.8 to 23.6 kWh/ton range when agglomerating minus 325 mesh bituminous coal.

Pentane, pure heptane, commercial heptane and dearomatized (hydrotreated) commercial heptane bridging liquids appeared to be equally capable of agglomerating the ground test coals while effectively rejecting ash minerals. Because of its low boiling temperature, the feed slurries required precooling when pentane was used as the bridging liquid. Dearomatized commercial heptane was the preferred bridging liquid because of its good performance, low cost, mild odor and low concentration of aromatic compounds.

The Dietz coal required addition of a considerable amount of asphalt to activate agglomeration, and acidification to pH 3 or 4 as well, before the high-shear step. Because of these added costs, agglomeration did not appear to be a cost-effective method for cleaning the coal. Therefore Dietz coal was dropped from further consideration during the program.

Bench-Scale Agglomeration and Steam Stripping

The bench-scale testing had three primary objectives:

1. Design, construct, and operate a continuous selective agglomeration system of about 25 lb/h capacity to demonstrate the feasibility of the process.
2. Optimize process conditions to minimize product ash contents and to reduce process costs.
3. Generate reliable design data to insure successful scale-up of the process to the 2-t/h PDU scale.

Parametric testing was conducted on the original five bituminous test coals plus the Hiawatha coal also selected for processing in the PDU. Samples from selected parametric tests were analyzed to learn the distribution of the toxic trace elements during selective agglomeration cleaning.

Bench-Scale System

The bench-scale system assembled by Amax R&D was based on the laboratory-scale testing results and included provisions for recovering and reusing the heptane bridging liquid. The system was designed to produce 25 to 50 lb/h of product on a dry basis and represented a 3- to 10-fold scale-up of the laboratory equipment. The mixing vessels, impellers and stripping columns were especially designed for this project and were fabricated locally. Although capable of operating together, the grinding, agglomeration, steam stripping, and final product dewatering steps were normally carried out separately.

The high-shear vessel was 6 inches in diameter and 6 inches high. It was fully baffled and contained about 2.8 liters of slurry. Shearing was provided by a single radial flow impeller located in the center of the vessel and driven by a 1-1/2 hp variable-speed motor. Impeller tip speeds evaluated were in the 5 to 18 m/s range. Heptane was added prior to the high-shear mixing. When necessary to promote agglomeration, asphalt emulsion was also added to the feed slurry.

The low-shear vessel was designed to provide a residence time of 2 to 5 minutes, insuring agglomerate growth to pellets of sufficient size for screening. The actual vessel was 8 inches in diameter and 18 inches tall and held about 18 liters of slurry. It was also fully baffled. A horizontal baffle divided the vessel into two low-shear mixing zones and two exit ports allowed operation with the vessel full or half full of slurry. Shearing was provided by two radial flow impellers on a single shaft, one located at the center of each mixing zone.

The agglomerated coal was recovered on a 10-inch x 16-inch inclined vibrating screen. Spray water was applied to the screen to wash residual tailings slurry from the agglomerates. The tailings (screen underflow) discharged into a 4-inch diameter by 18-inch froth skimming column designed to collect any heptane-bearing carbonaceous material floating in the tailings stream.

Heptane was removed from the agglomerated product by direct steam stripping. A single stage of steam stripping was used during initial testing. This stripping vessel was a 4-inch diameter column approximately 52 inches tall. A portion of this column was filled with 5/8-inch stainless steel pall rings to distribute flows and prevent excessive turbulence and back-mixing. Steam entered the column below the packing and the vapor (heptane and water) exited from the top at near-ambient pressure, while the agglomerated feed entered the top of the column and the product slurry discharged from the bottom through a U-tube. This provided a steam flow counter-current to the process slurry flow.

The stripping circuit was modified later to include a new vessel which received the agglomerated coal ahead of the above described column. This allowed the bulk of the heptane to be removed in the new first-stage stripper followed by additional heptane removal in the original column, which was modified to operate at elevated pressures (to 15 psig) and temperatures. This two-stage circuit better simulated the developing 2-t/h PDU stripping circuit design which also used a counter-current steam flow scenario.

The vapor from the steam stripping circuit was condensed in a coil submersed in a water bath. The bath was serviced with sufficient tap water to cool the condensed heptane/water stream to about 38º C. Once condensed and cooled, this heptane/water mixture was separated by gravity in a column where the heptane overflowed from the top and the water from the bottom via a U-tube.

An on-line data acquisition system was included in the selective agglomeration test unit. This system allowed real-time data acquisition of operating temperatures and provided a data log into which manually obtained operating conditions were entered.

Bench-Scale Agglomeration Summary

The results of the bench-scale agglomeration test work indicated that the product ash specification of 1 to 2 lb/MBtu, as well as the Btu recovery goal of at least 80% on a run-of-mine coal basis, were met for all of the coals tested. Of paramount importance in achieving these product ash levels was the particle size distribution to which the coal was ground. If sufficient mineral-matter liberation were not achieved, the desired product grade could not be attained. The coarsest particle size distribution to which each coal was ground while still achieving the product ash specifications are listed below. Typical product ash and Btu recovery values attained when operating at optimized conditions for the grind sizes shown are included in this tabulation:

	PSD, µm		Ash	Btu Recovery, %	
Coal	D80	MMD	lb/MBtu	Agglomeration	Run-of-Mine
Taggart	33	23	0.95	99.1	93.5
Sunnyside	60	34	1.79	98.3	88.6
Indiana VII	22	15	1.95	99.0	89.6
Elkhorn No. 3	68	39	1.69	96.8	91.6
Winifrede	12	7	1.91	99.2	88.8
Hiawatha	65	41	1.85	99.6	99.6

Various operating conditions were used to achieve these typical agglomeration results, i.e., several combinations of residence times, energy inputs, and heptane levels could ultimately achieve similar results. No difference was seen between the performance of the two grades of heptane or between fresh and recycled heptane. It was found however that the following guidelines should be followed to insure that consistent results are achieved:

1. Sufficient heptane must be used during high shear to achieve phase inversion and the formation of microagglomerates. Typically, the amount of heptane required increased with decreasing coal particle size and decreasing coal rank.
2. Sufficient energy or impeller tip speed (10 to 18 m/s) must be applied during high shear mixing to achieve complete dispersion of the heptane and enough particle-to-particle contact to form microagglomerates.

3. Sufficient residence time (typically 30 to 60 seconds) must be provided during high shear mixing to form microagglomerates (that is, to achieve phase inversion). Residence time requirements depended primarily on coal fineness and rank but were reduced by higher tip speeds.
4. The use of higher solids concentration during high-shear mixing reduced energy input requirements.
5. For lower rank and oxidized coals, an activator such as asphalt may be required to achieve phase inversion during high-shear mixing.
6. Sufficient heptane must be provided to allow agglomerate growth to the 2 to 3 mm size range during low-shear mixing. However, too much heptane resulted in poor agglomerate formation.
7. The production of consistent size and shape agglomerates in the 2 to 3 mm diameter range was paramount to achieving good recovery on the washing screen and low residual ash levels in the product.
8. Impeller tip speed (agitation intensity) during low shear mixing needed to be in the vicinity of 5 m/s for growth of well formed agglomerates with sufficient strength to be recovered on the vibrating screen. Too mild or too intense agitation resulted in poorly formed agglomerates and higher residual ash contents.
9. Residence time during low-shear mixing was found to have little effect on agglomerate growth since ultimately, agglomerate formation was controlled by heptane dosage and low-shear agitation intensity.
10. The discharge of the low-shear mixing vessel should be at the same elevation as the impeller. This configuration insured that continual discharge occurred under all operating conditions of the low shear mixer.
11. Higher solids concentrations during low-shear mixing resulted in higher residual ash levels in the products and made agglomerate growth difficult to control.
12. The vibrating screen used for agglomerate recovery must have sufficient forward linear motion to transport the agglomerates across the screen deck.
13. Screen spray water was required to wash the mineral-matter bearing process water off of the agglomerated coal.
14. The froth skimmer on the screen underflow recovered clean coal that was lost through the screen at times.

Overall, the selective agglomeration process was very robust. It either worked well or did not work at all. Therefore, as long as the coal grinding provided sufficient mineral-matter liberation and a consistent low-shear product of the appropriate pellet size was produced, the desired product grade was achieved along with consistently high Btu recovery.

Heptane Recovery

The continuous bench-scale steam stripping was usually carried out independently of the agglomeration circuit. Agglomerates were diluted to 10 to 15 percent solids in a well mixed feed tank, and the feed to stripper cycled on and off as required to maintain a first-stage stripper temperature between 90 and 95º C. Feed to the second-stage steam stripper was set at a fixed volumetric flow rate to control the combined system throughput. The pressure and temperature in the second-stage stripper were controlled manually to maintain a target operating pressure and operating temperature, typically 10 psig and 117º C, respectively, by adjusting the slurry and steam flows. The slurry level in the second-stage stripper was also controlled via a manual discharge valve. The initial testing with the single-stage stripping system indicated that the column could handle a coal feed rate approaching 20 lb/h without plugging.

The residual concentrations of heptane in the stripper products were determined by gas chromatography of a methylene chloride extract using procedures developed with the assistance of Huffman Laboratories of Golden, Colorado.

Steam Stripping Summary

In general, steam stripping to remove heptane from agglomerated products was a straight forward operation. The steam was applied directly to the reslurried agglomerates to evaporate the heptane, and the ratio of heptane to water in the exiting vapor phase from the stripping circuit was minimized to insure that steam consumption was kept as low as possible. A two-stage system should be used due to the advantage of carrying out steam stripping at elevated pressures and temperatures (lower residual heptane concentrations).

Continuous two-stage stripper testing was carried out at operating temperatures of approximately 92º C and 115º C in the first- and second-stage strippers, respectively. Residence times were 5 and 10 minutes for the first- and second-stages, respectively. Under these conditions, residual hydrocarbon concentrations on the order of 1,000 to 3,000 ppm (0.1 to 0.3%) on a dry coal basis were achieved in the final product. These residual concentrations represent about 99 percent heptane recovery and appeared to be independent of the coal tested and the type of heptane used, that is, either pure grade or commercial grade.

The effects of various steam stripping operating variables on the residual heptane content of the stripped products are summarized below:

1. No benefits were gained by providing residence times greater than five minutes when steam stripping heptane from agglomerated products at ambient-pressure boiling temperatures.
2. Steam stripping at elevated temperatures, as achieved by increased operating pressures, resulted in lower residual heptane concentrations.

3. Two stages of steam stripping achieved lower trace heptane concentrations than a single stage of steam stripping due to the increased temperature in the second stage.
4. No benefits were gained by use of large flows of excess steam.
5. Increasing the solids concentration at which the steam stripping was carried out had no detrimental effect on residual heptane concentrations.
6. The presence of asphalt (used as an activator during agglomeration) resulted in lower residual hydrocarbon concentrations under otherwise similar stripping conditions.
7. Regardless of whether a commercial or pure grade of heptane was used during agglomeration, total residual hydrocarbon concentrations were similar.

No major operational difficulties were encountered during the bench-scale stripper testing. Complete condensation was consistently achieved with minimal carryover of coal from the stripping circuit. Separation of the condensed water and heptane was easily accomplished in a gravity separator column with the heptane overflowing from the top and the water exiting the bottom. This separation was complete with only minimal solubility (<10 ppm) of heptane into the water phase.

Tailings Heptane Analysis

A set of agglomeration tailings samples (froth skimmer underflow) was analyzed for residual heptane content in order to plan the design of tailings disposal system for the PDU and a commercial plant. The samples were from an Elkhorn No. 3 coal agglomeration test, and the tailings contained about 50 percent ash on a dry solids basis. No more than 10 ppm of n-heptane was detected in any of the samples, except for the tailings filter cake, which contained 567 ppm heptane on a dry solids basis. There was less than 1 ppm of heptane detected in the tailings filtrate. These results indicated that tailings disposal in conventional waste disposal sites should not be a problem.

PDU Operations

The conceptual design of the Agglomeration Module for the PDU was a collaborative effort between Bechtel, Entech and Arcanum, and the detailed design was done by Bechtel Corporation with support from Entech Global engineers.

Process and Plant Description

The Selective Agglomeration Module contained three areas which are described below:

- Area 300 - Agglomeration
- Area 300 - Steam Stripping
- Area 300 - Heptane Recovery and Recycle

In addition to these specific areas, the module included a nitrogen gas blanket system, equipped with oxygen analyzers, to contain the heptane vapor and avoid explosive mixtures with air. The module also contained provisions for supplying steam and chilled water to the components. Hydrocarbon vapor detectors and alarms were installed in the rooms in and around the agglomeration areas. The equipment in Area 300 was sized for production of 2.0 st/h Sunnyside clean coal. The module was capable of cleaning Taggart coal to 1 lb/MBtu ash at a similar capacity, but the capacity was less for Indiana VII coal.

The ground coal slurry for selective agglomeration was provided by the existing Area 100 Grinding and Classification circuit and the clean coal and tailings were dewatered in the existing Area 400 thickener and filters. As with the column flotation, all areas of the PDU were operated from a central control room utilizing the integrated graphic data-logging and control system furnished by Honeywell.

Area 300 - Agglomeration

The ground coal from Area 100 was metered to one of two variable-speed high-shear mixing vessels along with n-heptane to begin the agglomeration. One mixer had a volume of 35 gallons and the other 75 gallons and they were arranged so that they could be used individually, in parallel or in series, in order to provide the requisite high-shear retention time to achieve inversion. From there the slurry proceeded to a 400-gallon low-shear mixing tank and on to a 48-mesh inclined vibrating screen where the agglomerates were collected and rinsed with spray water. The fine refuse tailings passing through the screen deck flowed to a froth skimming tank before being pumped to the Enviro-Clear thickener in Area 400. Any coal which passed through the screen deck floated to the surface of the slurry and was collected by the skimmer.

Area 300 - Steam Stripping

The Selective Agglomeration stripping circuit generally followed the two-stage system studied during the bench-scale testing. In this part of the module, the agglomerated coal from the screen and the froth from the froth skimmer were mixed with preheated water and pumped to Stripper A where the mixture was heated by the hot vapor leaving Stripper B. The hot slurry from Stripper A in turn was pumped to Stripper B where it was further heated by injection of 25 psig steam. The stripped slurry leaving Stripper B was pumped though a heat exchanger to the Area 400 clean coal filters.

Area 300 - Heptane Recovery and Recycle

The heptane in the vapor exiting the top of Stripper A was condensed in an air cooler for reuse in the agglomeration circuit. The water in the condensate was removed by a gravity separator and passed through a carbon filter so that it could either be reused or discarded into the local sewer system.

Selective Agglomeration Module Operation and Test Work

Beginning with Hiawatha coal, the testing strategy was to conduct separate parametric tests to optimized agglomeration, agglomerate recovery (screening and froth skimming), and stripping, respectively, for each coal. These would be followed by a 72-hour production run on each coal.

The test work began during January 1997 and was finished during July 1997. About 1,000 tons of coal were processed through the PDU during operation of the Selective Agglomeration Module.

Agglomeration of Hiawatha Coal

A considerable effort was devoted to the establishment of proper operating conditions in the high-shear and low-shear mixers when testing the first of the three coals. The parameters which were studied included heptane/coal ratio, impeller tip speeds and retention times. Good results were generally obtained with heptane/coal ratios between 0.24 and 0.30 to one for Hiawatha coal ground to D80=40 µm. Good agglomeration was achieved at retention times between 40 and 80 seconds at tip speeds of 11 to 15 m/s in the high-shear mixer. Some instability was noted under some operating conditions in the low-shear mixer while attempting to grow 2- to 5-mm agglomerates. The most consistent operation appeared to be with a 5 m/s tip speed and operation with the vessel half full of slurry.

Combinations of retention times and temperature were investigated for stripping the heptane from the clean coal. Relatively low residual heptane concentrations were reached consistently. Typical values were in the 2,000 to 3,000 ppm range, and no clear trend was seen relating temperature and retention times to lower residual heptane concentrations. The heptane was recovered from the vapor leaving Stripper A without difficulty.

The variable most effecting performance was the particle size distribution of the ground coal. A D80 of less than 40 µm appeared to be essential for meeting a 2 lb/MBtu ash specification. Btu recoveries were consistently over 98 percent during these tests.

The Hiawatha production run successfully met expectations. The average feed rate was 3,839 lb/h (dry basis) and the grind was D80=42 µm. There were two periods of downtime due to a pump failure. Overall, 106.5 tons were processed during the run for a clean coal yield of 92.8 weight percent and the following operating results:

- Heptane/Coal Ratio - 0.30 to 1
- Mixing Energy - 17 kWh/st feed coal
- Steam Consumption - 1,320 lb/st clean coal
- Residual Heptane:
    - Clean coal - 2,951 ppm dry coal basis
    - Tailings - 1,470 ppm total solids basis
- Residual Ash - 1.93 lb/MBtu (2.78 percent)
- Residual Sulfur - 0.35 lb/MBtu (0.50 percent)
- Btu Recovery - 98.9 percent

Agglomeration of Taggart Coal

A similar set of parametric tests were conducted on the ground Taggart coal, and the relationship between operating variables and performance seen for agglomeration and stripping were similar to the relationships seen during the Hiawatha testing.

Again, the variable most effecting performance was the particle size distribution. A D80 of less than 30 µm was needed to produce clean coal containing less than 1.0 lb of ash per million Btu. Btu recoveries were consistently over 97 percent during these tests.

The Taggart production run also was successful. The average feed rate was 3,305 lb/h (dry basis) and the grind was D80=30 µm. There was a short downtime due to a pump failure. Overall, 115.7 tons were processed during the run for a clean coal yield of 96.7 weight percent and the following operating results:

- Heptane/Coal Ratio - 0.39 to 1
- Mixing Energy - 16.1 kWh/st feed coal
- Steam Consumption - 1,553 lb/st clean coal
- Residual Heptane:
    - Clean coal - 5,115 ppm dry coal basis
    - Tailings - 4,094 ppm total solids basis
- Residual Ash - 1.06 lb/MBtu (1.59 percent)
- Residual Sulfur - 0.42 lb/MBtu (0.63 percent)
- Btu Recovery - 99.2 percent

Agglomeration of Indiana VII Coal

A similar set of parametric tests were conducted on the ground Indiana VII coal as conducted on the two previous coals, and the same relationships were seen. There was one difference, though. Between 5 and 10 lbs of asphalt were required to activate agglomeration of the Indiana VII coal because of its lower rank. The Indiana VII coal also required very fine grinding (D80=20 µm) in order to reach a residual ash content of less than 2.0 lb/MBtu in the clean coal. Despite the fine grinding, Btu recoveries consistently exceeded 99 percent.

Since the PDU did not have enough filter capacity to operate continuously for 72 hours on the finely ground Indiana VII coal, the PDU production run was operated at a coarser grind so that the reliability and robustness of the selective agglomeration process could be demonstrated over a longer running time. The run was conducted at an average feed rate of 3,491 lb/h (dry basis) and a grind of D80=67 µm. There were two short downtimes due to the failure of a tailings filter and a control valve. Overall, 113.5 tons were processed during the run for a clean coal yield of 93.5 weight percent and the following operating results:

- Heptane/Coal Ratio - 0.35 to 1
- Mixing Energy - 36.6 kWh/st feed coal
- Steam Consumption - 1,778 lb/st clean coal
- Residual Heptane:
    - Clean coal - 3,967 ppm dry coal basis
    - Tailings - 472 ppm total solids basis
- Residual Ash - 3.02 lb/MBtu (4.19 percent)
- Residual Sulfur - 0.31 lb/MBtu (0.43 percent)
- Btu Recovery - 99.9+ percent

The Btu recovery appears to be higher than for the other coals because of the asphalt used to activate agglomeration.

Discussion of Selective Agglomeration Module Operating Results

In general, changes in most of the agglomeration operating variables had only small effects on the amount of residual ash in the clean coal. It was only important that the coal be ground fine enough for liberation of the ash minerals. Generally, growth of the agglomerates to the 2- to 5-mm range provided the best operation. Similarly, once set within the proper range for inversion, agglomerate growth and screening efficiency, small changes in operating variables such as heptane dosage, shear rates, retention times and spray water usage had little effect upon the percentage recovery of coal. Under these conditions product recovery was very good, typically over 98 percent Btu recovery.

Asphalt was required to activate agglomeration of the Indiana VII coal, which was of high volatile C rank rather than the high volatile A rank of the other two coal tested in the PDU. Despite the activation, the Indiana VII coal still required a longer retention time in the high-shear mixing vessel than did the Hiawatha and Taggart coals.

The stripping and heptane recovery system was equally robust in its operation. At least 98 percent of the heptane was stripped from the coal, and there was no loss of performance or change in the operation when the condensed heptane was reused for agglomerating ground coal.

The design of the Selective Agglomeration Module provided a good work environment. All of the flows were fully contained so there was no odor from the heptane or high humidity from escaping steam. Neither was there any noticeable odor from the clean coal and fine refuse filter cakes. As seen while operating the Flotation Module, the Area 400 product dewatering step, especially clean coal filtration, was the leading bottleneck restricting production of clean coal.

Disposition of the Coal and PDU

As provided in the contract between the DOE and Cyprus Amax, the entire PDU was dismantled upon completion of the selective agglomeration task and the equipment packed and shipped to FETC Pittsburgh. A portion of the clean coal from the flotation and selective agglomeration production was shipped to Pennsylvania State University for future combustion testing. Some of the clean coal was sent to a local business as fuel in a light aggregate kiln and the remainder was consigned to a land fill for disposal.

REJECTION OF TOXIC TRACE ELEMENTS

In response to provisions of the 1990 Clean Air Act Amendments (CAAA), the reduction in the concentration of specified toxic trace elements was monitored during the bench-scale and PDU advanced cleaning. The elements of interest were antimony, arsenic, beryllium, cadmium, chromium, cobalt, lead, manganese, mercury, nickel, selenium and chlorine which could become hazardous air pollutants during combustion.

The amounts of the trace elements found in the samples varied from coal to coal, and the residual amounts in the coals after cleaning were dependent upon the source coal. Generally, when prepared from the same test coal, the advanced flotation clean coal and the selective agglomeration clean coal contained about the same amounts of the trace elements. Cadmium was detected in only a few of the ROM and test coals. It was detected in one of the clean coals, though, and in a number of the fine refuse samples. Lead was not detected (< 2 ppm) in any of the Sunnyside or Hiawatha samples.

The residual mercury and selenium analyses were of particular interest. Mercury was not detected (< 0.01 ppm) in some clean coal samples (Sunnyside, Indiana VII and Wentz Mine Taggart) and ranged up to 0.03 ppm in others (Winifrede coal cleaned by flotation). The fine refuse samples contained between 0.01 and 0.12 ppm mercury. Selenium analyses ranged from 0.41 ppm up to 5.7 ppm in the clean coals and from 0.30 ppm up to 7.1 ppm in the various fine refuse samples.

Results of Advanced Cleaning

There were substantial reductions (25 to 75 percent) in the concentrations of some impurities, especially ash, arsenic and manganese, on a heating value basis from the amounts in the as-received test coals. On the other hand, there was little or no reduction (less than 25 percent or negative) in the amounts of antimony, beryllium, cobalt, nickel, and selenium in the as-received test coals on the same basis. Little or no reduction in the concentration of an impurity means that the impurity is closely associated with or actually part of the carbonaceous components of the coal, whereas a substantial reduction signifies an association with the mineral matter in the coal. The reduction of other impurities, such as total sulfur, pyrite sulfur, cadmium, chromium, mercury, lead and chlorine varied from coal to coal. Reductions from the trace-element concentrations found in the ROM parent coals were generally greater than the reductions from the as-received test coals on a heating value basis.

Of particular interest, the PDU fine coal cleaning was effective for reducing the lb/MBtu concentration of mercury in the Taggart and Indiana VII coals by 39 percent or more. Similar reductions had been seen earlier for the bench-scale cleaning of Winifrede, Taggart, Sunnyside, Indiana VII, and Elkhorn No. 3 coals. The Hiawatha coal contained less mercury to begin with than the other coals, and the advanced fine coal cleaning had less impact upon the final concentration of mercury in the clean product from that coal than it did on the mercury concentrations in the other coals.

The reduction in the concentrations of the trace elements was confirmed by comparing the analyses of the clean coals and the corresponding fine refuse products. In particular, the concentration of mercury in refuse samples were two to four times as high as the concentrations in the ROM parent and PDU test coals, even in the case of the Hiawatha coal. The concentrations of chromium and manganese were very high in some of the fine refuse samples due to metal worn off the balls in the grinding mills, particularly when the stirred ball mill was used for the bench-scale grinding.

Summary

The two advanced cleaning processes -- column flotation and selective agglomeration -- appeared to be equally effective for reducing the concentrations of impurities in coal and equally effective for cleaning coal to premium fuel specifications. For certain coals, physically cleaning to premium fuel specifications substantially reduced the concentrations of some of the hazardous air pollutant trace elements, especially arsenic, chromium, cobalt, lead, manganese, mercury, selenium, and chlorine, in the coals. As such, fine coal cleaning can be a useful part of a hazardous air pollutant control strategy for coal-fired utilities.

PREPARATION OF COAL WATER SLURRY FUEL

The form in which the fuel is delivered to and handled by the end-user is an important consideration when marketing premium fuel prepared from coal. A pumpable, highly-loaded slurry of coal and water (coal-water slurry fuel or CWF) appears to be a highly attractive option.

To be a viable substitute for oil and natural gas, CWF must be fluid enough that it may be pumped and atomized efficiently yet it must not contain any more water than necessary to achieve such fluidity. For ordinary boiler firing, a suitable balance between loading and fluidity seems to be at 60 to 65 percent coal loading and 200 to 500 cP viscosity. Higher loadings are more desirable, though, so parametric tests were conducted on the clean coals from the bench-scale circuits to optimize the CWF formulations. The stability of the formulations was also examined during the optimization since separation of hard-packed sediments can lead to handling problems when using CWF.

CWF Preparation and Evaluation

CWF was produced in the laboratory by blending water and reagents with partially dried clean coal filter cakes. Slurry preparation tests were made on the filter cakes directly and on filter cakes after manipulation of the particle size distribution (PSD) to improve the packing of the particles in the slurry. The viscosity of most the CWF samples was reduced by adding a commercial naphthalene sulfonate dispersant (A-23M) to the mixture in order to disperse flocculated coal particles. Particle size manipulation was accomplished by regrinding a portion of the clean coal to a finer size distribution and blending the reground portion back with the unground portion to provide a bimodal distribution.

Effects of CWF Preparation Variables

The A-23M dispersant served to reduce the viscosity of the slurry thereby allowing formulation of higher loading CWF. The addition of A-23M allowed preparation of Taggart slurries containing 60 to 65 percent coal rather than 52 percent coal. There was little improvement in the loading of Taggart CWF when the A-23M additions increased above 0.5 percent by weight of the coal in the slurry. Similar patterns were seen for the other clean coals, and the amounts of A-23M used in most of the tests were at the point where the loading vs dispersant addition curve began to plateau.

Rank of Coal

The test coals were all high volatile bituminous coals but varied enough within this rank (as defined by the ASTM D-388 moist, mineral-matter free heating value) that a difference was seen in the loadings of optimized slurries during the parametric testing. Taggart, the highest ranking coal, provided the highest slurry loading, and Indiana VII, the lowest ranking coal, provided the lowest loading. The high ranking Winifrede coal did not follow the pattern shown by the other coals because it had been ground much finer than the other coals – to minus 20 µm.

The equilibrium moisture of the parent coal was an alternative indication of the rank of a high-volatile coal. It varied from 1 percent in the Taggart coal on up to 14 percent in the test coal from the Indiana VII seam. Thus, the lower loadings of CWF from the lower ranking coals can be attributed, in part, to the moisture which soaked into the coal particles and was not available to provide fluidity to the slurry.

Particle Size and Distribution

It was necessary to grind the coals to differing degrees of fineness in order to achieve the target ash rejection during cleaning. The fineness of grinding had a significant impact upon the loadings of the optimized slurries. There was some overlap with the effect of varying rank because Taggart coal, the coal which did not require as fine a grind as the other coals, also had the highest rank of the six coals. A multiple regression analysis of the data from the test program showed that the particle-size effect was a more significant effect than the effect of the rank of the parent coal.

The fineness of the grind also had an impact on the amount of A-23M required for producing CWF. In this case, a multiple regression analysis showed little or no effect of coal rank on the amount of dispersant required for the slurry.

The natural PSDs of the clean coals were quite uniform with little concentration of particles in any particular size range so the distributions were adjusted to allow preparation of higher-loading CWF. Since the PSD adjustments were accomplished by grinding a portion of the clean coal, this meant that the overall distribution also became finer. Plotted results showed that the PSD adjustment improved the loading of the CWF samples but only by a few percent. Because of its cost and complexity and limited benefit, the PSD adjustment did not appear to be a commercially viable approach to increasing the loading of CWF.

Coal Cleaning Procedure

The coal cleaning method, flotation or selective agglomeration, made little or no difference on the quality of the CWF. Particle size and coal rank were the main factors affecting slurryability. The clean coal from the selective agglomeration had been stripped with steam to recover the heptane bridging liquid and the small amount of residual heptane did not appear to affect the slurryability of the clean coal.

Stabilization

Hard-pack cake formed in CWF samples formulated with A-23M dispersant when they were stored overnight. Xanthan gum (Flocon) was investigated as a stabilizing reagent to inhibit such sedimentation. A soft easily-remixed sediment formed when 800 ppm Flocon 4800C gum was added to the CWF, even when the samples were stored for a week or more. Unfortunately, the xanthan gum also increased the viscosity of the slurries, so the coal loadings had to be reduced by about 1.4 percent in order to maintain the desired fluidity.

Because of the cost of the Flocon (several dollars per lb) and because of its uncertain effectiveness over long storage periods (months rather than weeks), it is recommended that CWF should be prepared without using a stabilizer and that it should be kept in mixing tanks and burned soon after preparation.

Commercial Specifications of Premium CWF

Selected, but readily available, coals can be cleaned to less than 2 lb ash per million Btu. Fine grinding was required, but one could still prepare CWF from at least two Eastern and two Western coals that would contain more than 8,500 Btu/lb and meet the premium fuel ash specification. CWF prepared from a lower-rank (high-volatile C) Midwestern coal had heating values between 7,000 and 7,500 Btu/lb.

CWF slurries prepared from high volatile A coals contained 60 to 62 percent coal and were formulated with A-23M dispersant to have viscosities of less than 500 cP at 100 s^{-1}. They were intended for use soon after preparation, and provisions would be needed in the fuel system for frequent mixing and for draining fuel lines when not in use. If desired, the fuel could be formulated with a stabilizer such as Flocon, and perhaps with less dispersant, to alleviate some of the need for remixing and line drainage by the user, but the loading and heating value specifications of the CWF would have to be reduced if one wished to maintain a viscosity of less than 500 cP.

As a cost-saving alternative, CWF from the high volatile A coals could also be formulated without dispersant. Such fuel would contain about 52 percent coal and have a higher heating value in the 7,200 to 7,500 Btu/lb range.

PROPERTIES OF PREMIUM FUEL PRODUCED IN PDU

The clean coal produced during the extended production runs in the PDU provides examples of the quality of the premium fuel which can be produced by advanced physical cleaning of fine coal. The properties of these clean coals are summarized below and a comparison made between the two technologies.

As indicated below, particularly good quality fuel was prepared from the Taggart and Hiawatha coals in the PDU. However, earlier laboratory and bench-scale testing had also shown that Sunnyside and Elkhorn No. 3 coals may also be good source coal candidates for preparation of premium fuel.

Composition and Yield of Clean Coal and CWF

The compositions of the clean coals (dry basis) from the PDU extended production runs (three by flotation and three by selective agglomeration) are presented below:

	Taggart Coal		Hiawatha Coal		Indiana VII Coal	
	Agglomeration	Flotation	Agglomeration	Flotation	Agglomeration	Flotation
Ash, %	1.64	1.83	2.73	2.70	4.27	3.23
, lb/MBtu	1.09	1.22	1.91	1.89	3.08	2.33
Sulfur, %	0.63	0.72	0.50	0.63	0.43	0.59
, lb/MBtu	0.42	0.48	0.35	0.44	0.31	0.43
HHV, Btu/lb	15,072	15,045	14,302	14,296	13,836	13,849
Btu Recovery, %	99.2	96.9	98.9	88.0	99.9	82.9

The sulfur specification of less than 0.6 lb/MBtu was met in all six instances, and the ash specification of less than 2.0 lb/MBtu was met for the Taggart and Hiawatha coals. It should be pointed that the extended production runs were not necessarily conducted at the operating conditions where the ash specification would have been met. Specifically, less than 1.0 lb/MBtu ash coal was produced during certain flotation and selective agglomeration parametric tests on the Taggart coal. Similarly, less than 2.0 lb/MBtu ash coal was produced during certain flotation and selective agglomeration parametric tests on the Indiana VII coal albeit with some difficulty operating the grinding and dewatering portions of the PDU circuit.

Btu recoveries met project goals in each instance except for the flotation cleaning of the Indiana VII coal where the recovery would have fell short of the project goal of 80 percent Btu recovery from the ROM coal.

Slurry preparation tests were not conducted on the filter cakes from the PDU extended operations. However, there was sufficient information available from the slurry preparation testing done on the bench-scale production to allow projection of slurry loading of 60 percent for CWF prepared from the Taggart and Hiawatha clean coals and 52 percent for CWF prepared from Indiana VII coal. Heating values would be on the order of 8,500 to 9,100 Btu/lb for the Taggart and Hiawatha CWF and 7,200 Btu/lb for the Indiana VII CWF.

Ash Properties of Clean Coals

It was found that the 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 cleaned by column flotation. A similar pattern was seen for these two coals when cleaned by selective agglomeration except that the decline was not as great in the case of the Indiana VII coal. The fine coal cleaning did not have much impact upon the fusion temperatures of the ash in the Hiawatha coal. The reducing atmosphere ash-softening temperatures of the clean coals were as follows:

	Agglomeration		Flotation	
	Feed Coal	Clean Coal	Feed Coal	Clean Coal
Taggart	2552 ºF	2396 ºF	2485 ºF	2235 ºF
Hiawatha	2145 ºF	2181 ºF	2141 ºF	2102 ºF
Indiana VII	2479 ºF	2362 ºF	2350 ºF	2050 ºF

Except for titanium dioxide, and iron oxide in the case of Taggart coal, the concentrations of the ash constituents, including the alkali metals, were significantly reduced from the amounts in the feed coals on a heating value (lb/MBtu) basis by both the flotation and the selective agglomeration process. The slagging and fouling characteristics of the ashes were little changed by the cleaning and remained in the low and medium categories.

Comparison of Technologies

A comparison of the PDU results, suggests that column flotation and selective agglomeration were equally effective methods for cleaning coal to premium fuel specifications. Undoubtedly the results were coal-specific but there were indications from the Btu recovery comparisons that selective agglomeration provided a somewhat higher product yield. This difference in yield may be most noticeable when the flotation response of the coal was poor or when very fine grinding was needed to liberate the ash minerals as true for Indiana VII coal.

ECONOMICS OF COMMERCIAL PREMIUM FUEL PRODUCTION

An important goal of this project was to develop the process so as to produce premium fuel at a cost of less than $2.50 per million Btu including the mine-mouth cost of the raw coal. For this reason, Bechtel conducted commercial production cost studies for two conceptual plants producing premium CWF by advanced physical fine coal cleaning. One plant utilized column flotation for the cleaning technology, and the other plant utilized selective agglomeration for the cleaning technology. Plant design and operating parameters for the two conceptual facilities were based on the results of the bench-scale and PDU testing described in this report.

Conceptual Plants

The conceptual premium CWF production plants would be located in an industrial area of an Ohio valley state and near potential customers for the fuel. The plants would produce 2.5 million short tons of CWF per year containing 1.5 million short tons of coal. Feed stock for the plants would be purchased from mines in the central Appalachian area that produce coal that upgrades in a manner similar to the Taggart, Elkhorn No. 3, Sunnyside and Hiawatha test coals used for the bench-scale and PDU testing. The CWF would contain 60-62 percent coal (8,900-9,400 Btu/lb), less than 2.0 lb ash per million Btu, less than 0.6 lb sulfur per million Btu, and would have a viscosity of 500 cP.

Bechtel assembled capital and operating data for the study and developed equipment flowsheets and levellized cost projections for producing premium CWF by fine grinding and application of the two advanced cleaning technologies. The conceptual plants included sections for coal receiving and storage, crushing and grinding, advanced physical cleaning, clean coal dewatering, CWF preparation, storage and load-out, tailings handling, and recycle water clarification. The selective agglomeration advanced cleaning section included facilities for heptane recovery and reuse. For the base case, the plants would operate 24 hr/day, 7 days/week for a scheduled 7,600 hours per year.

An advanced flotation premium fuel production plant was found to be less expensive to place into service than a selective agglomeration plant:

	Estimated Cost, $millions	
	Flotation	Agglomeration
Construction and Start-up Cost	69.6	97.2
Working Capital	10.0	11.0
Total	79.6	108.2

Total fixed and variable operating and maintenance (O&M) costs for producing CWF by the two cleaning methods were estimated to be $2.15/MBtu and $2.42/MBtu for advanced flotation and selective agglomeration, respectively. The cost of the feed coal delivered to the premium fuel plants (at $1.24/MBtu or about $32.40/st) was the major part of these estimates. The cost of the feed coal included preparation and loading costs at the mine and $5.20/st ($0.20/MBtu) for freight to the premium fuel production plant. In each case, the cost of producing premium CWF compared favorably with the cost of No. 6 fuel oil (about $3.35/MBtu or $0.50/gallon at the time) and met the project goal of less than $2.50/MBtu.

Conclusions and Recommendations of the Cost Study

The estimated cost of commercial production of premium CWF using either column flotation or selective agglomeration was encouraging. Column flotation was particularly promising because of its significantly lower processing cost at $0.91/MBtu compared to the cost of $1.18/MBtu estimated for selective agglomeration.

It was found during the sensitivity analysis that one of the significant factors vitally affecting production costs was the annual sustainable production rate. Product costs would escalate drastically if the annual product rate of 1.5 million short tons cannot be achieved in plants built according to the conceptual designs presented in the study. Two significant technical factors that could adversely affect production are (a) reduced plant availability due to worse than anticipated plant operability or maintenance requirements and (b) feed coal that is either harder to grind or requires finer grinding than expected. The latter possibilities would reduce the grinding capacity of the plants and have an adverse effect upon the annual production of CWF.

These technical uncertainties are best resolved by operating experience with a larger scale plant and for a longer term than was possible with the PDU. Installation of a demonstration/production plant using a single train of commercial equipment with a capacity in the 125,000 to 150,000 short ton per year range was suggested. Operation of such a plant would also afford an opportunity for customer testing of the fuel.

NEAR-TERM APPLICATION OF ADVANCED TECHNOLOGIES

Near-term application was an extension of the premium fuel project to specifically address the use of advanced flotation and selective agglomeration processes for recovering 28-mesh x 0 coal lost in existing coal preparation plants. The goal was to produce clean coal which could be sold in existing markets. Such applications would represent immediate near-term benefits from the project and would complement the long-term benefits gained from the production of premium fuel from coal.

Both of the physical fine-coal cleaning technologies being developed for production of premium fuel (advanced column flotation and selective agglomeration) were considered for near-term applications. Column flotation technology was considered to be ready for commercial fine coal recovery applications. However, only non-recovery systems with diesel fuel, kerosene or heating oil bridging liquids were considered ready for near-term applications of selective agglomeration since the technology for recycling volatile bridging liquids had not been developed much beyond the laboratory stage at the time this task began.

Locations for Near-Term Applications

As a first step, team-member Amax Coal Company suggested three locations for application of the new technologies. The three locations were the Ayrshire Preparation Plant in Indiana, the Lady Dunn Preparation plant in West Virginia and the Wabash Preparation Plant in Illinois. Samples were collected from each plant for laboratory amenability tests, and Bechtel engineers obtained the existing plant layout and operating data that they would need for the conceptual-design and economic-feasibility studies.

Ayrshire Preparation Plant

The Ayrshire Mine and Preparation Plant northeast of Evansville produced surface mined coal from the Indiana VI seam. The preparation plant was a 1,200 st/h jigging operation originally placed into service in 1973. Product quality was an important consideration at Ayrshire since low sulfur coals were being purchased at the time to blend with the plant production in order to meet customer specifications. The main focus of the near-term application was the 80 st/h of fine refuse going to the slurry pond. It was viewed as a potential source of low sulfur coal which could replace some of the coal being purchased as blending stock.

Lady Dunn Preparation Plant

The Lady Dunn Preparation Plant east of Charleston, West Virginia, received Stockton and Eagle seam coal from a nearby underground mine. The plant had a heavy-media vessel/shaking table/mechanical-cell flotation circuit and had a capacity of 550 st/h when the task began in 1992. A multiphase expansion to 1200 st/h, involving replacement of the shaking tables with heavy-media cyclones and spiral separators was on the planning board at the time. The initial evaluation indicated that the Lady Dunn plant could benefit from the use of advanced cleaning technology to treat the additional fines that would be generated by the expansion.

Wabash Preparation Plant

The Wabash Mine and Preparation Plant near Keensburg, Illinois, produced coal from an underground mine in the Illinois No. 5 seam. The preparation plant was a 1,500-st/h heavy-media vessel/heavy-media cyclone operation that had been placed into service a few months earlier. A minus 0.15-mm cyclone overflow was being discarded, but which could be cleaned by the advanced physical fine coal cleaning processes and sold.

Economic and Technical Feasibility of Proposed Applications

The plant samples were characterized at Amax R&D and successful laboratory and agglomeration tests conducted at Amax R&D, CAER and Arcanum.

Bechtel performed an engineering analysis of the economic and technical feasibility of the proposed near-term applications, that is, column flotation and selective agglomeration at Ayrshire and Lady Dunn and column flotation at Wabash using the data from the laboratory evaluations. They considered three marketing options for the clean coal to be produced by each application, namely 1) dewatered centrifuge cake blended with the existing production, 2) dry powder fuel produced from the centrifuge cake, and 3) briquettes produced from the dry powder fuel.

Capital and Processing Costs

During their analysis, Bechtel found that between 21 and 98.8 st/h of good quality clean coal would be produced by the proposed applications. This would be new production in the Ayrshire and Wabash cases. As part of the economic analysis, Bechtel estimated the cost of installing the advanced circuits in the three plant locations and projected the total processing costs for operating the circuits, including capital charges. The projected capital costs, in 1993 dollars, for installing the circuits in each plant were as follows:

	Ayrshire Plant		Lady Dunn Plant		Wabash
	Flotation	Agglomeration	Flotation	Agglomeration	Flotation
Advanced Cleaning	$3.66M	$6.80M	$1.50M	$1.60M	$9.16M
Thermal Drying	5.36M	4.22M	3.4M	1.7M	5.4M

Producing centrifuge cake for blending with current plant production was the lowest-cost option at each location. The cost of producing centrifuge cake after column flotation cleaning ranged from $5.63/st at Lady Dunn up to $8.73/st at Wabash. Column flotation was also less expensive than selective agglomeration at Ayrshire and Lady Dunn, although the difference was only $3.18/st at Lady Dunn.

Drying added between $7.36/st and $10.65/st to the total processing cost at the three locations. It was estimated that briquetting the dried coal would add between $6.17/st and $7.26/st to the processing cost. This led to total processing costs between $24.02/st and $36.06/st for briquetted clean coal on a bone-dry basis. On a heating value basis, the total processing cost, including briquetting, ranged from $0.84/MBtu for flotation clean coal at Lady Dunn on up to $1.32/MBtu for agglomeration clean coal at Ayrshire.

In view of the encouraging economic and technical assessment of the column flotation near-term applications, the project team with the strong support received from Cyprus Amax Coal Company recommended larger-scale column flotation testing at the Lady Dunn Preparation Plant.

Column Flotation Testing at Lady Dunn Preparation Plant

Pilot testing of a 30-inch diameter Microcel^{TM} column began at the Lady Dunn preparation plant in June, 1995 in order to confirm the laboratory results and to obtain additional scale-up information. The recovery of the coarser particle sizes of coal, that is, coal particles in the 0.25 to 0.75 mm range, was of particular interest during this work.

The Lady Dunn flotation feed typically contained 40 percent ash and had a high percentage of minus 325 mesh coal and clay in the slurry. Column performance was evaluated from the percentage recovery of the coal in various particle-size fractions and from the ash content of the products. The plant had existing mechanical flotation cells so the test results could be directly compared to conventional technology.

Information was gathered from preliminary testing and from two series of parametric tests. The results illustrated very well the potential for coarse coal flotation in a properly operated system. Particles up to 0.25 mm in size floated consistently well. Coarse coal up to 0.50 mm in size also floated well, but coal recovery dropped off rapidly above that size. Since it is difficult to avoid misplaced material when separating fine particle sizes, making a nominal 0.25-mm cut and sending the minus 0.25-mm fraction to a flotation column should work well in most coal processing plants. As long as the misplaced coarse material in the feed slurry is smaller than 0.50 mm, the column can provide very good recovery of coal with a low ash content.

In traditional coal processing plant applications, the particle-size cut ahead of flotation is made at a nominal 0.150 mm (100 mesh). The difficulty in making such a fine cut results in a considerable amount of fine material (usually high in ash) remaining in the coarser fraction. In all gravity separation devices designed for cleaning plus 0.150-mm material, much of this minus 0.150-mm material reports to the clean coal launder without cleaning (i.e., as high-ash coal). Thus, the difficulty in removing fines from clean coal streams results in higher ash final products. By utilizing wash water, a flotation column can remove the high ash slimes that otherwise would be entrained in the froth. Therefore, it can handle slimes better than other cleaning devices readily available to preparation plant operators, yet it can still clean plus 0.25-mm particles which do not respond well to conventional flotation.

All indications were that column flotation would perform well at the Lady Dunn Plant. The original mechanical flotation cells produced an average of 14 to 16 percent ash clean coal at a 20 percent combustible recovery. Results from testing the 30-inch diameter column, on the other hand, indicated that clean coal containing 10 to 11 percent ash can be obtained from the 0.25-mm x 0 fines at a combustible recovery of 75 percent.

The success of this test work was made tangible by the installation of three Microcel^{TM} flotation columns, each four meters in diameter, in the Lady Dunn Preparation Plant. These are the largest known flotation columns for cleaning coal. Cyprus Amax Coal Company installed the columns in the plant on the basis of the good results achieved by the test work described in this report. The new columns have been successfully cleaning 0.25-mm x 0 coal and producing results that fit on the ash/recovery curves seen during the pilot testing .

Auxiliary Operations

The pilot scale flotation investigation was supplemented with laboratory and bench-scale studies to dewater the clean coal froth from the 30-inch column and also to improve its marketability by conversion to CWF slurry fuel or briquetting to a lump fuel.

Dewatering

Clean coal slurry from the 30-inch column testing was shipped to the Federal Energy Technology Center at Pittsburgh for centrifuge dewatering tests using their GranuFlow process. Performance of screen-bowl and solid-bowl centrifuges were compared, and cakes with the following percentage moisture contents were obtained:

	No Additive	6 - 8 % Orimulsion
Screen Bowl	39.4	35.2-35.7
Solid Bowl	34.8	31.0

In addition to the centrifuge testing, 122 laboratory vacuum filtration leaf tests were conducted on froth from the 30-inch column by Westech Engineering Inc. personnel. Because of the residual clay in the Lady Dunn clean coal slurries, preflocculation was required to achieve good filtration performance. Severe filter cloth blinding occurred after a few tests so it was necessary to include a cloth washing step in the filtration cycles. Filtering coarse spiral concentrate along with the froth slurry was found to offer little advantage with respect to capacity or moisture removal. A horizontal belt filter did appear to offer a somewhat higher capacity on a lb/h/sq ft basis than a drum belt filter, but the moisture contents of the resulting cakes were about the same, that is, in the 34 to 43 percent range. Because these cake moistures were similar to the centrifuge cake moistures, Lady Dunn management decided to continue with their original plan to use a screen-bowl centrifuge for dewatering the column flotation froth after the plant expansion.

Hydrophobic Dewatering

Hydrophobic dewatering is an innovative process for dewatering fine coal that is being developed at Virginia Tech. During the process liquid butane is mixed with a coal slurry such as the froth from column flotation. The butane displaces the water from the particles so that the butane and the coal float to the surface of the slurry where they may be separated from the water phase. Evaporation of the butane from the floating solids left coal containing as little as 1 percent moisture during laboratory testing for this project. It is expected that the butane vapor would be recovered for reuse so that the process would be an economically attractive alternative to filtration and thermal drying the fine coal.

CWF Slurry Preparation

Marketing clean coal from near-term column flotation as slurry fuel rather than as filter cake or centrifuge cake was considered. Slurry preparation tests were performed on froth slurry from the Microcel™ testing at the Lady Dunn plant. It was found that at a projected viscosity of 500 cP, slurry loadings of 62, 63 and 68 percent coal could be achieved for blends containing 0, 10 and 40 percent, respectively, of the ground spiral concentrate. In each case, the slurry contained one percent A-23M dispersant on a dry coal basis.

Briquetting

A portion of the clean coal from the 30-inch column testing was submitted to TraDet Inc. for binderless briquetting tests. Good quality specimens of the briquette production were returned by TraDet, who reported that the briquetting was done at near-ambient temperature on the flotation product after it had been air-dried to between 1.0 and 2.4 percent moisture. The briquettes contained 11.8 percent ash and 34.2 percent volatile matter and had a heating value of 12,900 Btu/lb.

The crush strengths of the briquettes were between 50 and 200 lbs, and these strengths correlated well with the amount of energy transferred to the briquettes (between 8 and 29 kWh/ton) during the briquetting process. TraDet considered any strength over 100 lbs to be acceptable for briquettes such as these.

Conclusions and Recommendations for Near-Term Applications

The conceptual engineering analysis of the laboratory column flotation and selective agglomeration test results and the confirmation bench-scale and pilot testing of column flotation showed that advanced physical fine-coal cleaning processes can be advantageously integrated into existing coal preparation plants. The following observations were made regarding this work:

- Column flotation can recover a lower-ash clean coal than the usual mechanical-cell flotation and at a higher recovery of combustibles. The following example is for the Lady Dunn application:
    - Microcel™ column – 10 to 11 percent ash clean coal, 75 percent recovery

  - Mechanical cells – 14 to 16 percent ash clean coal, 20 percent recovery

- Column flotation can be effectively applied to streams containing coal as coarse as 0.5 mm and, less effectively, as coarse as 1.0 mm.
- High-pressure binderless bench-scale briquetting was effective for reconstituting the clean coal.
- Selective agglomeration performance projected from laboratory testing was similar to or somewhat better than the performance of column flotation.
- Projected near-term application costs for producing dewatered clean coal by column flotation of raw coal fines were in the \$5.60 to \$8.70 per dry short ton range.
- Projected near-term application costs for producing dewatered clean coal by selective agglomeration with a non-recoverable bridging liquid such as diesel fuel were significantly higher than the projected cost of recovering the clean coal by column flotation. Selective agglomeration was particularly less competitive when cleaning midwestern Ayrshire coal which did not agglomerate as easily as the eastern Lady Dunn coal.
- Thermal drying of the clean coal for blending with the existing plant production or for separate sale as powder fuel adds \$7.60 to \$10.60 per short ton to the production cost of the coal recovered by advanced cleaning.
- The total projected cost of producing briquetted fuel (but not including the cost of the raw coal fines) was less than \$25.10 per short ton for four of the five near-term applications evaluated.

The following recommendations are offered to operators of coal preparation plants:

- Advanced physical fine coal cleaning options should be considered for installation in new plants and when refurbishing or expanding existing plants. It is likely that additional revenue can be generated over the revenues from the “no fine coal cleaning” or the “mechanical-cell flotation” options.
- In order to reduce costs, agglomeration with recoverable bridging liquids such as heptane and pentane, should be explored as alternatives to fuel oil and diesel fuel.

Methods for improving the marketability of the recovered fine coal, such as GranuFlow processing, conversion to CWF, powder fuel, and especially binderless briquetting, should be developed further.

CONCLUDING REMARKS

This project is an important milestone on the way to the commercial production of coal-based premium fuel as a replacement for oil and gas fired in some utility and industrial boilers. Much has been accomplished by this project and much learned. Some of the more important conclusions are listed below:

- There are coals available in the United States backed by large reserves that can be finely ground and cleaned to meet the premium-fuel specifications of less than 2 lb ash per million Btu and less than 0.6 lb sulfur per million Btu.
- The advanced column flotation and selective agglomeration physical fine coal cleaning processes are capable of recovering 80 to 90 percent of the heating value in available ROM coal while producing clean coal meeting the premium-fuel ash and sulfur specifications.
- The column flotation and selective agglomeration equipment and processes are robust and reliable for producing the target yield and quality of clean coal as demonstrated by processing over 2,100 tons of coal from three different mines though the 2 t/h integrated process development unit
- If desired, the clean coal can be formulated into usable coal water slurry fuel with a heating value of 8,500 to 9,100 Btu/lb.
- Advanced physical fine coal cleaning rejects certain toxic trace elements from coal and could be part of a strategy to control hazardous air pollutant emissions from coal burning boilers.
- The production of premium fuel from coal in a Midwestern industrial area by either technology would cost less than $2.50/MBtu, in other words, the cost would be competitive with the cost of fuel oil.
- Advanced column flotation can and has been applied effectively in existing preparation plants for recovering minus 28 mesh fine coal that would otherwise be lost to refuse.

1. INTRODUCTION

The Pittsburgh Energy Technology Center (PETC), now the Federal Energy Technology Center (FETC), Pittsburgh of the U. S. Department of Energy (DOE) awarded cost-sharing contract No. DE-AC22-92PC92208 on September 30, 1992 to an Amax-led team for "Engineering Development of Advanced Physical Fine Coal Cleaning for Premium Fuel Applications." The coal cleaning methods targeted by the program were the advanced column froth flotation and selective agglomeration processes researched and developed under the DOE Acid Rain Control Initiative.

The program stressed the engineering development of processes for preparation of ultra-clean coal. The ultra-clean coal would be burned as a cost-effective premium coal water slurry fuel (CWF) to replace a portion of the oil and gas now firing utility and industrial boilers, and it could also be burned in 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 established specifications for the maximum allowable ash and sulfur levels in the fuel and the handling and combustion properties of the CWF.

PROJECT GOALS AND OBJECTIVES

The ultimate goal of the Department of Energy is to develop a coal-based fuel that will be a viable alternative for fuel oil or natural gas in future years. The current project had three major objectives which would further progress toward this long-range goal:

- The primary objective was to develop the design base for prototype commercial advanced fine coal cleaning facilities capable of producing ultra-clean coals suitable for conversion to coal-water slurry fuel for premium fuel applications. The fine coal cleaning technologies to be developed were advanced column flotation and selective agglomeration and the goal was to produce fuel meeting the following specifications --
    - Less than 2 pounds of ash per million Btu (860 grams per gigajoule) and preferably less than 1 pound of ash per million Btu (420 grams per gigajoule).
    - Less than 0.6 pound of sulfur per million Btu (258 grams per gigajoule).
    - Recovery of at least 80 percent of the heating value from the run-of-mine raw coal.
    - Production cost of the CWF to be less than $2.50 per million Btu ($2.37 per gigajoule) including the cost of the coal.
- A second objective was to develop the design base for near-term application of the advanced coal cleaning technologies in new or existing coal preparation plants to

efficiently process minus 28-mesh coal fines and convert them economically into marketable products.

- A third objective was to determine the removal of toxic trace elements from coal by the advanced column flotation and selective agglomeration technologies.

This report summarizes the work that was done to accomplish the above objectives. It also presents the important findings of the study and the conclusions drawn from the effort. Topical reports to the Department of Energy were issued upon completion of important tasks and subtasks, and these contained detailed results of the specific areas investigated during the 5-year project. Chapter 13 contains a list of these reports and also a list of public presentations describing particular aspects of the work.

PROJECT APPROACH

The overall project effort was divided into four phases and further divided into tasks and subtasks. Table 1 contains a complete list of the project tasks and subtasks.

Phase I consisted of the project planning activities (including selection of the specific coals to be studied during project) and the laboratory- and bench-scale advanced flotation and selective agglomeration process research and development studies. Specific subtasks were included to study the grinding of the coals and the liberation of the ash minerals, to select the type of bridging liquid to be used for selective agglomeration and to develop CWF formulations for the clean coals. Near-term applications of the advanced cleaning technologies were investigated during a separate task of Phase I. There were also tasks for the design of an integrated 2-st/h (2-short ton/hour or 1.8-metric ton per hour) process development unit (PDU) containing advanced flotation and selective agglomeration modules for cleaning test coals. The PDU was built and operated at Amax R&D in Golden, Colorado to produce tonnage quantities of the clean coals. It provided performance and scale-up parameters for the conceptual design of commercial facilities for production of premium fuel from coal.

Phase II was for the detailed construction and operation of the PDU with the advanced flotation module in place. Phase III was for construction of the selective agglomeration module and operation of the PDU with the selective agglomeration module in place. During both phases, PDU performance data were obtained by parametric testing on three coals followed by 72-hour production runs with each one.

Phase IV was for the dismantling and disposition of the PDU, a conceptual study of the commercial production of premium CWF when using the technologies, and for the preparation of the project final report.

The project team was headed by Cyprus Amax Minerals Company through its subsidiaries Amax Research & Development Center (Amax R&D) and Cyprus Amax Coal Company (Midwest and Cannelton Divisions). Entech Global managed the project for Amax R&D and provided research and development services in all project areas at the Amax R&D Center in Golden, Colorado. Bechtel Corporation performed the engineering analysis of potential near-term applications and designed the 2-st/h PDU operated by Entech Global at Amax R&D. Bechtel also studied the economics of commercial premium CWF production. The PDU was constructed by TIC and Mech El.

The Center for Applied Energy Research (CAER) of the University of Kentucky, and Center for Coal and Mineral Processing (CCMP) of the Virginia Polytechnic Institute and State University provided technical assistance with the advanced flotation technologies and Arcanum Corporation provided similar assistance with the selective agglomeration technology. Dr. John P. Dooher of Adelphi University and Dr. Douglas V. Keller, Jr. of Syracuse University were consultants to the project in the areas of CWF formulation and selective agglomeration, respectively.

SCHEDULE

Work on the project began on September 30, 1992 and was completed on September 30, 1997. Schedules for the various tasks and phases were overlapped in order to complete the project during this time frame.

ORGANIZATION OF REPORT

This report is organized differently from the organization task and subtask in that the cleaning technologies and coal selection/acquisitions aspects common to both technologies are described first followed by all of the advanced flotation work discussed in one chapter and all of the selective agglomeration work is discussed in another chapter. After that, the toxic trace element reduction, CWF formulation, fuel properties, commercial production, and near-term application studies are discussed in separate chapters. There are final chapters for concluding remarks and lists of reports and presentations.

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
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

2. ADVANCED FINE COAL CLEANING TECHNOLOGIES

Efficient process flowsheets have evolved for economically preparing the quality of coal most demanded by the existing market place (5 to 15 percent ash). Only on rare occasions can these flowsheets be adjusted to produce clean coal meeting the target premium fuel ash specification of less than 2 lb/MBtu (in most cases this is around 2.8 percent ash or less) since the flowsheets are designed to clean coarse coal. Some pieces of coarse coal, for instance lumps between 1 and 2 inches across, may contain thin clay partings and attachments of rock as well as other mineral matter finely disseminated through the coal. The rejection of these composite pieces of middling coal by the preparation plant would lead to a significant loss of saleable coal. Fortunately for the mine operator, though, the ash contribution from such pieces is acceptable to most buyers.

The logical way to prevent the loss of coal when trying to meet low ash specifications would be to crush and grind the coal before cleaning in order to break the lumps into separate smaller pieces of liberated mineral matter and low-ash coal. Unfortunately, the technology for cleaning this fine coal (shaking tables, spiral and other launder separators, cyclones, froth flotation, etc) has not been developed to the extent needed for efficient and economical production of low-ash fuel. Coal cleaning technology needs to be advanced in this area.

To fill this need, a number of processes were investigated during the 1972 to 1992 period by the U. S. Department of Energy and by others for physically separating impurities from fine coal. Much of this interest centered on desulfurization, but this work clearly showed that the residual amount of ash in the coal was also reduced to significantly lower levels than the levels achieved by conventional cleaning at coarse mesh sizes. Two of these processes in particular – advanced column flotation and selective agglomeration – appeared to be especially attractive and warranted further development as methods for preparing low-ash premium fuel from coal. Each of these two processes had in fact been the focus of successful proof-of-concept development projects***** co-sponsored by the Department of Energy Acid Rain Control Initiative.

ADVANCED COLUMN FLOTATION

Advanced column flotation is an improvement of the froth flotation operation often employed in coal preparation plant for cleaning fine coal slurries. It utilizes a counter-current flow of feed slurry and air bubbles to separate coal particles with hydrophobic (water-repellent) surfaces from mineral refuse particles with hydrophilic (water-wetted)

***** Southern Company Services, “Engineering Development of Selective Agglomeration,” Contract No. DE-AC22-89PC88879, Final Report, April 1993.

ICF Kaiser Engineers, “Engineering Development of Advanced Physical Fine Coal Cleaning Technologies - Froth Flotation,” Contract No. DE-AC22-88PC88881, Final Report, March 1995.

surfaces. The separation is done in a tall column rather than in a series of mechanically agitated cells. Wash water addition to the column above the feed point are a key feature of column flotation when cleaning fine coal. When added in sufficient volume, this wash water creates a net downward flow of water which flushes fine refuse from the cleaning zone between the point where the feed slurry enters the column and where the froth overflows the column.

The dispersion of air in a deep column is often thought to be better than in shallower mechanical cells and the resulting finer bubbles, or “microbubbles,” appear to collect fine coal particles more efficiently than the bubbles in mechanical cells. A number of methods for introducing air into the column were considered during the laboratory-scale testing and two methods, KenFlote™ and Microcel™, were evaluated during the bench-scale testing. Aeration was through an internal sparger in the KenFlote™ column and through an external in-line static mixer in the Microcel™ column. The Microcel™ system was employed for the PDU and the on-site near-term application testing. Further details are presented in Chapters 5, 9 and 11 of this report.

SELECTIVE AGGLOMERATION

Selective agglomeration cleaning is also based on the difference between the surface properties of coal and fine refuse particles. When an oily bridging liquid is added to the slurry, the fine hydrophobic coal particles become coated with the oil. When subjected to intense high shear-rate mixing, the oiled particles will adhere to each other in clusters or “microagglomerates.” The refuse particles, on the other hand, are not affected by the bridging liquid and remain dispersed in the liquid phase. Formation of microagglomerates takes a finite length of time, called the “phase inversion time,” and results in a visible change in the appearance of the slurry. Continued mixing under less intense low shear-rate conditions allow the microagglomerates to grow in size until they may be separated from the dispersed refuse particles by screening. Alternatively, the low-shear mixing step may be omitted or the time greatly reduced in which case the microagglomerates are recovered by froth flotation instead of by screening.

Various oily liquids may be employed for bridging the coal particles. As discussed in Chapter 6, two types were considered for this project:

1. Non-volatile oils such as diesel fuel, fuel oil and kerosene which remain with the product coal after the separation.
2. Volatile oils such as pentane, hexane and heptane which can be recovered from the product coal and reused.

As discussed in chapters 6 and 11, the premium fuel preparation tasks focused on the use of reusable bridging liquids because of their good performance and potential for lowering operating costs. On the other hand, the use of non-volatile bridging liquids was emphasized for near term applications (Chapter 11) since the technology for recovering and reusing bridging liquids had not been developed beyond the conceptual laboratory stage. Advancement of volatile bridging liquids to commercial usage was believed to be beyond the time frame for a near-term application.

3. COAL SELECTION, ACQUISITION AND PROPERTIES

Successful achievement of the ultimate goal of this program – the development of a coal-based fuel that will be a viable alternative for fuel oil or natural gas in future years – will depend upon a plentiful supply of suitable source coal for upgrading into premium fuel. Source coal selection criteria were established to ensure that suitable source coals would be utilized during the process development studies since not all United States coals are likely to be acceptable feedstock for preparation of premium fuel. The plan was to select six coals for the Phase I laboratory and bench-scale testing and after completion of Phase I, select three from among those six for the 2-st/h Phase II and Phase III PDU scale testing.

COAL SELECTION CRITERIA

The basic coal selection criteria were established by the contract Statement of Work, but these were supplemented by additional criteria developed by the project team and approved by the DOE [**R-1**]*****. The criteria may be divided into three groups according to coal property and availability/cost requirements and to process related preferences:

1. Coal Property Requirements --
    - Well under 0.6 lb/MBtu of sulfur in the organic form. Low-sulfur coals meeting this specification are plentiful in certain coal formations but not in others.
    - Ash minerals and pyrite sufficiently liberated by practical grinding methods so that the target impurity specifications may be met.
    - At least one low-rank coal to be included in the evaluation.

2. Availability and Cost Requirements –
    - From seam with large reserve, preferably exceeding 500 million short tons.
    - From an active mine capable of providing truckload quantities of coal for this project.
    - From geographically diverse locations.
    - Market value of less than $1.25/MBtu (about $30/st) in order to allow a reasonable incremental cost for the advanced cleaning and still allow total fuel production cost to be less than the goal of $2.50/Mbtu at commercial scale.

3. Process Related Preferences –
    - Lower ash content.
    - Lower total and pyritic sulfur content.
    - Ash-mineral and pyrite liberation at coarser sizes.
    - Lower inherent moisture.
    - Higher Hardgrove Grindability Index (easier to grind).
    - Higher hydrophobicity (but still wettable with water).

***** References refer to project reports (**R-**) and presentations (**P-**) listed in Chapter 13.

SELECTION PROCEDURE

An initial list of candidate coals was drawn from published accounts and past Amax R&D experience preparing low-ash coal for other projects. This list was supplemented by recommendations from DOE, CAER, Amax Coal Company and other sources. Eventually at least 32 coals from the states of Pennsylvania, Maryland, West Virginia, Virginia, Ohio, Indiana, Illinois, Kentucky, Alabama, Montana, Wyoming, Colorado, and Utah were given serious consideration. Three (from Alabama, Pennsylvania and Kentucky) were later removed from the list because of inadequate reserve bases.

In some cases, sufficient information was on hand to judge the suitability of a coal for use during the project. More often though, 5-kg samples of typical production were obtained from the candidate mine sources and tested at Amax R&D to determine their amenability to advanced cleaning. The amenability testing included washability, proximate and forms of sulfur analyses, and tests to determine the response of finely ground samples of the coals to laboratory selective agglomeration and tree flotation testing. The laboratory selective agglomeration tests were found to be a particularly useful method for quantifying the degree of ash mineral liberation achieved by fine grinding. A special procedure, which included an acidification step, was developed for the laboratory agglomeration tests on the low rank coal included in the evaluation.

Using a scoring system to weight amenability test results and the various criteria listed above, the coals were ranked according to their suitability as feed stock for production of premium fuel. A separate ranking was prepared for the four subbituminous coals on the list in order to ensure selection of one of them for the Phase I testing. The amenability testing results and ranking procedures were discussed in a project report [**R-1**] and a symposium presentation [**P-10**].

COALS FOR PHASE I LABORATORY AND BENCH-SCALE TESTING

Five bituminous coals and one subbituminous coal were selected for the Phase I laboratory and bench-scale testing using the criteria described above. With the possible exception of the subbituminous coal selection, each one was a good prospect for production of premium fuel during the Phase II and Phase III PDU operation since they responded well during the amenability testing portion of the selection process. The selected coals were as follows:

- <u>Taggart Seam, Wentz Mine, Virginia (2.0% ash, 0.6% sulfur, 3.0% moisture).</u> The Taggart coal, mined and washed by the Westmoreland Coal Company for steam coal and specialty markets, was of hvA bituminous rank. It was the highest scoring coal seen during the selection process and responded very well to the advanced cleaning amenability testing. Upper Elkhorn No. 3 is another name for the Taggart seam in Virginia.

- Sunnyside Seam, Sunnyside Mine, Utah (4.8% ash, 0.6% sulfur, 6.5% moisture). The Sunnyside coal, mined and washed by the Sunnyside Coal Company for metallurgical markets, also was of hvA bituminous rank. It scored very high during the selection process and responded well to the advanced cleaning amenability testing.
- Elkhorn No. 3 Seam, Chapperal Mine, Kentucky (5.6% ash, 0.8% sulfur, 7.0% moisture). The Elkhorn No. 3 hvA bituminous washed coal mined by the Costain Coal Company was representative of the very large production of steam coal from eastern Kentucky and scored well in the selection process.
- Winifrede Seam, Sandlick Mine, West Virginia (7.9% ash, 0.9% sulfur, 6.4% moisture). The Winifrede hvA bituminous washed coal mined by the Cannelton Coal Company was typical of coals produced on a large scale in West Virginia. It required very fine grinding for liberation of the ash minerals so it did not score as well during the selection process as the previous three coals.
- Indiana VII Seam, Minnehaha Mine, Indiana (7.5% ash, 0.4% sulfur, 18.9% moisture). This Indiana VII washed hvC bituminous steam coal contained less sulfur than most Midwestern coals and scored better than other coals from the region. It did require fine grinding for liberation of the ash minerals and did not respond as well to the amenability testing so it did not score as high during the selection process as the first three coals that were selected. The mine was owned by Amax Coal Company at the time, but it has since been sold to the Kindill Coal Company and renamed the Kindill No. 3 Mine.
- Dietz Seam, Spring Creek Mine, Montana (3.9% ash, 0.3% sulfur, 21.5% moisture). The Dietz subbituminous B coal was mined by a subsidiary of Nerco (now Kennecott Coal Company) for direct sale to utility customers. It responded better to the laboratory advanced cleaning amenability tests than the other three low-rank coals that were evaluated.

The selected test coals exhibited a diverse range of attributes. Substantial production is represented from six states in the eastern, central and western parts of the United States. The bituminous coals ranged in rank from high volatile C to high volatile A rank and had ash-free inherent moisture contents between 1.75 and 15.6 percent. The low-rank selection was representative of the very important Powder River Basin area. Some (such as the Taggart coal) were expected to grade up to premium fuel quality without difficulty, while others (such as the Winifrede, Indiana VII and Dietz) were expected to challenge the advanced cleaning technologies.

Two samples of No. 2 Gas seam coals from West Virginia also ranked quite high during the selection process and were alternative recommendations. An effort was also made to identify high pyritic-sulfur low organic-sulfur coals which might be amenable to the advanced cleaning. One of the suggested candidates in this category was found to contain more organic sulfur than allowed by the premium fuel specification, and another was of medium volatile rank and would have been extremely hydrophobic and difficult to work with in a flotation or selective agglomeration system. The pyritic sulfur in the third coal (from the Ohio No. 7 seam) was found to be largely rejected by conventional washing plant cleaning. The resulting washed coal was similar to the Winifrede coal with respect to its response to advanced cleaning.

Twenty-four tons of each selected test coal were purchased directly from the source and trucked to Ralston Development Company near Golden, Colorado. The truck lots were crushed to minus 1/2 inch, sampled and stored in bulk bags for transfer as needed to Amax R&D during Phase I. Several purchases of the Indiana VII coal were made because the flotation response of the coal seemed to deteriorate during storage. The properties of these coals are presented in Table A-1 in the Appendix. The sulfur contents of the six test coals met or nearly met compliance specifications, and all contained less than 0.6 lb of organic sulfur per million Btu. One-ton lots of the ROM (run-of-mine) raw coals from these mines were also obtained, sampled and analyzed in order to calculate the Btu recovery and toxic trace element rejection accomplished at the washing plant when preparing the coals for market. Analyses of the ROM coals are presented in Appendix Table A-2 along with calculated washing plant recoveries.

COALS FOR PHASE II AND PHASE III PDU OPERATIONS

As described later in Chapters 4, 5, 6 and 8 of this report, the Taggart and Sunnyside coals responded well to the laboratory and bench-scale advanced cleaning and CWF preparation technologies. Clean coals were produced by both column flotation and by selective agglomeration that met the target ash, sulfur and Btu recovery specifications for premium fuel after reasonable amounts of grinding, and the clean coals could be converted into useful CWF. The Indiana VII coal required finer grinding for liberation of the impurities so it did not respond quite as well to the advanced cleaning as did the Taggart and Sunnyside coals, but the clean coals prepared from the Indiana VII still appeared to meet project goals.

The other three coals did not respond as well to the advanced cleaning and were not considered further for testing in the PDU. The ash minerals in the Elkhorn No. 3 coal were liberated at a relatively coarse grind but finer grinding was needed for liberation of the pyritic sulfur. Despite the finer grinding, some of the time the observed sulfur reductions failed to meet expectations for the Elkhorn No. 3 coal. Neither the Winifrede nor the Dietz coal responded satisfactorily to froth flotation. Heating value, ash and sulfur specifications were met by bench-scale selective agglomeration of the Winifrede coal, but it was clear that production costs would exceed the project goal of $2.50/MBtu because of the low unit capacities of the cleaning equipment and fine grinding needed to meet product quality specifications. The same situation applied to the subbituminous Dietz coal because of the added cost of the acid treatment needed before agglomeration.

It was found that the Wentz Mine and the Sunnyside Mine had closed when the time came to order the coals for the 2 st/h Phase II and Phase III PDU operations. A source for Taggart coal was quickly found from the Steer Branch Mine operated by the Red River Mining Company unit of Humphreys Enterprises. The Steer Branch Mine was located near the Wentz Mine, and Humphreys had taken over the accounts for Taggart coal previously served by Westmoreland. The Steer Branch coal contained slightly more ash, at 3.3 percent, than the Wentz Taggart coal.

Finding a substitute for the Sunnyside coal was more difficult since companies mining Sunnyside coal in the same area as the Sunnyside Mine blended their production with production from other seams and could not provide car-load quantities of the unblended coal. For this reason similar coals from other seams in Utah and Colorado were examined for their response to advanced cleaning. Hiawatha coal from the Crandall Canyon Mine of the Genwal Coal Company was found to closely resemble the original Sunnyside coal. The most noticeable difference between the two coals was the amount of ash in the coal. The Hiawatha coal was not washed before sale since it was marketed as steam coal rather than as metallurgical coal. For this reason, it contained about 9 percent ash instead of the 5 percent ash found in the Sunnyside coal.

Eight rail cars, or about 800 tons, of the Taggart coal were purchased and delivered to a coal yard in Denver for unloading and storage. Four rail cars of Hiawatha and four cars of Indiana VII were similarly purchased and stored in Denver. These coals were subsequently trucked to Ralston Development Company as needed for crushing and transfer to Amax R&D for the PDU operation. Subsequent purchases of Hiawatha and Indiana VII coals were trucked directly to Ralston. Typical properties of the three PDU coals are presented in Table 2. Additional information is provided in Appendix Table A-3. About 2,400 tons of coal were purchased for the PDU of which 2,100 tons were processed in the PDU. Additional Hiawatha and Indiana VII coal had been purchased because the original purchases appeared to have deteriorated during the storage period between operation of the advanced flotation and the selective agglomeration modules. The unused coal was trucked to Public Service Company of Colorado and to Western Aggregates for use as a fuel. Further details of the selection and purchase of the coals for Phase II and Phase III are provided in the topical report for Subtask 8.1 [**R-19**].

Table 2. Properties of PDU Coals

	Taggart		Indiana VII		Hiawatha	
	As-Received	Bone Dry	As-Received	Bone Dry	As-Received	Bone Dry
Proximate, %:						
Ash	3.30	3.50	7.94	9.55	7.75	8.20
Volatile Matter	32.13	34.12	27.36	32.92	40.02	42.35
Fixed Carbon	58.73	62.38	47.81	57.53	46.72	49.45
Moisture	5.84		16.89		5.51	
Sulfur, %:						
Total	0.61	0.65	0.42	0.51	0.49	0.52
Pyrite	0.05	0.05	0.12	0.15	0.07	0.07
Sulfate	< 0.01	< 0.01	0.01	0.01	< 0.01	< 0.01
Heating Value, Btu/lb	13,874	14,735	10,828	13,028	12,725	13,647
Equilibrium Moisture, %	2.6		14.5		4.3	
Density, kg/m^3		1,260		1,360		1,275
Hardgrove Grindability Index	49		54		44	
Coal Rank	hvA		hvC		hvA	
Preparation Plant Btu Recovery, %		84.9		90.5		100.0

4. GRINDING/LIBERATION OF TEST COALS

Prior experience has shown that liberation of attached mineral grains from the individual coal particles is a key consideration when cleaning coal by physical processes such as froth flotation or selective agglomeration which depend upon a difference between the surface properties of pure coal and pure mineral matter. A nearly complete difference in surface properties is important because composite mineral particles with relatively small surface exposures of carbonaceous material will adhere to air bubbles and overflow the flotation machine along with the mineral-free coal particles. The same situation occurs during selective agglomeration where small attachments of coal will allow composite mineral particles to be included in the pellets of agglomerated coal.

Liberation is ordinarily achieved by grinding until the composite particles of coal and mineral matter are broken into smaller individual particles composed either of nearly pure coal or of nearly pure mineral matter. In many cases, grinding to minus 45 µm (325 mesh) and finer is required in order to achieve the needed degree of liberation.

Because of the importance and the high cost of grinding, considerable attention was given to the subject during the project. In particular, various grinding circuit configurations were evaluated during the Phase I bench-scale studies, and the liberation characteristics for each test coal was quantified so that the required grind size could be identified that would provide clean coal meeting the target premium fuel ash specification during operation of the 2-st/h PDU.

Later on, during Phases II and III, additional grinding equipment testing was done at vendor locations before final selection of the PDU grinding mills. The PDU grinding circuit is described in the last half of this chapter and the grinding performance and operating characteristics presented for each of the PDU coals.

BENCH SCALE GRINDING STUDIES

The results of the bench-scale grinding studies were presented in two topical reports, one related to advanced flotation cleaning [**R-6**] and the other related to selective agglomeration cleaning [**R-13**]. The effects on performance of varying the configuration of the grinding circuit, the diameter of the grinding media in the grinding mills, and the PSD of the crushed feed coal were investigated. The liberation of the ash and sulfur minerals versus PSD of the ground coal were quantified by various methods and scale-up parameters derived for design of the PDU.

Configuration of Grinding Circuit

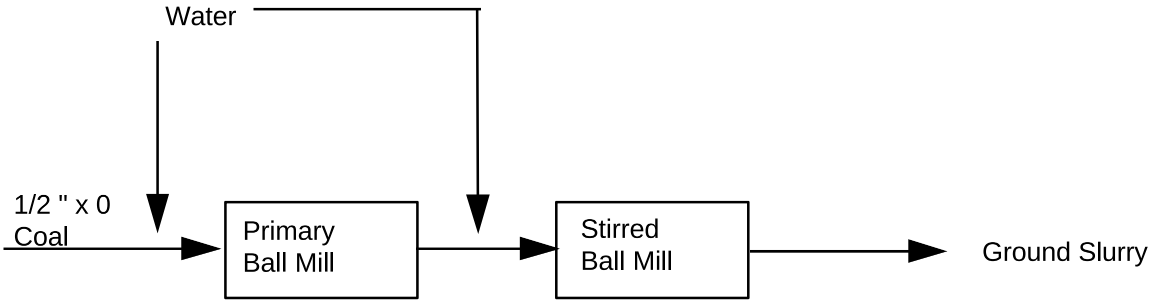
Wet grinding systems were investigated for preparation of feed slurry for the advanced cleaning since dry grinding was thought to have an adverse effect upon surface based separations. Industrially, most wet grinding is done either with tumbling media or with stirred media mills, and such technology was adopted for this project. Since it was expected that very fine grinding would be necessary for certain of the test coals, two-stage systems were specifically examined.

Open, closed and selective grinding configurations were evaluated during the bench-scale testing. Flowsheets for these configurations are presented in Figure 1. As shown in Figure 1a, the ground slurry from the first stage of open-circuit grinding flowed directly to the second stage and from there directly to advanced cleaning. This was a simple system since there was no recycle stream of oversize material to consider, and it had been utilized in previous DOE-sponsored projects. There was a recycle stream of oversize material during closed-circuit grinding, as shown for the finish grinding step in Figure 1b. Closed-circuit grinding was considered to be more efficient than open-circuit grinding provided one had an efficient particle size separation device for the classification step. The selective grinding alternative (Figure 1c) included a gravity separation device to reject high-pyrite particles from the circulating load of oversize material. Grinding efficiency would improve since less material was ground to the finished particle size. Several other variations of the basic flowsheets shown in Figure 1 were also investigated during the bench-scale testing.

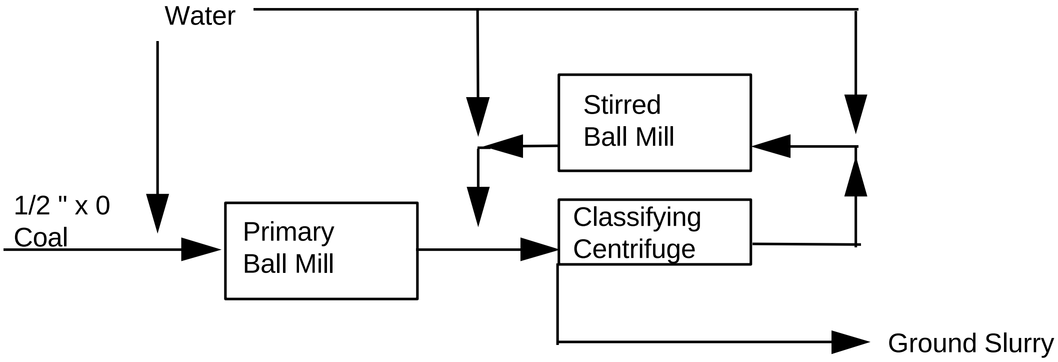
For the most part, available equipment at Amax R&D was utilized for the grinding investigations. The key pieces were a 4-ft x 4-ft dia Marcy overflow ball mill, a 40-liter Draiswerke stirred ball mill, a 48-inch Sweco screen, and a rented 7-inch diameter solid-bowl Bird classifying centrifuge. Generally the capacity of the ball mill did not match the capacity of the fine grinding mill. Thus, the two stages of grinding were not operated together. Instead, the slurry from the primary grinding step was stored and metered to the fine grinding mill as needed rather than at the rate produced. Slurries were transferred with variable speed diaphragm and progressive-cavity pumps.

Open-Circuit Primary and Fine Grinding

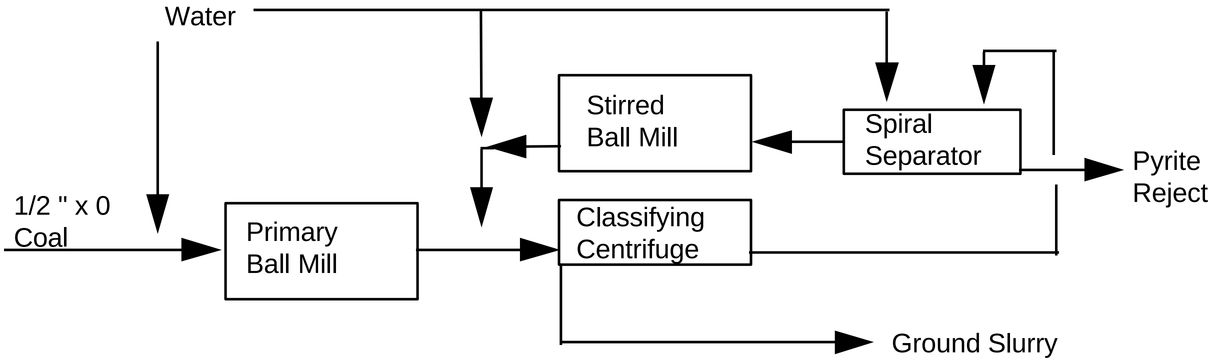
Open-circuit configurations were investigated first in order to provide a baseline for evaluating the improvement in performance when using the other configurations. These tests also provided slurry for evaluation of mineral-matter liberation from the test coals. Feed rate was the only operating variable investigated during these tests. The solids loading of the slurry was adjusted to be as high as possible while still providing sufficient fluidity for proper flow through the mills. The principal measured responses were the PSD of the ground slurries and the results of the liberation testing. Particle size distributions were determined by sieve and SediGraph analysis.



a. Open-Circuit Series Fine Grinding



b. Closed-Circuit Fine Grinding



c. Selective Fine Grinding

Figure 1. Continuous Bench-Scale Fine Grinding Circuits

Between two and nine primary grinding tests were made in the 4-foot ball mill on each of the six test coals. Grinding performance is plotted in Figure 2. The objective of these tests was to grind minus 1/2-inch crushed coal to minus 48 or 35 mesh (minus 0.3 to 0.42 mm) in order to provide suitable feed slurry to a fine grinding mill. Such slurry has an 80 percent passing particle size (D80) in the neighborhood of 120 to 150 µm. Results of the primary grinding tests which best met this criterion were as follows:

Test	Coal	HGI	Feed Rate lb/h dry coal	D80, µm	Energy kWh/st
B2-1	Indiana VII	55	574	130	47.0
B5-1	Sunnyside	54	480	124	56.3
B4-1	Taggart	52	431	119	62.6
B3-4	Winifrede	47	411	112	65.6
B6-1	Elkhorn No. 3	46	374	121	72.2
B15-1	Dietz	41	431	149	62.6

The power draft of the 4-ft mill was estimated to be 13.5 kW (18 hp) for calculation of the energy usages listed above. It appeared that the Hardgrove grindability index (HGI) is a good indication of the probable capacity of the primary grinding mill. Three additional tests were made where the coal was crushed to minus 1/4 inch rather than to minus 1/2 inch, but very little improvement was seen in the performance of the mill.

The initial second stage fine grinding tests in the 40-liter stirred ball mill were made with 6-mm diameter chromium steel balls. Results were disappointing until 4-mm balls were substituted for the 6-mm balls. The results of subsequent tests using the 4-mm balls are plotted in Figure 3. The plots are reasonably linear so one may interpolate the results in order to project grinding performance when producing slurry with differing D80 particle size distributions. The interpolated performances for producing minus 325 mesh (D80=20-24 µm) slurry from the primary grinding mill product were as follows:

Coal	HGI	Feed Rate lb/h dry coal	Energy kWh/st
Indiana VII	55	> 660	< 103
Sunnyside	54	660	103
Taggart	52	590	114
Winifrede	47	550	124
Elkhorn No. 3	46	480	142
Dietz	41	310	219

Again, grinding performance was related to the HGI of the coal. The power draft of the 40-liter stirred ball mill was estimated to be 34 kW (45 hp) for calculation of the energy usages listed above.

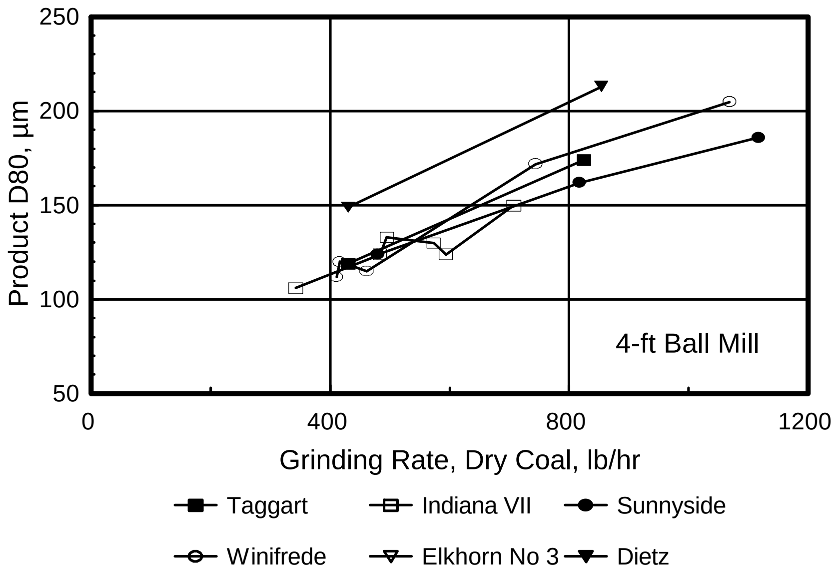


Figure 2. Grinding Performance of 4-ft Ball Mill

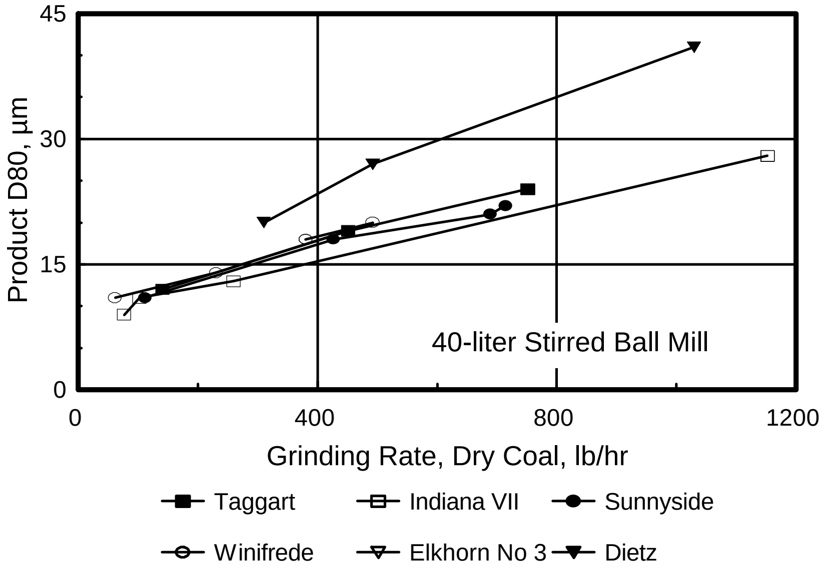


Figure 3. Grinding Performance of 40-liter Stirred Ball Mill

Concurrent liberation studies, described later in this chapter, showed that the Taggart, Elkhorn No. 3 and Sunnyside coals did not require exceptionally fine grinding before cleaning. In fact, the optimum PSD appeared to be in the range where ordinary ball milling would suffice. Consequently, additional fine grinding tests were performed using the 4-foot ball mill in place of the stirred ball mill. The results are discussed in connection with the closed circuit testing described next.

Closed-Circuit Fine Grinding

Bench-scale closed-circuit fine grinding tests were made on the Winifrede, Indiana VII and Elkhorn No. 3 coals. These tests were all two-stage configurations as shown in Figure 1b. Based upon the good classification results seen by Bechtel while performing a prior DOE project (Contract DE-AC22-87PC79867), a 7-inch diameter Bird solid-bowl centrifuge was used as the fine coal classifier during the initial closed-circuit grinding. A variable frequency speed control system was added to slow the centrifuge down and the sheaves were changed to increase the scroll speed differential and cake raking capacity so that the centrifuge would make the desired 325 mesh particle size split and perform as a classifier in the grinding circuit. A 48-inch diameter Sweco screen was substituted for the centrifuge during later testing when it was not necessary to produce a minus 325 mesh product.

Figure 4 compares the performance of the open-circuit and closed-circuit systems for grinding Winifrede coal. In this case the grinding capacity of the mill almost doubled when operated in closed-circuit with the centrifuge classifier. The improvement was much smaller when grinding Indiana VII and Elkhorn No. 3 coals. The difference may have been due to the intentionally coarser product produced while grinding the latter coals and perhaps also due to the higher grindability of the Indiana VII coal. Changing to closed-circuit grinding had little impact upon the PSD of matching products except for a reduction in the amount of tramp oversize in the finished slurry.

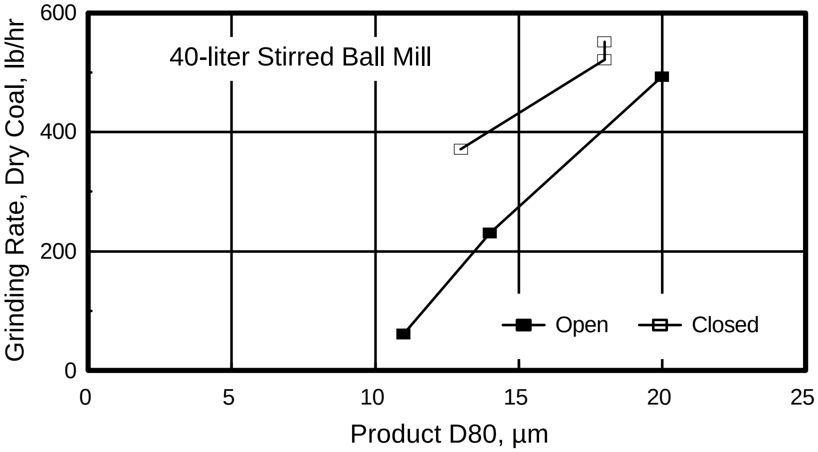


Figure 4. Open and Closed-Circuit Grinding Comparison of Winifrede Coal

During other testing, the 4-foot ball mill was used as the fine grinding mill in place of the stirred ball mill. It was effective for closed-circuit fine grinding down to minus 200 mesh when 1/2-inch to 1-inch diameter balls were used in place of the 1-inch to 2-inch balls used in the mill for primary grinding. The benefits of closed-circuit operation (with 62- and 100-mesh Sweco screens) during primary grinding to D80=45 µm were also demonstrated. Grinding rates almost doubled in the case of the Indiana VII coal.

Selective Grinding

Preliminary sampling indicated an accumulation of ash and pyrite in the circulating load of centrifuge cake when closed-circuit grinding the Winifrede and Indiana VII coals:

	Winifrede Coal		Indiana VII Coal	
Averages	Ash, %	S(py), %	Ash, %	S(py), %
Feed Slurry	8.04	0.15	9.18	0.13
Circulating Load	10.66	0.28	11.50	0.34

A 1/5th-scale Humphreys spiral (5 turns at 12 inches in diameter) was used as a gravity separator while grinding minus 48-mesh Indiana VII coal to minus 325 mesh with the selective grinding shown in Figure 1c. About 2.9 percent of the feed coal was rejected as a gravity concentrate containing 17.83 percent ash and 1.03 percent pyrite sulfur. This amounted to 29 percent of the pyrite in the feed coal but the effect was only to reduce the total sulfur down to 0.40 percent from the 0.43 percent found in the coal originally. The rejection of such a small weight of feed as a separate product did not impact grinding performance to any significant extent.

Laboratory selective agglomeration comparisons were made on the products from the three grinding circuit configurations:

	Indiana VII Clean Coal		
Circuit	Ash, %	S(t), %	S(py), %
Open Circuit	3.04	0.45	0.103
Closed Circuit	2.72	0.47	0.092
Selective	2.61	0.44	0.037

It was concluded from this work that, though a selective grinding circuit could very well improve the quality of the clean coal produced by advanced cleaning technologies, the improvement would be very small when working with coals from which most of the pyrite had been removed by a washing plant ahead of the advanced cleaning.

Effect of Slurry Loading and Dispersant Additions

The allowable loading of the slurry in the grinding mills was limited by the viscosity of the slurry. Thus, the solids loadings of bituminous coal slurries in the primary grinding mill were generally in the 38 to 44 percent dry coal range and in the 35 to 38 percent dry coal range in the fine grinding mill. The Dietz subbituminous coal loadings were significantly less because of the amount of water which soaked into the coal structure.

All of the fine grinding slurries were extremely pseudoplastic, that is, had very high resistance to flow at low shear rates.

Operation at the lower percent solids called for by the pseudoplasticity had a negative impact upon grinding efficiency. During previous operation of the stirred ball mill at Amax R&D, this was overcome by the use of A-23M dispersant during grinding. A-23M is the dispersant ordinarily used when preparing coal-water slurry fuel. Unfortunately, A-23M virtually destroys the response of fine coal to flotation and to selective agglomeration. Further tests were made with inorganic dispersants which might not have such a deleterious effect upon flotation or agglomeration. None was found. One percent sodium hexametaphosphate, for example, did not have a serious effect upon the cleaning, but neither did it have a beneficial effect upon slurry viscosity. As a result, the mills were operated at the low percent solids level.

Liberation of Ash and Sulfur Minerals

The initial assessment of mineral-matter liberation achieved by grinding the test coals was obtained by washability (heavy-liquid) tests on the crushed coals and on the coal in the slurries from the open-circuit grinding tests. Later, selective agglomeration and tree-flotation release analysis tests were also performed on the slurries. Additional liberation tests were also made on slurries produced during selected closed circuit grinding runs. Details are provided in the Subtask 4.1 and 6.2 Topical Reports [**R-6**, **R-13**].

Washability Testing

The results of washability tests on the minus 1/2-inch and minus 6-mesh crushed coals and on the minus 48-mesh, minus 325-mesh and minus 20-µm ground coals are presented in the Subtask 4.1 Topical Report [**R-6**]. If the 1.6-specific gravity separation commonly seen during conventional washing is accepted for advanced cleaning, only the float coal from the Taggart seam would have met the premium fuel ash specification. However, fine coal washability tests such as these were not found to be the best method for assessing liberation of very fine coal on a laboratory scale. As described in the next section, selective agglomeration tests showed much better liberation of mineral matter than the washability tests and were much more convenient to accomplish.

Agglomeration Testing

Heptane containing 1/4 percent 2-ethylhexanol was utilized as the bridging liquid for the agglomeration testing, and the agglomerates were recovered by washing on a 48-mesh sieve [**R-13]**. The reagents were easily stripped from the products by evaporation in a warm oven. The 2-ethylhexanol was added to lower the surface tension of the heptane so that it would wet the surface of the coal particles more effectively and activate agglomeration sooner. All of the coals agglomerated within 1 minute except for the Indiana VII and Dietz coals which required between 5 and 15 minutes. The subbituminous Dietz coal also required acidification of the slurry before agglomeration and replacement of the 2-ethylhexanol with 2 percent asphalt in order to activate agglomeration.

The residual ash contents of the six coals after agglomeration were plotted versus the D80 grind size of samples as in Figure 5. Heating value recoveries exceeded 96 percent during these tests, and the rejects ordinarily contained more than 70 percent ash.

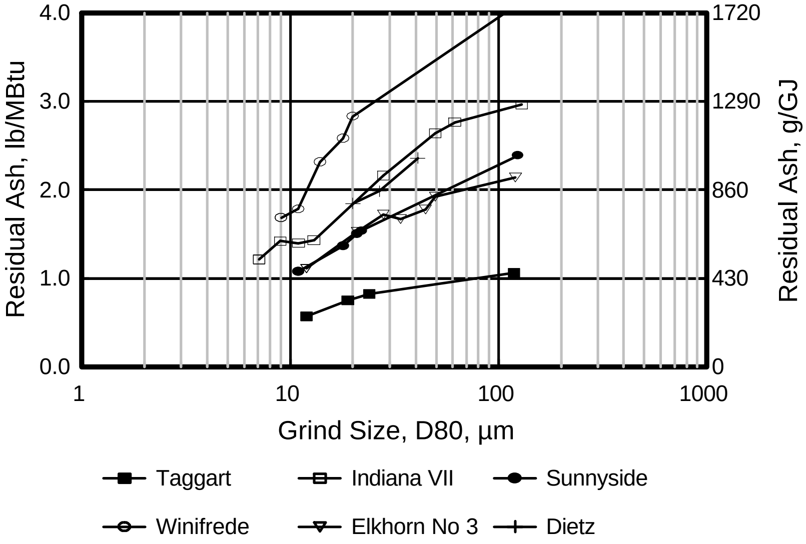


Figure 5. Ash-Mineral Liberation of Six Test Coals by Open-Circuit Grinding

The plots shown in Figure 5 were reasonably linear so one can interpolate between the residual ash values in order to estimate the D80 grind size that will allow the clean coals to meet the premium fuel ash and sulfur specifications within a comfortable margin for error. The interpolated values for each coal were as follows:

	Residual Amount, lb/MBtu		Grind Size
	Ash	Sulfur	D80, µm
Taggart	< 1.0	< 0.4	45
Sunnyside	< 2.0	< 0.5	45
Elkhorn No. 3	< 2.0	< 0.6	45
Indiana VII	< 2.0	< 0.4	20
Winifrede	< 2.0	< 0.6	11
Dietz	< 2.0	< 0.3	20

Additional liberation tests were performed to determine whether closed-circuit grinding had any impact upon the liberation of ash and sulfur minerals. Figure 6 shows the plotted residual ash versus grind data for Indiana VII coal. Similar plots are available for the sulfur distributions and for the Elkhorn No. 3 and Winifrede coals [**R-6**, **R-13**]. With the possible exception of the very finely ground Winifrede coal, all of these plots

Indicate that mineral liberation is practically the same whether the coals were ground in an open-circuit or in a closed-circuit system.

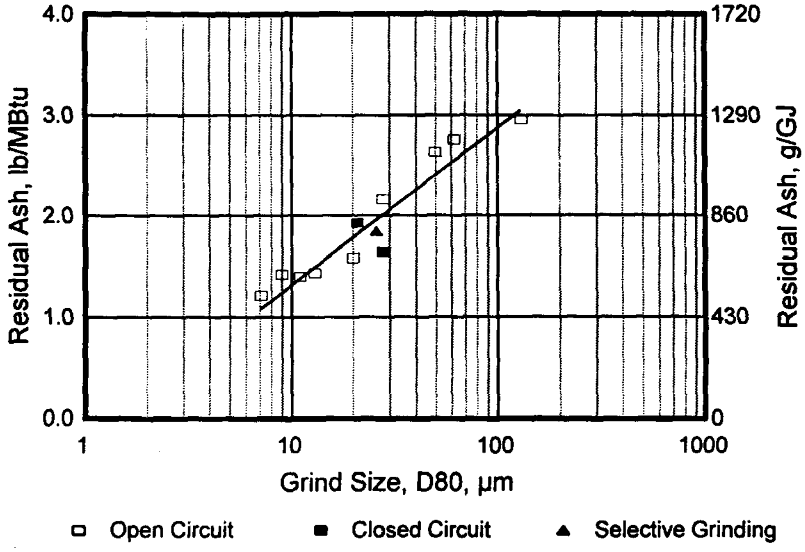


Figure 6. Ash-Mineral Liberation of Indiana VII Coal by Open- and Closed-Circuit Grinding

Froth Flotation Release Analysis

Denver cell laboratory tree flotation tests were also performed to analyze the release of liberated and locked mineral matter in the ground coals **[R-6]**. Instead of the single result of an agglomeration test, tree flotation provided a spectrum of the residual ash versus recovery response of each coal. Such data were useful for assessing the number of composite middling particles in the ground samples that might be rejected by adjustment of operating parameters during flotation. Generally, though, the tree flotation procedure was found to be less convenient to use for liberation studies than selective agglomeration, and the results were subject to more scatter due to small variations in the test procedure. For the most part the tree flotation results agreed with the selective agglomeration results except that less ash was rejected from the Indiana VII coal by flotation and the Btu recovery from the finely ground Winifrede coal was poor.

Grinding Parameter Recommendations for PDU

Two-stage grinding (open-circuit primary grinding and closed-circuit fine grinding) was recommended for the PDU. A stirred ball mill was preferred for the second stage and was required for coals such as Winifrede which needed very fine grinding for liberation. Single-stage closed-circuit ball milling was an option for coals such Taggart, Sunnyside and Elkhorn No. 3.

The centrifuge was an effective classifier for making fine particle size splits but would not be needed in the PDU unless coals such as Winifrede and possibly Indiana VII were being cleaned. There was little incentive to apply the selective grinding technology to any of the test coals because of their low pyrite content.

Grinding circuit energy consumptions were projected from the bench-scale results for use when planning the design and operation of the PDU. The projections were as follows:

	HGI	Target Grind D80, µm	Primary Grinding kWh/st	Fine Grinding kWh/st	Total Energy kWh/st
Taggart	52	45	87		87
Sunnyside	54	45	83		83
Elkhorn No. 3	46	45	107		107
Indiana VII	55	20	27	110	137
Winifrede	47	11	29	280	309
Dietz	41	20	82	103	185

The above energy projections are on a bone-dry basis.

PDU GRINDING CIRCUIT

Additional grinding tests were conducted at vendor facilities before final selection of grinding equipment for the PDU. The first of these tests was at Svedala Industries, York PA and subsequent tests were at Union Process, Akron OH and Netzsch, Exton PA. All of these tests were with stirred ball mills for the fine grinding stage. A Vertimill™ and a Sala agitated media (SAM) mill were tested at Svedala [**7th Quarterly Report**], an Attritor mill at Union Process [**8th Quarterly Report**] and a Netzsch bead mill at Netzsch [**9th and 10th Quarterly Reports**].

Svedala looked at Indiana VII and Winifrede coals. During their work, crushed coal was first ground in a ball mill and the slurry stored for grinding later in either the Vertimill™ or the SAM mill. The Vertimill™, loaded with 3/4-inch balls, was capable of producing minus 200 mesh Indiana VII coal but was not effective for producing minus 20-µm slurry from Winifrede coal. However, 20-µm slurry could be produced with the SAM mill loaded with worn 8-mm x 8-mm slugs. The energy consumptions that Svedala projected from their grinding tests generally agreed with the numbers projected from the Amax R&D testing. However, the Vertimill™ that Svedala recommended for the PDU was a very large and expensive piece of equipment and would not have fit well in the available space in the PDU building. The alternative was to install 4 SAM mills, each with a separate feed system.

Union Process tested minus 48-mesh preground Indiana VII coal in a continuous flow Attritor mill with 1/4-inch steel balls. Performance was similar to that seen at Amax R&D when grinding Indiana VII coal in the closed-circuit 40-liter stirred ball mill system.

The mill recommended by Union Process for service in the PDU was also very large and expensive.

The Netzsch tests were on 48x270-mesh Indiana VII coal screened from slurry that had been ground in a ball mill at Amax R&D to simulate the circulating load from a closed-circuit grinding system. The tests were at various feed rates to a 22.7-liter stirred mill charged with either cast steel, chromium steel or glass 3-mm dia media in a confined chamber. Acceptable results were achieved with all three types of media. Net grinding energy consumptions were in the 40 to 48 kWh/st range when producing a 98+ percent minus 325 mesh product. A Netzsch mill was selected for the PDU based upon its efficient grinding, compact size and lower overall installation cost.

PDU Grinding Performance

The PDU grinding circuit was based on two 5-ft dia by 10-ft ball mills that were available from the Southern Services Company project.[*] As described in Chapter 5, the two ball mills in series were supplemented with a Netzsch stirred ball mill (bead mill) for the final fine grinding. Screen- and cyclone-classification provisions were included for particle-size control and for closed-circuit operation as recommended by the bench-scale testing [**R-6**, **R-13**].

Grinding circuit flowsheet development and optimization continued with each of the PDU coals during operation of the Flotation Module [**R-21**]. For example, 25 tests aimed at optimizing the circuit were conducted while grinding the Steer Branch Taggart coal. Specifically, the effects of feed rate, ball loading in the mills, cyclone size, screen opening size, and circuit type (open or closed) were evaluated.

The fine liberation requirements of the Indiana VII coal led to 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 D80 values, and unexpected downtime. Several changes were made which improved the operation:

- **Reduce Mill Speed** - The speed of each of the ball mills was reduced to 30 rpm, or 65 percent critical, from the original 35 rpm, or 80 percent critical.
- **Reduce Ball Size** - The top-size of the balls in the primary mill was reduced from 3 inches to 2 inches while the top-size in the secondary mill was reduced from 2-1/2 inches to 1-1/2 inches.
- **Reduce Ball Charge** - The weight of balls in each mill was too high for the smaller amount of Indiana VII coal being ground so the loading was reduced to 8,100 pounds in each.
- **Increase Solids Concentration** - Judicious control of the water additions to the mills allowed the operation of each mill on slurry at over 38 percent

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[*] Southern Company Services, “Engineering Development of Selective Agglomeration,” Contract No. DE-AC22-89PC88879, Final Report, April 1993.

solids, matching more closely the solids concentrations utilized during the bench-scale grinding.

The operation of the grinding circuit had been pretty well tuned by the time the selective agglomeration module was in operation. The grinding performance from the production runs during those tests [**R-23**] provided useful data for projecting performance of commercial coal grinding operations. Different circuit configurations were used for each coal during the production runs:

- **Taggart** - 2-inch cyclones and 150-mesh screens, recycle cyclone underflow and screen overflow to Netzsch mill
- **Hiawatha** - 2-inch cyclones and 70-mesh screens, recycle cyclone underflow and screen overflow to Netzsch mill
- **Indiana VII** - 3-inch cyclones and 100-mesh screens, recycle cyclone underflow and screen overflow to second stage ball mill

Table 3 provides a comparison of the grinding performances for the production runs on each coal. The energy consumptions listed in the table were based on kilowatt meter readings for the ball mills and an ammeter reading for the Netzsch mill. The estimated energy consumptions were comparable to the bench-scale projections for the Taggart and Hiawatha coals but were considerably less for the Indiana VII coal. Part of the difference was because the Indiana VII coal was ground to only D80=67 µm during the PDU extended production run instead to D80=20 µm as during the bench-scale work. More importantly though, it appears that the stirred ball mill grinding was not operated very efficiently in either the bench-scale circuits or in the PDU circuit.

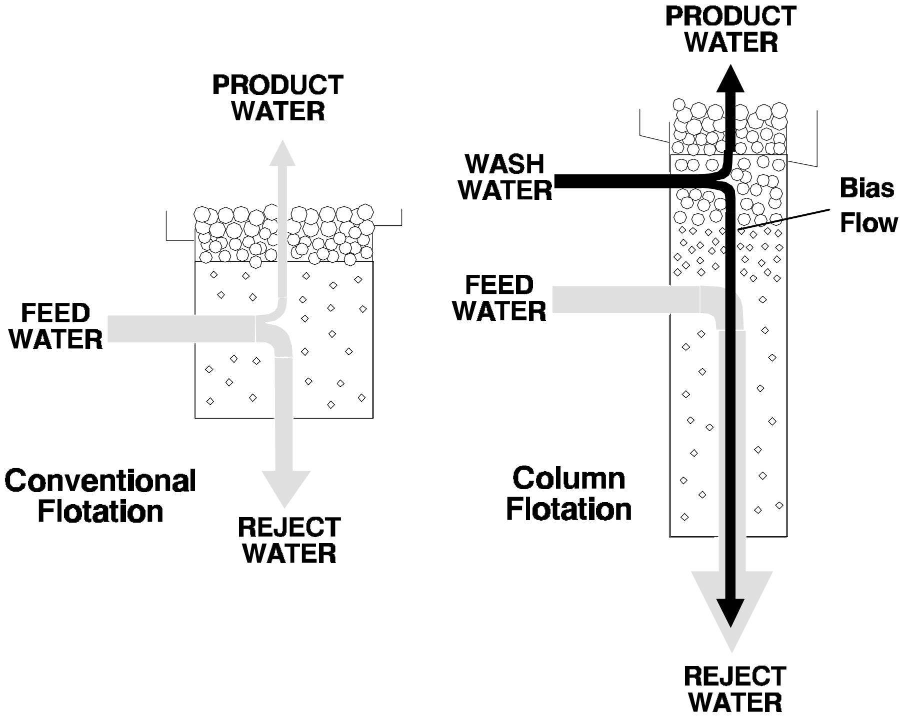
Data were also obtained during the PDU production runs regarding the efficiency of the cyclone classifiers. Partition curves for the Taggart, Hiawatha and Indiana VII production runs are shown in Figure A-1 in the Appendix. In the Taggart and Hiawatha cases the D50 separation (50/50 split to overflow and underflow) was between 40 and 50 µm, and in the Indiana VII case the D50 separation was around 150 µm. The partition curves also show considerable retention of fines in the cyclone underflow which meant that finished material was unnecessarily recycled to the grinding mills. Ordinarily this would be overcome by adding more dilution water to the cyclone feed slurry, but that was not an option here because of the amount of water it would have added to the flotation and selective agglomeration circuits.

Table 3. PDU Grinding Performance

	Taggart	Hiawatha	Indiana VII
Crushed Coal:			
Feed Rate, dry lb/h	3,305	3,839	3,491
D80, µm	5,330	5,191	5,586
Ball Mill 1 Product:			
% solids	47.0	39.6	39.8
D80, µm	145	177	133
Ball Mill 2 Product:			
% solids	47.0	39.6	31.5
D80, µm	52	100	69
Recycle, lb/h	2,500	5,200	1,909
Screen Undersize, D80, µm	30	42	67
Estimated Energy, kWh/st:			
Ball Mills	27.8	24.0	26.4
Netzsch Mill	60.5	52.1	
Total	88.3	76.1	26.4
Operating Work Index	52	54	24

5. COLUMN FLOTATION STUDIES

Very fine coal, such as the ground test coals, is generally cleaned by froth flotation. Column flotation was selected as the advanced flotation technology for this project since conventional flotation machines, which utilize high-shear impellers for agitation and bubble generation, do not produce low-ash coal very well. Hydraulic entrainment and entrapment of fine mineral particles in the clean coal froth are inherent problems with such machines. Column flotation, on the other hand, utilizes counter-current flow of feed slurry and air bubbles to separate coal particles from refuse particles under somewhat quiescent conditions. The addition of wash water above the feed point is a key feature of column flotation when cleaning fine coal. Such an addition is shown in Figure 7. When added in sufficient volume, the wash water creates a net downward bias flow which flushes refuse particles from the cleaning zone between the froth interface and the level where the feed slurry enters the column. The feed water that is displaced by the wash water flows down the column to the tailings outlet carrying with it any impurities that were not attached to the rising air bubbles.



From Drawing by G. R. Luttrell

Figure 7. Wash Water Flow During Column Flotation

The fraction of the wash water or bias ratio, in percent, that is available to flush impurities from the froth zone is calculated from wash water (WW) and air-free clean coal slurry (CCF) volumetric flow rates as follows:

$$Bias.ratio = 100\left(1 - {CCF}/{WW}\right).$$

Laboratory, bench-scale and PDU tests were conducted during the program applying column flotation technology to the production of clean coal meeting premium fuel specifications.

LABORATORY PROCESS OPTIMIZATION

The specific objective of the laboratory process optimization research was to determine the preferred column configurations and operating conditions for cleaning the six test coals. A topical report [**R-7**] was issued describing the laboratory process optimization.

A number of column flotation systems have been developed for coal flotation. The design and geometry of most of these systems are similar except for the manner of introducing and dispersing air in the slurry (the bubble generating system). Of the better known systems, the Ken-Flote™, GL&V Ontario packed column and the Microcel™ systems have consistently shown superior performance. Accordingly, the configurations of these three systems were selected for study during the laboratory optimization of column flotation.

The laboratory work was divided among the team members to accomplish project objectives. CAER had lead responsibility for investigating equipment design parameters and circuit comparisons. They specifically examined the effect of differing column height and did a comparison study of the packed column and the Ken-Flote™ column fitted with internal (Foam-Jet) and external (Turbo Sparger) aeration systems. CCMP tested the Microcel™ aeration system. Reagent comparisons were made at Amax R&D. The six test coals were divided between the three locations for the flotation response testing to determine coal-specific design parameters. One of the coals was tested at all three locations in order to obtain comparisons of flotation results among the three differing procedures.

Bituminous Coals

The bituminous coal testing was accomplished in 2-, 3- or 4-inch diameter columns which were between 8 and 26 feet tall [**R-7**]. The columns were set up for continuous feed and continuous discharge of froth and refuse and, depending upon their diameter, could handle between 50 and 500 grams per minute of fine coal. The feed slurries were prepared in the bench-scale grinding circuit at Amax R&D. The main operating variables studied during the parametric testing were feed rate and percent solids, retention time, wash water rate, aeration rate, reagent type and dosage, and column height. The differences in response between several particle size distributions were evaluated for some of the coals.

The five ground bituminous test coals generally responded quite well to the column flotation. Residual ash, sulfur and Btu recovery specifications were easily met while floating the Elkhorn No. 3, Taggart and Sunnyside coals. In fact, the Taggart coal could be cleaned to less than 1.0 lb/MBtu ash. It appeared that either the residual ash or the Btu recovery specification might need to be relaxed, though, if Indiana VII or

Winifrede coals were to be processed into premium fuel by column flotation. The Winifrede coal also needed to be ground finer than the minus 325-mesh grind tested in the laboratory-scale columns.

Each of the aeration configurations separated ash from coal quite well. The packed column required more air, though, and may not have been quite as efficient for rejecting ash. The separations with the Ken-Flote™ Foam-Jet and Microcel™ systems were similar to each other, so there was little reason to choose one over the other at this stage of process development for production of ultra-clean coal. As such it was recommended that the follow-up bench-scale testing include both the Ken-Flote™ and the Microcel™ columns.

Retention time (feed rate) and wash water additions appeared to be the most important variables when operating the flotation columns. Finer particle size distributions required longer retention times and more wash water in proportion to the feed rate. Lower solids concentrations in the column, and in the feed to the column, may be desirable, but within the range tested column height was not a critical factor effecting performance. Collector requirements (diesel fuel in these tests) were similar to mechanical cell flotation (around 1 lb/st). Frother dosages may be a critical operating parameter, but it was not clear from the laboratory work what the proper type or amount should be.

Subbituminous Coal

Only laboratory Denver-cell tests were made on the Dietz subbituminous coal [**R-7**]. As one would expect considering the rank of the coal, flotation was practically non-existent when following usual coal flotation practice. Large quantities of reagents were required before any flotation occurred at all, and pH adjustment had little impact upon the behavior. However, the selective agglomeration procedure developed for the liberation studies described in the previous section showed that subbituminous coal could be oil coated, and presumably made hydrophobic, by a sequence of acidification and high-shear treatments. Scoping tests were made to adapt this procedure to the preparation of microagglomerates recoverable by froth flotation.

Tests were made on ground Dietz coal using heptane bridging liquid containing 2 percent asphalt. The slurry was first acidified to pH 3.5 with hydrochloric acid and the sheared in a Waring blender along with the bridging liquid. The microagglomerated slurry was transferred to the Denver cell and floated with MIBC frother. The froth was reslurried and cleaned two more times to simulate the washing action occurring during column flotation. Reasonably good results were obtained with 40-percent bridging liquid. The clean coal contained 2.67 percent ash (2.09 lb/MBtu) at 86.9 percent Btu recovery. Unfortunately, performance rapidly declined when less bridging liquid was used. No further development work was done on the procedure because it did not appear that the efficiency of the process would overcome the cost of the acid and asphalt and the cost of recycling the bridging liquid.

Since there did not appear to be an economically viable scheme for flotation of Dietz subbituminous coal, it was not tested in the column flotation system, and with the permission of DOE project management, further development of subbituminous coal column flotation was dropped from the premium fuel program.

BENCH-SCALE FLOTATION

The primary objective of bench-scale flotation testing was to verify that the performance and operating characteristics observed during the laboratory column flotation can be scaled up to a 100 lb/h system when cleaning the specified test coals. The scale-up parameters determined from this work will be used for design of the 2.0-st/h PDU. A detailed account of the bench-scale testing was presented in a topical report [**R-9**].

A DOE-owned 12-inch diameter Ken-Flote™ column was procured for the bench-scale testing. The equipment had last been used as part of the Emerging Technologies program at the Pittsburgh Energy Technology Center by Process Technology Inc and they helped set up the system at Amax R&D. On a cross-sectional area basis this column represented a 9- to 16-fold scale-up of the laboratory equipment in use at Amax R&D, CAER and CCMP. The Ken-Flote™ column was supplemented by a 12-inch Microcel™ column purchased from Minerals and Coal Technologies Inc. The two columns were installed in parallel to take advantage of the pumps and data logging facilities included with the Ken-Flote™ system. Feed slurries were prepared by grinding crushed coal in the ball mills available in the Amax R&D pilot plant.

Parametric tests were first conducted on the five bituminous test coals using the Ken-Flote™ system. Comparison tests and further rounds of process optimization were subsequently made in the Microcel™ column for the three coals which appeared to be best suited for the PDU operation. The effects of varying feed rate/retention time, wash water addition, aeration and reagent dosage on Btu recovery and ash rejection were quantified during the parametric testing and optimization. Samples from selected parametric tests were analyzed to determine the distribution of the toxic trace elements during advanced flotation cleaning (Chapter 7).

Flotation Equipment

The 12-inch Ken-Flote™ and the Microcel™ flotation columns were both fabricated from steel and were 14 feet tall. However, they differed in the manner in which the air was introduced into the slurry.

The Ken-Flote™ column was furnished with a Foam-Jet sparger fitted with four porous metal plugs. This sparger consisted of two concentric tubes. The inner tube was filled with water under pressure, and the annular space between the tube contained compressed air. The four holes connecting the inner tube with the outside were filled with porous metal plugs. A small orifice drilled though each plug allowed a high-velocity flow of water to pass from the inner tube to the outside carrying with it tiny bubbles of the air which had passed through the porous metal.

The Microcel™ column was furnished with a microbubble generating system consisting of a centrifugal pump and a Koflo in-line static mixer. The pump drew tailings slurry from the bottom of the column and returned the slurry 12 inches higher up in the column. Compressed air was injected into the pump flow just ahead of the in-line mixer where it was dispersed into microbubbles which flowed into the column along with the recycle tailings slurry.

Feed slurry from the grinding circuit storage tank was metered to the columns with a variable-speed progressive cavity pump controlled to supply the desired feed rate. Flotation reagents and dilution were added in the feed sump as needed. The column feed rate was specified based on volumetric and percent solids requirements from the test plan and controlled from an in-line flowmeter to maintain the required set point. A specific amount of froth wash water was added via a submerged distributor in the froth column. Spray water was added to facilitate flow of the clean coal froth down the overflow launder. The column feed slurry and water addition flow rates were recorded by a computer data logging system. Aeration rates were also monitored and controlled via a computer feedback loop.

The froth/slurry interface level in the Ken-Flote™ column was maintained using the signal from an air bubbler tube to indicate the interface level and set the speed of a progressive cavity tailings pump. The interface level for the Microcel™ column was sensed by a pressure sensor mounted on the side of the column. The signal from the sensor controlled an air-operated pinch valve which regulated the gravity flow of tailings slurry from the Microcel™ column. In each case, the flow rate of the tailings stream was measured with a magnetic flow meter and the froth flow estimated by the difference between the tailings flow and the sum of the feed slurry volume and the wash water volume.

Control of the column flotation unit was accomplished using “fuzzy-logic" software developed by Process Technology Inc. All computer inputs and outputs were continuously displayed and logged into a file which could be exported to a spreadsheet program.

Parametric Flotation Testing

The initial operation of the bench-scale flotation rig consisted of short parametric tests on each coal using the Ken-Flote™ column. The primary variables considered were feed rate in dry pounds per hour, percent solids in the feed slurry (which sets retention time), aeration rate in cubic feet per minute, and wash water additions in gallons per ton of dry feed coal.

Because of time constraints, the Microcel™ testing focused on the three coals selected for study in the PDU during Phase II. Several rounds of additional optimization testing were conducted on these coals in the Microcel™ unit in order to provide information for designing the PDU.

The test slurries ground for the flotation campaigns met the following specifications:

	Top Size	D80, µm
Taggart	62 mesh	103
Indiana VII	325 mesh	22-24
Sunnyside	62 mesh	106
	100 mesh	70
	150 mesh	50
Winifrede	20 µm	11 -15
Elkhorn No. 3	62 mesh	108
	200 mesh	45

Ken-Flote™ Results

Data were collected for 110 tests with the Ken-Flote™ column spread out over a six-month period [**R-9**]. The Ken-Flote™ work began with the Elkhorn No. 3 coal, but subsequent work on Sunnyside coal provided a better assessment of the effects of changing flotation variables since by then the crew had gained a better understanding of the operating characteristics of the equipment. Eight tests were performed on the Taggart coal, and the coal responded very well to the column flotation. The Winifrede and Indiana VII coals presented special problems because of the very fine grinding required to liberate the ash from these two coals and because of the poor flotation characteristics of Indiana VII coal.

Elkhorn No. 3 Coal

Two series of parametric tests were made on Elkhorn No. 3 coal. The first series was on slurry that had been ground to minus 62 mesh, and the second on slurry ground to minus 200 mesh. Heating value recoveries in excess of 90 percent could be achieved with clean coal product ash contents (residual ash) of less than 2.0 lb/MBtu at both the minus 62 mesh and the minus 200 mesh grinds. Performance appeared to be somewhat better at the minus 62 mesh grind. Higher wash water additions and longer residence times were required at the finer grind in order to achieve acceptable results.

There was a clear trend showing that a more positive downward bias flow of wash water resulted in better rejection of ash from the froth. At equal bias flows, ash rejection from minus 62-mesh coal was just as good as from minus 200-mesh coal. This means that grinding finer than 62 mesh did not liberate enough more ash mineral from Elkhorn No. 3 coal to have any impact upon the amount of residual ash in the froth. Something over 2000 gallons of wash water were required per ton of the finer coal in order to meet the 2 lb/MBtu specification, but less was required at the coarser grind. The difference in wash water requirements can be attributed to the volume of water carried over with the froth. The froth from the coarser grind averaged 15.4 percent solids while the froth from the finer grind averaged 9.3 percent solids. It is possible that the amount of water and entrained ash mineral carried over from the finer slurry could have been reduced by optimization of the reagent dosages. Btu recovery fell, though, during a test where the MIBC was cut back to 0.5 lb/ton. Cutting back the dosage of diesel fuel had less impact upon flotation response.

Eight minutes of retention time were adequate for flotation of minus 62-mesh coal in the Ken-Flote™ system, but a slightly longer time was needed for the minus 200-mesh coal. The highest froth loading seen was 0.12 ton/h/sq ft when feeding minus 62-mesh coal. The best Hancock efficiency, at 54.1 percent, was noted for the test which had the highest ratio of wash water usage. Varying aeration, coal feed rates, and percent solids had little impact upon the Hancock efficiency index*****. Performance goals were best met during the following bench-scale tests:

	Test 9	Test 12
Operating Conditions:		
Feed Rate, lb coal/hour	120	180
Feed Slurry D80, µm	108	108
Aeration, cfm	1.70	1.70
Wash Water, gallon/ton feed coal	2200	1600
Wash Water, gpm	2.20	2.40
MIBC, lb/ton	1.0	1.0
Diesel Fuel, lb/ton	1.5	1.5
Retention Time, minutes	8.55	8.05
Performance:		
Residual Ash in Clean Coal, %	2.40	2.69
Residual Ash in Clean Coal, lb/MBtu	1.65	1.86
Higher Heating Value (HHV) Recovery, %	90.4	91.3
HHV Recovery from Raw Coal, %	85.5	86.4
Bias Flow Ratio, %	38.6	-9.5
Hancock Efficiency Index	54.1	44.7

Sunnyside Coal

Two series of parametric tests were also made on Sunnyside coal in the Ken-Flote™ system. The first series was on slurry that had been ground to minus 62 mesh, and the second on slurry ground to minus 150 mesh. Because of the benefits of increased wash water flows observed during flotation of Elkhorn No. 3 coal, larger amounts of wash water were used for these tests than during the previous tests.

Heating value recoveries in excess of 90 percent were achieved at both grinds, but residual ash concentrations of less than 2.0 lb/MBtu were marginally achieved when grinding only to minus 62 mesh. The 2.0 lb/MBtu ash specification was easily met when floating the finer minus 150-mesh slurry. At equivalent bias flows, ash rejection was better after grinding to minus 150 mesh which showed that finer grinding did improve liberation of the ash minerals in Sunnyside coal.

***** The Hancock efficiency index is defined as the percentage of the heating value recovered with the clean coal minus the percentage of the ash reporting with the clean coal.

All of the tests on the minus 62-mesh Sunnyside coal were with 2200 gallons of wash water per ton of feed coal. In each case there was a positive downward bias flow. For minus 150 mesh coal, increasing the wash water addition from 2200 gallons/ton on up to 3200 gallons/ton increased the bias flow but had little impact upon ash rejection. The extra wash water had little effect upon ash rejection because the original amount of wash water added exceeded the amount of water carried over in the froth by a comfortable margin.

Changing aeration rates had a mixed effect when floating Sunnyside coal. Increasing the aeration seemed to increase the amount of residual ash when floating the minus 62-mesh coal but seemed to decrease the amount of residual ash when floating the minus 150-mesh coal. The changing aeration must have affected the attachment of middling and ash minerals to the air bubbles since the aeration rate had little impact upon the bias flow washing entrained material from the froth. The highest Btu recovery was with 1.5 cfm air.

Eight minutes of retention time were adequate for flotation of both the minus 62 mesh coal and the minus 150-mesh Sunnyside coal. The highest froth loadings were 0.12 ton/h/sq ft when feeding minus 62 mesh coal and 0.10 ton/h/sq ft when feeding minus 150-mesh coal. Hancock efficiencies were higher when floating the minus 150 mesh coal than when floating the minus 62-mesh coal, and the highest efficiency, 48.7, was seen during a test where the clean coal contained only 1.69 lb/MBtu residual ash. Varying aeration, coal feed rates and percent solids had little impact upon the Hancock efficiency index. Operating conditions and performance results for two bench-scale tests representing base conditions for optimizing Sunnyside coal flotation follow:

	Test 54	Test 71
Operating Conditions:		
Feed Rate, lb coal/hour	150	180
Feed Slurry D80, µm	106	50
Aeration, cfm	1.50	1.40
Wash Water, gallon/ton feed coal	2160	2600
Wash Water, gpm	2.70	3.90
MIBC, lb/ton	1.0	1.5
Diesel Fuel, lb/ton	1.5	2.5
Retention Time, minutes	8.7	7.9
Performance:		
Residual Ash in Clean Coal, %	2.82	2.49
Residual Ash in Clean Coal, lb/MBtu	1.96	1.72
Higher Heating Value (HHV) Recovery, %	93.7	91.2
HHV Recovery from Raw Coal, %	84.4	82.2
Bias Flow Ratio, %	17.8	15.6
Hancock Efficiency Index	35.9	47.1

Taggart Coal

The parametric tests on Taggart coal in the Ken-Flote™ system were performed on slurry that had been ground to minus 62 mesh. Heating value recoveries in excess of 90 percent were achieved for all but one of the tests, and residual ash concentrations of less than 1.0 lb/MBtu were achieved in each case. The bias flow for these tests were all in the -13 to -27 percent range since only one wash water/feed rate ratio was tested. Results were satisfactory with a negative bias flow because the feed contained only 2 percent ash. In other words, very little ash needed be flushed from the coal by the wash water.

As with the Elkhorn No. 3 and Sunnyside coals, 8 minutes of retention time were adequate for flotation of the Taggart coal. The highest froth loading was 0.12 t/h/sq ft when feeding minus 62-mesh coal. Hancock efficiencies were in the 27 to 33 range and were highest when recovery was sacrificed slightly either by a shorter retention time or by reduced aeration. The following test represented the best conditions for further optimization:

	Test 97
Operating Conditions:	
Feed Rate, lb coal/hour	200
Feed Slurry D80, µm	104
Aeration, cfm	1.50
Wash Water, gallon/ton feed coal	1740
Wash Water, gpm	2.90
MIBC, lb/ton	1.0
Diesel Fuel, lb/ton	1.0
Retention Time, minutes	7.2
Performance:	
Residual Ash in Clean Coal, %	1.38
Residual Ash in Clean Coal, lb/MBtu	0.91
Higher Heating Value (HHV) Recovery, %	94.7
HHV Recovery from Raw Coal, %	89.3
Bias Flow Ratio, %	-13.8
Hancock Efficiency Index	31.9

Winifrede Coal

The parametric tests on Winifrede coal in the Ken-Flote™ system were on slurry that had been ground to minus 20 µm. Because heating value recoveries were poor during the initial tests, reagent dosages were increased. The additional reagents recovered more coal but ash rejection was poor. Much more wash water was used during the final testing, but residual ash concentrations remained well above 2 lb/MBtu.

Residual ash concentrations of less than 2 lb/MBtu were only achieved when the Btu recovery was less than 20 percent. The only time residual ash concentrations approached 2 lb/MBtu was when the bias flow was downward by at least 50 percent.

The bias flow was negative (that is, upward) for most of the tests because of the low solids content of the froth. The solids concentrations in the froths ranged from 2.9 to 3.7 percent (or 6240 to 8030 gallons water per ton) except when very little material was being recovered. Less frother was added during one test in order to reduce the amount of water entrained in the froth. The change improved ash rejection somewhat, but the froth still failed to meet the residual ash goal.

Mostly, eleven minutes of retention time were required for good recovery of the minus 20 µm Winifrede coal in the Ken-Flote™ column. The highest Hancock efficiencies were between 27.2 and 28.1, and residual ash concentrations ranged from 3.29 on up to 3.77 lb/MBtu. Heating value recoveries ranged between 57 and 69 percent during these tests. Operating conditions and performance results for Test 49 are summarized below since they represent base conditions for further optimization. Results for Test 31 are also shown since it was the only test on Winifrede coal where the 2 lb/MBtu ash specification was met during the bench-scale testing:

	Test 31	Test 49
Operating Conditions:		
Feed Rate, lb coal/hour	75	75
Feed Slurry D80, µm	11	11
Aeration, cfm	1.50	1.70
Wash Water, gallon/ton feed coal	1600	5440
Wash Water, gpm	1.00	3.40
MIBC, lb/ton	1.0	1.5
Diesel Fuel, lb/ton	1.5	3.5
Retention Time, minutes	10.0	12.8
Performance:		
Residual Ash in Clean Coal, %	2.72	4.69
Residual Ash in Clean Coal, lb/MBtu	1.87	3.29
Higher Heating Value (HHV) Recovery, %	18.7	57.3
HHV Recovery from Raw Coal, %	16.8	51.3
Bias Flow Ratio, %	57.6	17.4
Hancock Efficiency Index	13.1	28.1

Indiana VII Coal

Parametric tests were conducted only on minus 325-mesh Indiana VII coal in the 12-inch Ken-Flote™ system. Recovery from the fresh Indiana VII coal was erratic. Reagent dosages were quite high -- 4 lb/ton MIBC and 10 lb/ton diesel fuel in most cases -- so the erratic recovery was not due to a reagent shortage. Residual ash concentrations of less than 2 lb/MBtu were achieved for only a few of the tests and only for heating value recoveries of 80 percent at best.

There were clear relationships between wash water usage, bias flow ratio and the amount of residual ash in the froth. Downward bias flows of 30 percent or more was necessary to reject sufficient ash to meet the 2 lb/MBtu specification. About 4000 gallons of wash water per ton of feed coal was required to achieve this bias ratio. This 4000 gallons was about twice the volume of the water in the overflowing froth.

Lowering the feed rate and feeding a more dilute slurry improved ash rejection. Changing the feed rate had little impact upon recovery but did have an impact on the residual ash concentration. Apparently the entrained ash did not drain well from the more heavily laden froths seen at the higher feed rates. Retention times at the faster and slower feed rates were comparable because of changes in the feed slurry solids concentrations.

Most of the Indiana VII flotation tests were made with 1.9 cfm air. Recovery fell when the aeration rate was reduced to 1.5 cfm but the residual ash declined at the same time. The reduced aeration also reduced the amount of water carried over by the froth. The latter change had a favorable impact upon the bias ratio. The more efficient use of wash water probably accounted for the reduction in the residual ash content of the froth at the lower aeration rates.

Fifteen minutes of retention time were required for good flotation recovery of the minus 325 mesh Indiana VII coal in the Ken-Flote™ column fitted with the Foam-Jet sparger. The highest Hancock efficiencies were the 59.6 and 60.5 noted for two tests which differed only by their reagents dosages. Residual ash concentrations were 1.95 and 2.09 lb/MBtu, respectively, and heating value recoveries were 81.4 and 84.9 percent, respectively. Operating conditions and performance results for these tests represent reasonable base conditions for beginning further rounds of optimization:

	Test 107	Test 110
Operating Conditions:		
Feed Rate, lb coal/hour	40	40
Feed Slurry D80, µm	22	22
Aeration, cfm	1.90	1.90
Wash Water, gallon/ton feed coal	4260	4260
Wash Water, gpm	1.42	1.42
MIBC, lb/ton	4.0	6.4
Diesel Fuel, lb/ton	10.0	16.0
Retention Time, minutes	15.2	15.4
Performance:		
Residual Ash in Clean Coal, %	2.75	2.94
Residual Ash in Clean Coal, lb/MBtu	1.95	2.09
Higher Heating Value (HHV) Recovery, %	81.4	85.0
HHV Recovery from Raw Coal, %	73.7	76.9
Bias Flow Ratio, %	29.7	32.2
Hancock Efficiency Index	59.6	60.5

Microcel™ Results

Microcel™ flotation data were collected for 106 parametric tests on the three coals selected for study in the PDU (Sunnyside, Taggart, and Indiana VII) [**R-9**]. The Microcel™ testing included a four-day production run on the Indiana VII coal and a brief run on Winifrede coal.

Indiana VII Coal

Regression statistics from the initial test matrix indicated that wash water usage, feed rate, and frother dosages had significant effects upon ash rejection and Btu recovery. The goal of 90 percent Btu recovery from the test coal (80 percent from raw coal) was met during most of the first-step matrix tests in the Microcel™ column, but the goal of less than 2.0 lb/MBtu residual ash was not. Operating points were calculated from the correlation equations, though, where both the recovery and ash rejection goals would be met. A selected number of these operating points were tested in the Microcel™ column during succeeding optimization steps. The requisite ash rejection was achieved consistently during the final part of the optimization testing. Generally, the Microcel™ results appeared to follow a more consistent pattern than the Ken-Flote™ results for minus 325 mesh Indiana VII coal.

There were clear relationships between wash water usage, bias flow ratio, and the amount of ash remaining in the froth. At least 3000 gallons of wash water were required per ton of feed in order to achieve a positive downward bias flow.

Additional aeration improved recovery somewhat but ash rejection suffered. Apparently the extra aeration impeded drainage of water from the froth which in turn reduced the bias flow and the effectiveness of the wash water.

It was found during the optimization step that frother dosage had an important effect upon recovery. Reducing the frother dosage was a convenient method for retarding middling flotation and entrainment of ash in the froth, thereby reducing the amount of residual ash in the clean coal. A range of recoveries could be achieved at retention times between 7 and 10 minutes merely by adjusting the amount of MIBC frother added to the feed slurry.

The highest Hancock efficiency index seen during the Indiana VII Microcel™ flotation was 74.8. Unfortunately, the product did not meet the residual ash goal. The highest Hancock efficiency index seen when the 2.0 lb/MBtu residual ash specification was met was 60.2. The Btu recovery during that test was 79.35 percent – close to the recovery projected from the laboratory scale work. A reduction in the MIBC frother dosage was the most significant operating difference between the two tests. Operating and performance data for these tests were as follows:

	Test 146	Test 214
Operating Conditions:		
Feed Rate, lb coal/hour	90	90
Feed Slurry D80, µm	23	23
Aeration, cfm	1.30	1.30
Wash Water, gallon/ton feed coal	6670	5590
Wash Water, gpm	5.00	4.19
MIBC, lb/ton	3.22	2.62
Diesel Fuel, lb/ton	5.00	5.00
Retention Time, minutes	7.8	8.8
Performance:		
Residual Ash in Clean Coal, %	3.55	2.78
Residual Ash in Clean Coal, lb/MBtu	2.54	1.97
Higher Heating Value (HHV) Recovery, %	97.0	79.4
HHV Recovery from Raw Coal, %	87.8	71.8
Froth Loading, ton/h/sq ft	0.05	0.04

Bias Flow Ratio, %	81.2	73.0
Hancock Efficiency Index	74.8	60.2

A four-shift production run was made on the Indiana VII coal to produce filter cake for the Pennsylvania State University Energy and Fuels Research Center coal slurry combustion program. Fifteen drums of filter cake containing 2441 pounds of coal (dry basis) were produced. Composite analyses (dry basis) of the shipment were as follows:

Ash, %	2.84
Ash, lb/MBtu	2.0
Sulfur, %	0.61
Sulfur, lb/MBtu	0.43
Heating Value, Btu/lb	14,154
Moisture, % (as shipped)	46.6

Operating conditions for the production run were patterned after those for Test 214 above. Consistent performance was achieved from period to period during the run, but the frother addition was cut back during the last two periods in order to produce filter cake containing less than 2.0 lb of ash per million Btu. The overall Btu recovery during the production run was 79.9 percent.

Taggart Coal

Unlike Indiana VII coal, the Taggart coal floated easily. In fact, the natural flotability of the Taggart coal produced comparable yield and quality values without regard to changes in the operating parameters. Heating value recoveries from the prewashed test coal were consistently between 98.7 and 99.3 percent and residual ash concentrations were consistently between 0.88 and 1.05 lb/MBtu.

Because results with the Taggart coal were exceptionally good throughout the parametric testing, the performance limits of the Microcel™ system remained unknown. For this reason, additional tests were performed to determine the threshold capacity and ash rejection limits. It was found from these tests that the capacity of the 12-inch column could easily reach 300 lb/hour when floating minus 62-mesh coal (0.17 tph/sq ft) and that considerable less MIBC and diesel fuel were needed than were used during the original matrix testing. Furthermore, residual ash contents of well under 1.0 lb/MBtu were achieved consistently during the final part of the optimization testing.

As noted during the Ken-Flote™ testing, Taggart coal needed less wash water than needed for the Indiana VII coal because there was much less ash in the feed coal to begin with and because the higher solids loading in the froth (16 plus percent during the Microcel™ testing) allowed more efficient use of the wash water.

The highest Hancock efficiency index seen during the Taggart Microcel™ flotation was 36.4. However, Btu recovery was slightly better during a preceding test with more reagent. A 1.0 lb/MBtu ash specification was easily met in both case. Operating and performance data for these two tests are presented below:

	Test 171	Test 172
Operating Conditions:		
Feed Rate, lb coal/hour	300	300
Feed Slurry D80, µm	103	103
Aeration, cfm	1.50	1.50
Wash Water, gallon/ton feed coal	1200	1200
Wash Water, gpm	2.99	3.00
MIBC, lb/ton	0.25	0.25
Diesel Fuel, lb/ton	0.50	0.25
Retention Time, minutes	8.0	7.8
Performance:		
Residual Ash in Clean Coal, %	1.34	1.22
Residual Ash in Clean Coal, lb/MBtu	0.88	0.80
Higher Heating Value (HHV) Recovery, %	95.9	91.8
HHV Recovery from Raw Coal, %	90.4	86.5
Bias Flow Ratio, %	-15.4	0.0
Hancock Efficiency Index	32.0	36.4

Sunnyside Coal

Sunnyside coal floated quite well in the 12-inch Microcel™ column, and the Btu recovery and residual ash concentration goals were met during many of the parametric tests. For this reason, the optimization work focused on the maximum allowable feed rate and identification of minimum reagent dosages. Adjustments in the reagent dosages improved selectivity and provided an overall reduction in reagent consumption.

The Microcel™ system may have provided somewhat better Btu recovery at specific residual ash concentrations than the Ken-Flote™ system. There were clear relationships between wash water usage, bias flow ratio and the amount of ash remaining in the froth. About 1500 gallons of wash water were required per ton of feed coal in order to a achieve a positive downward bias flow when using 1.0 lb MIBC frother per ton of feed coal. There were indications that the capacity of the 12-inch column approached the 300 lb/hour seen for the Taggart coal, but ash rejection suffered at the high feed rates.

Frother dosage had an important effect upon recovery. Reducing the dosage was a convenient method for holding back middling flotation and entrainment of ash minerals thereby reducing the residual amount of ash in the clean coal. The full range of recoveries could be achieved at retention times between 7 and 10 minutes by adjusting the dosage of MIBC frother.

The highest Hancock efficiency index seen during the Sunnyside Microcel™ flotation was 46.3. The efficiency index was almost as good with more frother and less diesel fuel, and the Btu recovery was 98.1 percent instead of 76.8 percent. A 2.0 lb/MBtu ash specification was met in both cases. Operating and performance data for these tests follow:

	Test 197	Test 205
Operating Conditions:		
Feed Rate, lb coal/hour	150	150
Feed Slurry D80, µm	70	70
Aeration, cfm	1.50	1.50
Wash Water, gallon/ton feed coal	2400	2400
Wash Water, gpm	3.00	3.00
MIBC, lb/ton	0.25	0.67
Diesel Fuel, lb/ton	1.00	0.23
Retention Time, minutes	10.3	10.8
Performance:		
Residual Ash in Clean Coal, %	2.03	2.69
Residual Ash in Clean Coal, lb/MBtu	1.40	1.86
Higher Heating Value (HHV) Recovery, %	76.8	98.1
HHV Recovery from Raw Coal, %	69.2	88.4
Froth Loading, ton/h/sq ft	0.07	0.09
Bias Flow Ratio, %	65.7	56.3
Hancock Efficiency Index	46.3	44.4

Discussion of Bench-Scale Flotation Results

The amount of residual ash in clean coal was generally found to correlate closer to the bias ratio than to any other operating variable. Figure 8 is an example of the correlation for Indiana VII coal. The bias ratio largely depended upon the amount of wash water added at the top of the column and the amount of water carried over with the froth. It appears that the amount of water added at the top should exceed the amount carried over with the froth by a comfortable margin (bias ratio >50 percent) to ensure a positive downward flow of wash water to the tailing.

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 (Figure 9). This is consistent with the capacity/particle size relationship generally seen for column flotation. The particle size distribution of the feed slurry also had an impact upon the optimum amount of wash water needed for ash rejection (Figure 10). The coarsely ground slurries (Taggart, Elkhorn No. 3, and Sunnyside) did not need nearly as much wash water as did the finely ground slurries (Indiana VII and Winifrede). Part of the need for extra wash water when floating finely ground slurry can be attributed to the lower solids concentration in the froth when floating finer coal (Figure 10). The drainage of water from the froth was also influenced by the feed and aeration rates, by the reagent schedule and probably by the froth depth.

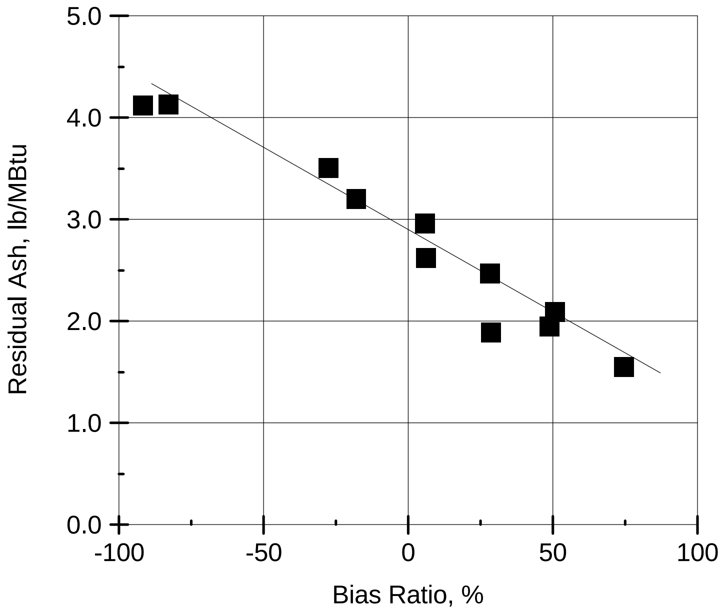


Figure 8. Residual Ash versus Bias Flow for Indiana VII Coal Flotation in Ken-Flote™ Column

It appears from a comparison of optimized operating conditions that reagent requirements were less with the Microcel™ system than with the Ken-Flote™ system. Wash water requirements were less for one coal, greater for another coal and about the same for the third coal. The Microcel™ column appeared to have a greater capacity than the Ken-Flote™ column for flotation of Taggart and Indiana VII coals and somewhat less capacity for flotation of the Sunnyside coal. Microcel™ Btu recovery was significantly better for Sunnyside coal, but in other respects, and for the other two coals, the separation performances of the two systems were close to being the same as illustrated by the grade-recovery results for the Sunnyside coal plotted in Figure 11.

Rejection of Sulfur

The five test coals were all relatively low in sulfur and each one had been washed at the mine to reject mineral matter. As a result, the flotation feed samples did not contain much pyritic sulfur, the highest of the five original head samples being the 0.17 percent pyritic sulfur (0.12 lb/MBtu) in the Elkhorn No. 3 washed coal. The organic sulfur contents of the test coals ranged between 0.34 percent (0.25 lb/MBtu) for the Indiana VII coal on up to 0.79 percent (0.57 lb/MBtu) for the Winifrede coal.

Total sulfur and pyritic sulfur balances were obtained for selected tests. Only minor reductions in sulfur contents were accomplished since most of the sulfur in these coals was in the organic form. Pyritic sulfur was rejected from the Sunnyside, Indiana VII, and Winifrede coals, but the amount rejected had little impact upon the final concentration of sulfur in the respective clean coals. The sulfur contents of Elkhorn No. 3 clean coals varied erratically and did not always meet the premium fuel goal. Very likely the Elkhorn No. 3 test coal had been loaded while the source of the mine-run coal feeding the washing plant was changing.

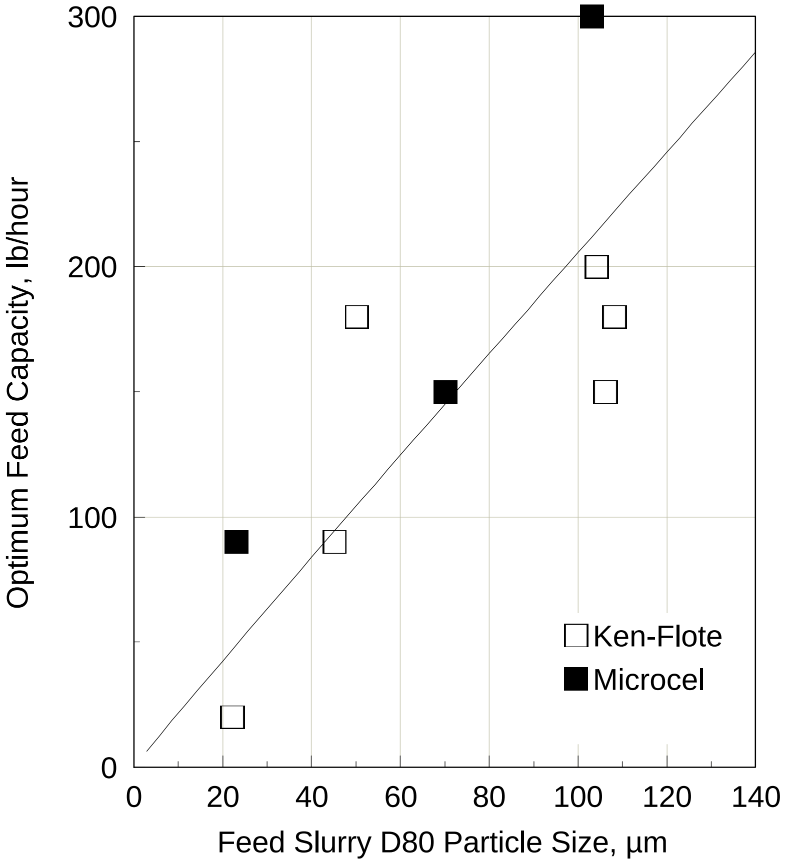


Figure 9. Flotation Capacities of 12-inch Columns versus Particle Size Distribution of Coal Slurry

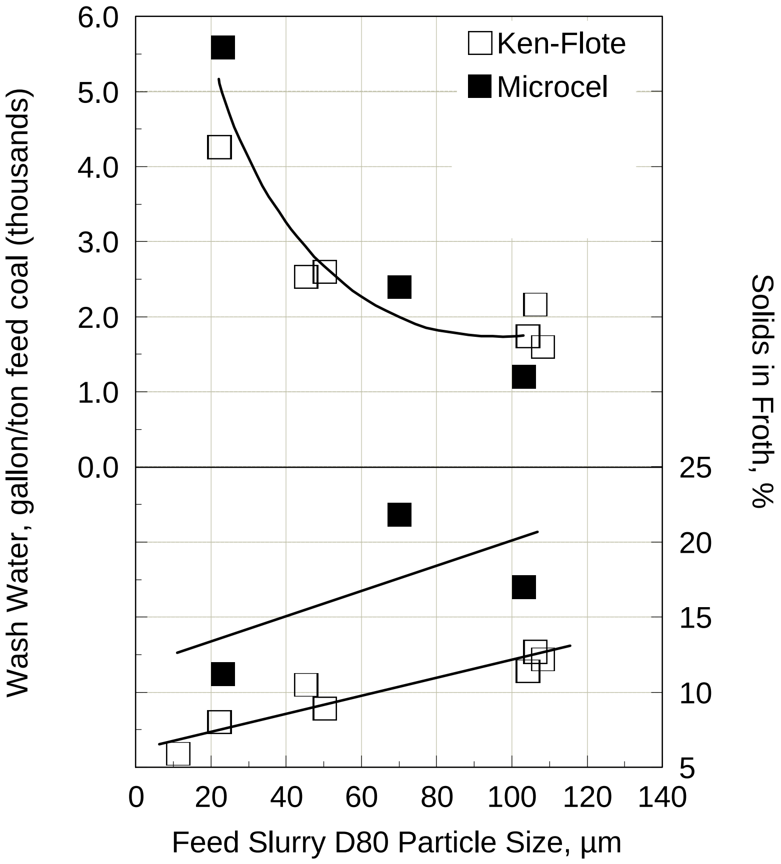


Figure 10. Effect of Particle Size Distribution on Wash Water Requirements for Ken-Flote™ and Microcel™ Flotation

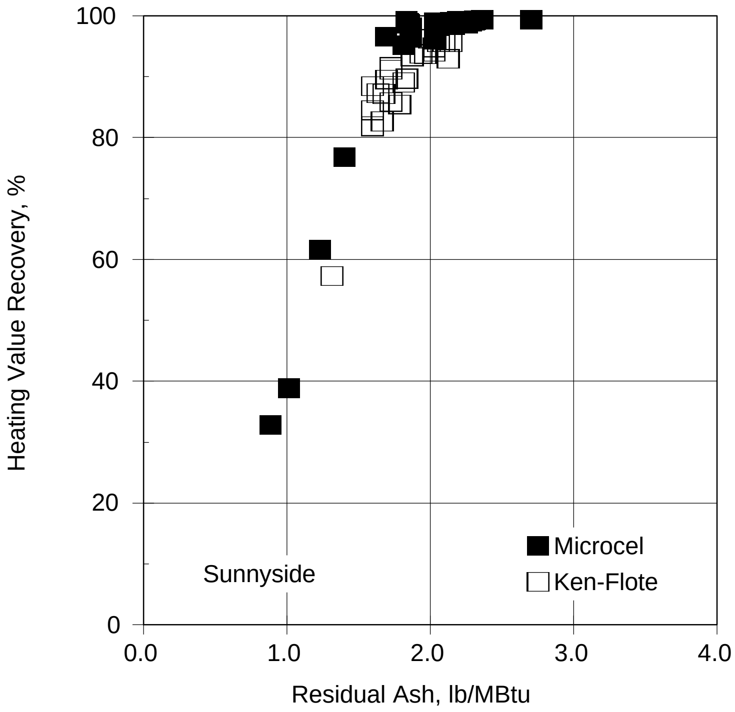


Figure 11. Grade-Recovery Plot for Flotation of Sunnyside Coal

Scale-Up Parameters for Design of PDU

The equipment capacity and performance scale-up parameters derived from the bench-scale testing that were recommended for design of the PDU [**R-9**] are presented in Table 4 for each of the test coals. As indicated earlier in Chapter 3, Taggart, Sunnyside and Indiana VII coals were selected from these five for PDU testing.

PDU OPERATIONS

The conceptual design of the PDU and Flotation Module was a collaborative effort between Bechtel, Entech, CCMP, and CAER. Much of the early effort was devoted to consider the type and scale-up of the flotation column to be used, the installation of a cyclone/fine screen particle size classification system, the types of fine grinding mill and refuse thickener to be installed, and whether to include one or two stages of flotation and to reuse DOE-owned filters and other available equipment [**R-10**, **R-21**]. Once these issues were resolved, the detailed design of the PDU and Flotation Module was performed by Bechtel Corporation with support from Entech Global engineers. The detailed design [**R-11**] was for a plant that would fit inside an existing building at the Amax R&D Center in Golden, Colorado and utilize existing support services at that location.

Table 4. Equipment Capacity and Performance Scale-up Parameters Derived from 12-Inch Column Flotation of Five Washed Coals

Note: **Bold face** entries were determined during the test program. Other entries were derived from the bold-face entries.

	Taggart	Elkhorn No. 3	Sunnyside	Indiana VII	Winifrede
Feed Coal:					
Ash, %	**2.10**	**6.00**	**5.50**	**9.50**	**8.50**
Rate, st/h per sq ft	0.17	0.18	0.12	0.05	0.03
Feed Slurry, % solids	10	10	10	6	6
Feed Slurry, gpm/sq ft	6.6	7.2	4.7	3.6	0.9
Grind, D80 µm	**104**	**104**	**70**	**24**	**12**
Clean Coal:					
Ash, %	**1.37**	**2.75**	**2.74**	**2.82**	**2.91**
Ash, lb/MBtu	0.90	1.90	1.90	2.00	2.00
Yield, wt %	94.2	86.7	92.0	74.0	74.9
Btu recovery, %	**95.0**	**90.0**	**95.0**	**80.0**	**80.0**
Froth Carrying Capacity, st/h per sq ft	**0.16**	**0.16**	**0.11**	**0.04**	**0.02**
Froth Slurry, % solids	**17**	**16**	**20**	**12**	**6**
Fine Refuse:					
Ash, %	14.0	27.2	37.2	28.5	25.2
Weight Distribution, %	5.8	13.3	8.0	26.0	25.1
Tailings Slurry, % solids	0.50	1.19	0.58	0.85	0.46
Tailing, gpm/sq ft	7.8	8.2	6.6	6.6	5.8
Wash Water, gallon/short ton feed coal	**1800**	**1800**	**2200**	**6500**	**18800**
Wash Water, gpm/sq ft	4.8	4.8	4.0	4.3	6.3
Superficial Wash Water Flow, ft/minute	0.64	0.64	0.54	0.58	0.84
Bias Flow Ratio, %	35	30	56	73	80
Aeration Rate, scfm/sq ft	**1.9**	**1.9**	**1.9**	**1.7**	**1.7**
Superficial Air Flow, ft/minute	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, lb/short ton	**0.25**	**1.00**	**0.67**	**3.00**	**1.50**
Diesel Fuel, lb/short ton	**0.50**	**1.50**	**0.23**	**5.00**	**3.50**

The construction contract for the PDU and Flotation Module was awarded to TIC - The Industrial Company, of Steamboat Springs, Colorado on February 18, 1995. Construction was completed on August 31, 1995. Start-up and shake-down proceeded according to a test plan [**R-20**] approved by DOE project management and was completed in time for the parametric testing to begin in January 1996.

Process and Plant Description

As described in the detailed design [**R-11**] and reports [**R-20**, **R-21**], the advanced coal cleaning PDU and Flotation Module was divided into areas for design and operating purposes:

- Area 100 - Raw coal handling
- Area 100 - Grinding and classification
- Area 200 - Column flotation
- Area 400 - Clean coal dewatering
- Area 400 - Fine refuse (tailings) dewatering

Installation of the Area 300 Selective Agglomeration Module was deferred until after completion of the column flotation testing since operation of the agglomeration module required modification of the grinding circuit and the clarified water system.

All areas of the PDU were operated from a central control room utilizing an integrated graphic data-logging and control system furnished by Honeywell. Generally there were one control room operator and three area operators working on each shift. The operations were supervised by an engineer and supported by a technician performing analytical duties.

Equipment for the PDU and Flotation Module was sized to allow production of 2 st/h of Taggart and Hiawatha clean coals. It was anticipated that the plant capacity would be less for the Indiana VII coal because of the finer grinding that would be needed for that coal and because of its poorer flotation response.

Area 100 - Raw Coal Handling

The coals cleaned in the PDU and Flotation Module were taken from the normal 2-inch x 0 production of the source mines and most were delivered by rail to a coal yard in Denver. From there they were trucked as needed to Ralston Development Company where they were crushed to 1/2 inch and hauled to a covered storage pile at Amax R&D. A front-end loader was used to move the coal from the pile to a 15-ton receiving hopper for metering into the coal grinding circuit.

Area 100 - Grinding and Classification

As shown in Figure 12, the PDU utilized two mills in series for the initial grinding. These mills were charged with steel balls. The two ball mills were followed by a fine grinding

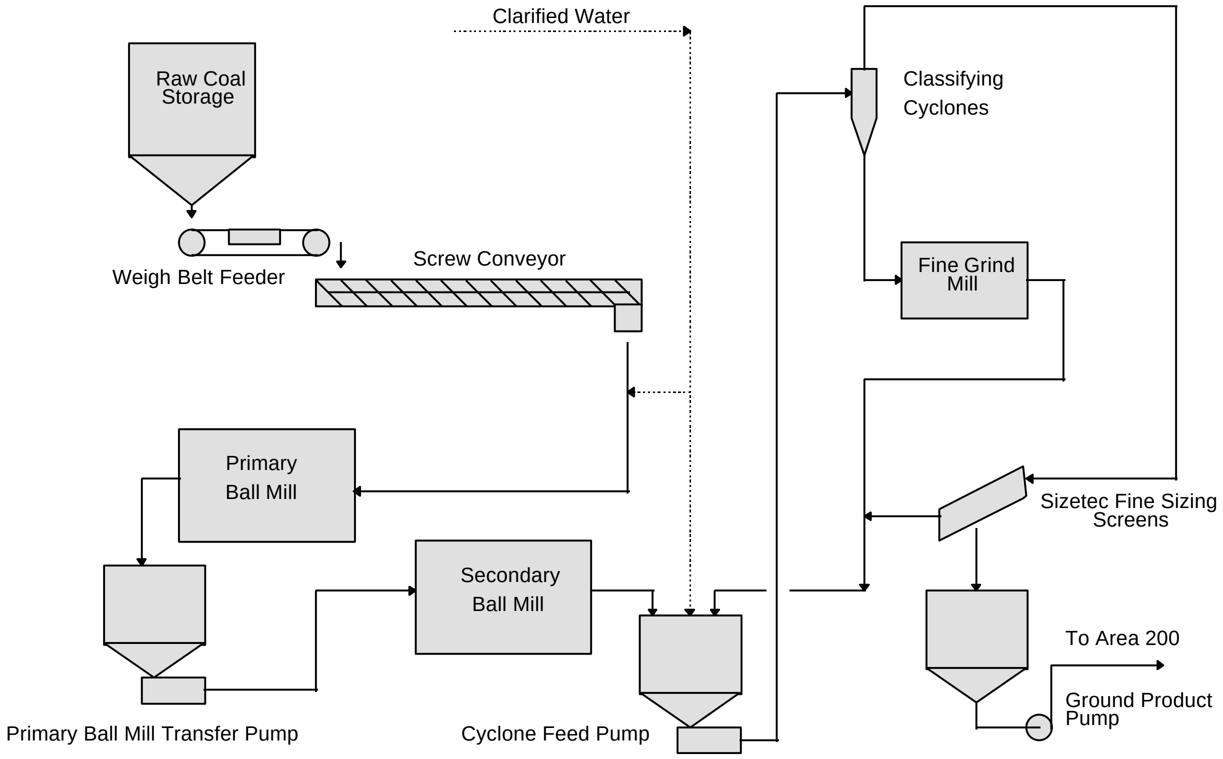


Figure 12. PDU Area 100 - Grinding / Classification Circuit

mill to achieve final liberation of the ash minerals. The fine grinding mill was charged with glass beads.

Clarified recycle water was added to the ball mills along with the coal to form a slurry in the mills. The slurry exiting the secondary ball mill was pumped to a cluster of 2- and 3-inch diameter classifying cyclones. To insure that the particle top size constraint was maintained, the cyclone overflow stream was sent to a pair of high frequency fine sizing screens to remove any oversize material before flotation.

The screen oversize material and the cyclone spigot product were ordinarily pumped to the bead mill for further grinding and return to the classifying cyclone feed pump. There was considerable flexibility in this part of the circuit since either the 2-inch or the 3-inch cyclones could be used for the classification and the oversize and spigot products could be routed to the secondary ball mill instead of to the fine grinding mill.

Area 200 - Column Flotation

A schematic process flow diagram of Area 200 is shown in Figure 13. The area contained a Microcel™ flotation column purchased from Control International. The column was 6 feet in diameter and almost 29 feet tall. Slurry entered the unit below the froth interface at a point approximately 8 feet below the overflow. The slurry moved downward through the column encountering a rising flow of air bubbles. The rising bubbles and attached coal particles formed a layer of froth at the top of the column that

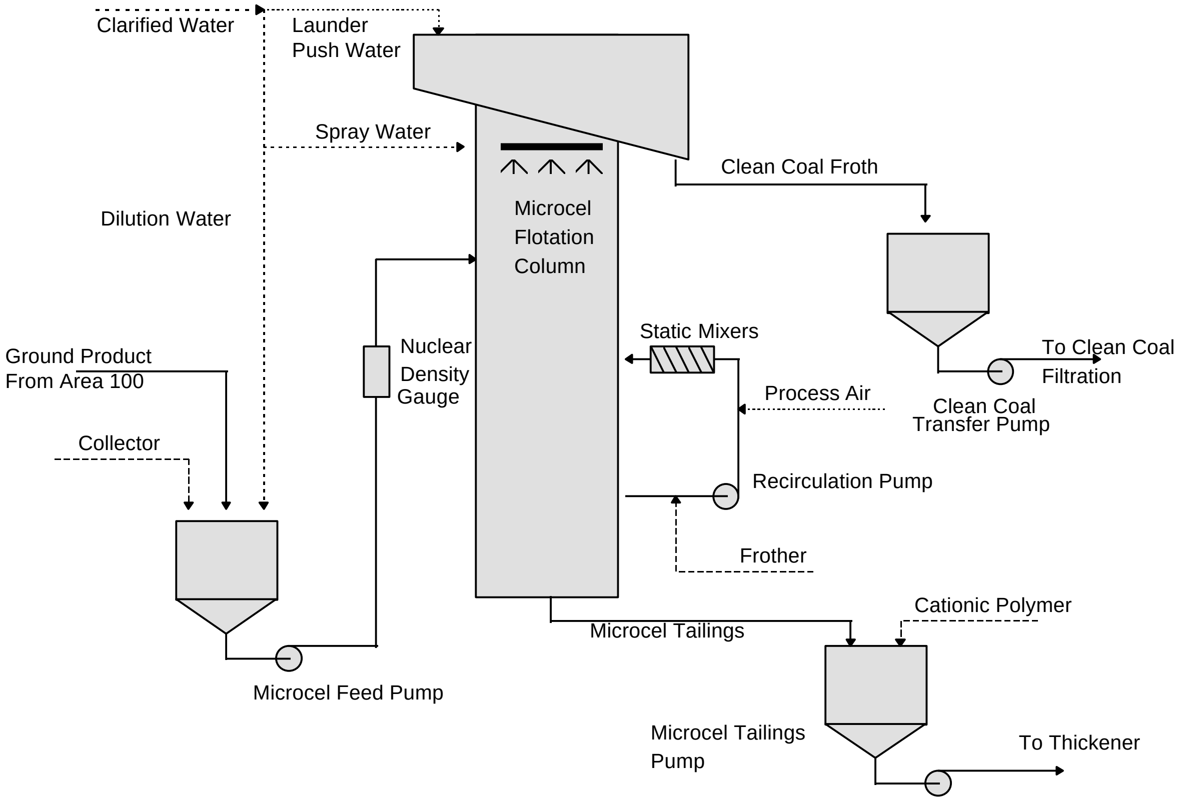


Figure 13. PDU Area 200 - Microcel™ Column Flotation Circuit

was 1 to 5 feet deep. A downward flow of wash water was distributed into the froth zone to wash out unwanted entrained mineral matter and clay particles. The air bubbles were generated by shearing pressurized air that had been injected into tailings slurry pumped though four externally mounted static in-line mixers. MIBC frother was metered into the centrifugal pump which recirculated the tailings through the mixers. The slurry level in the column was controlled by increasing or restricting the flow of tailings exiting the unit using the signal from a pressure transducer to locate the position of the froth/slurry interface.

Area 400 - Clean Coal and Tailings Dewatering

Three filters were used to dewater the clean coal (Figure 14). A Westech vacuum drum filter was used as the primary filtration unit while two Netzsch presses filtered the remaining clean coal slurry. The filter cakes were collected in bulk bags for storage or disposal. Filtrates were transferred to an Enviro-Clear thickener where they were clarified for reuse.

Tailings from the Microcel™ column were also sent to the Enviro-Clear thickener for initial dewatering (Figure 15). Cationic and anionic polymer flocculants were added to the tailings stream to accelerate sedimentation. The clarified water overflowing the top of the unit was pumped back into the process. The thickened solids formed a slurry of

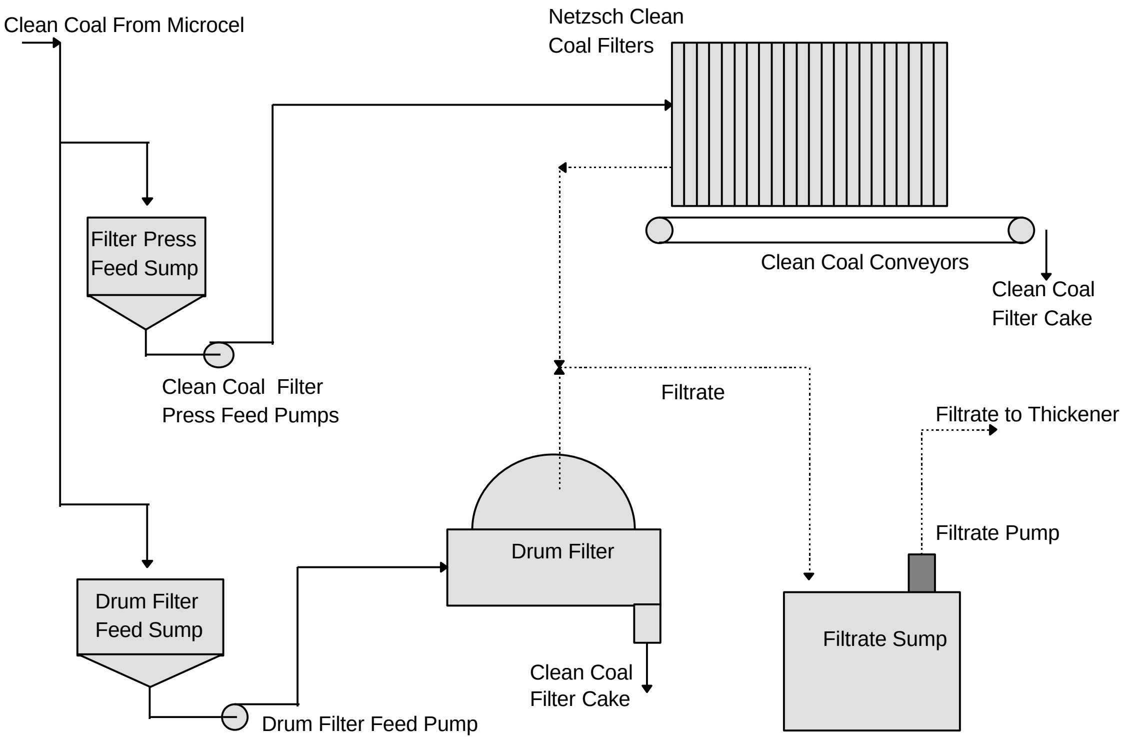


Figure 14. PDU Area 400 - Clean Coal Dewatering Circuit

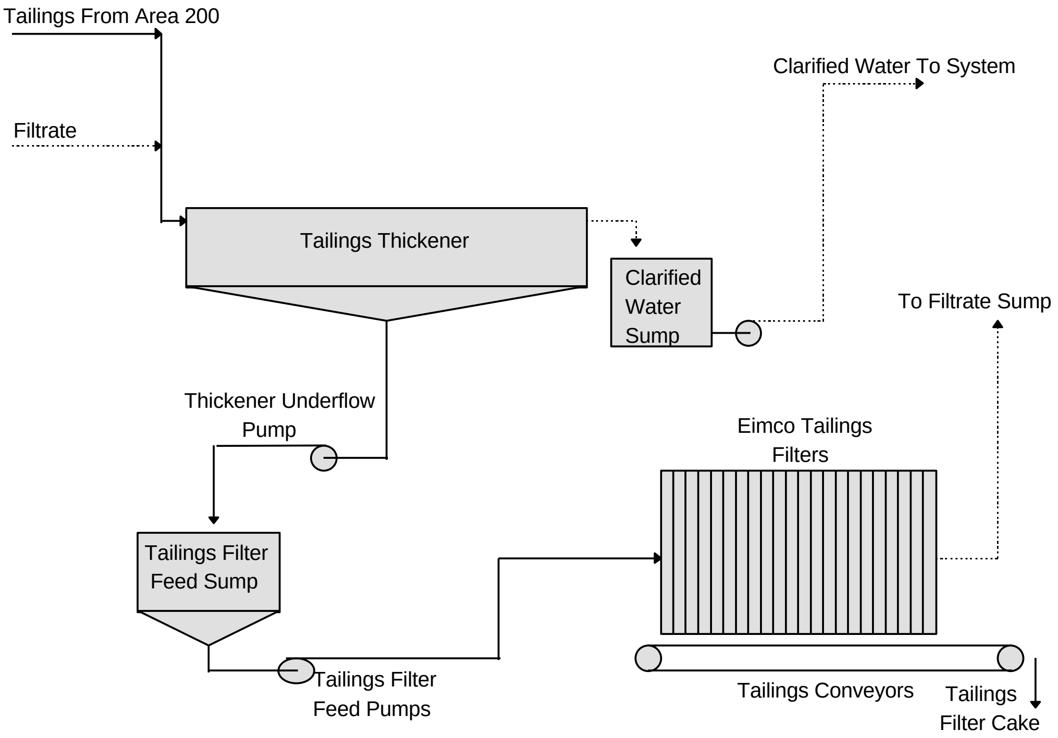


Figure 15. PDU Area 400 - Tailings Dewatering Circuit

20 to 30 percent solids which was filtered in two Schriver plate-and-frame presses for land-fill disposal in bulk bags.

Flotation Module Operation and Test Work

The operation and testing of the Flotation Module was in accordance with the test plan [**R-20**] approved by the DOE. Beginning with Taggart coal, the basic strategy was to conduct a planned series of 19 parametric tests, follow-up with 4 or more additional tests to identify optimum operating conditions, and complete the series with a 72-hour round-the-clock production run before moving on to the next coal. The process variables included in the parametric test matrix included MIBC and fuel oil additions, percent solids in the feed slurry, aeration rate, wash water addition, recirculation rate to the aerator, and the feed rate of the coal. Additional tests were made to tune the operation of the grinding circuit for each coal in order to produce the desired particle size distribution and liberation for the flotation separation.

The test work began during January 1996 and was finished with Hiawatha coal during September 1996. Over 1,000 tons of coal were processed through the PDU during operation of the Flotation Module.

Flotation of Taggart Coal

Twenty-five tests were conducted aimed at tuning the ball mill circuit for grinding the Taggart coal before flotation. It was found that the desired clean coal quality of 1 lb ash/MBtu could be achieved in the PDU at D80=52 µm by using both ball mills, 100-mesh screens and the 3-inch cyclones [**R-21**].

The ground Taggart coal readily floated. In fact, the natural flotability of the coal produced comparable yield and quality values regardless of changes in the operating parameters. Noticeable changes in the yield and quality usually were observed only when the input parameters were varied dramatically. Overall, the quality goal of 1 lb ash/MBtu was met or exceeded in four of the parametric tests. The clean coal yield varied from 58.5 to 96.6 percent while the Btu recovery and residual ash varied from 60.1 to 98.0 percent and 0.77 to 1.23 lb/MBtu, respectively. This is illustrated by the grade-recovery plot in Figure 16 which also includes the results of the follow-up optimization tests and the production run.

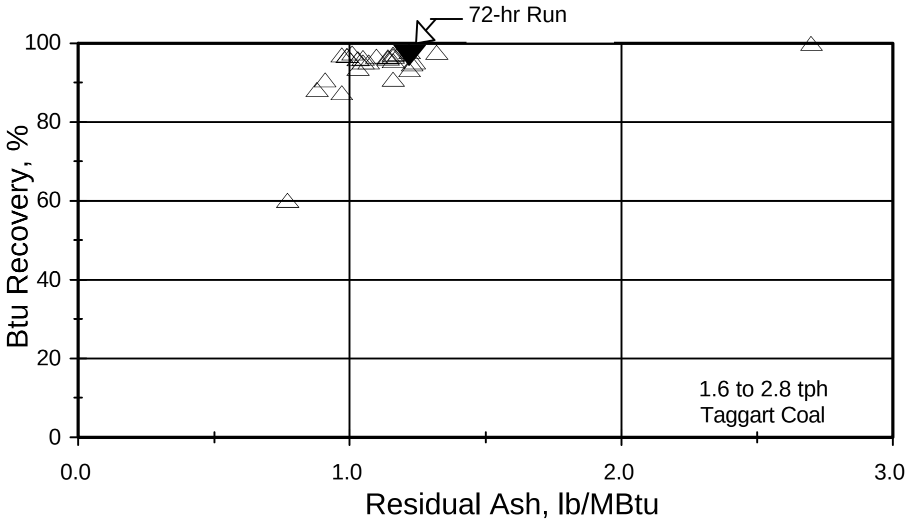


Figure 16. Grade-Recovery Curve for Flotation of Taggart Coal in PDU

Stepwise regression analyses procedures were applied to the test results to link output responses such as yield and clean coal quality to the input test variables such as feed rate, wash water rate, air rate, collector addition, and frother addition. Equations were developed which fit the data quite well and which could be used to indicate the relative importance of each independent variable in the response equation. In the case of the Taggart flotation, frother dosage was the most important variable affecting yield and residual ash in the clean coal [**R-21**].

The regression equations were used to determine optimum setpoints for achieving the 1 lb ash/MBtu and 80 percent Btu recovery. Seven tests were performed and the residual ash goal was achieved in two tests. Btu recovery for the two tests were 87.5 and 88.3 percent while the residual ash contents were 0.88 lb/MBtu and 0.97 lb/MBtu, respectively. The aforementioned results corresponded to a feed rate of 4,200 lb/h and a grind of D80=60 µm. A residual ash of 0.99 lb/MBtu at 96.7 percent Btu recovery had been achieved at the same feed rate during an earlier parametric test at a finer grind (D80=51 µm).

Pennsylvania State University requested shipment of 50 tons of the clean coal to them for future combustion testing. Because they planned to prepare CWF from the clean coal, they preferred a coarser particle size distribution than optimum for rejecting ash from Taggart coal. For this reason, and because of filtration limitations encountered while processing the Taggart coal, the production run was conducted at a coarser grind (D80=71 µm) and at a lower feed rate (3,800 lb/h) than preferred by the parametric and optimization testing. Aside from a failed belt splice, uninterrupted operation was achieved showing excellent reliability of the operation during the 72 hours. Overall, 137.7 tons were processed during the run for a weight yield of 95.3 percent, Btu recovery of 96.9 percent, and a clean coal residual ash of 1.22 lb/MBtu.

Flotation of Indiana VII Coal

Flotation of the Indiana VII coal in the PDU commenced during April 1996 and concluded during July. Though the residual ash goal of 2 lb ash/MBtu was difficult to achieve, the operation was considered to be quite successful [**R-20**].

All of the PDU test work was performed at a grind of D80=20-24 µm as indicated by laboratory release analysis testing. As described in Chapter 4, some modification of the Area 100 Grinding and Classification circuit was necessary in order to produce such a fine grind. Eventually, it was found that the desired clean coal quality of 2 lb ash/MBtu could be achieved in the PDU by using both ball mills and the fine grinding mill, 270 mesh screens and the 2-inch cyclones [**R-21**].

The residual ash goal of 2.0 lb/MBtu was achieved on five occasions during the parametric testing. Unfortunately, product yield and Btu recovery suffered significantly during these particular tests. Overall, the clean coal yield varied from 12.0 to 89.7 percent while the Btu recovery and residual ash contents varied from 13.2 to 96.4 percent and 1.81 to 3.25 lb/MBtu, respectively. The grade-recovery data are plotted in Figure 17 which also includes the results of the follow-up optimization tests and the production run.

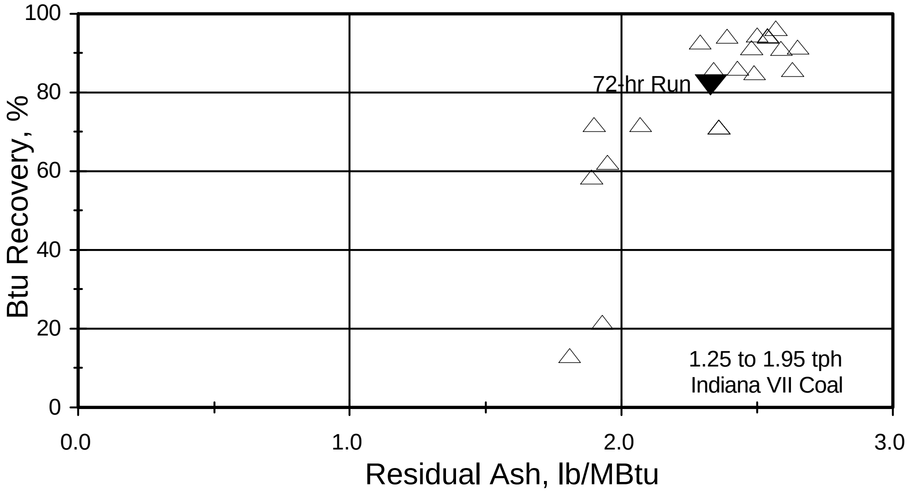


Figure 17. Grade-Recovery Curve for Flotation of Indiana VII Coal in PDU

As with the Taggart coal testing, stepwise regression analyses procedures were applied to the test results to link product yield and quality to operating variables. As before, the regression equations fit the data quite well. Most of the operating variables had significance impacts upon product yield. The most important of these were fuel oil dosage, particle size, and those variables which effected the bubble size coming from the aerator (aeration rate, frother dosage and recirculation rate). Aeration and recirculation rates and percent solids had strong effects upon the amount of residual ash in the clean coal.

Based on the regression analysis, four tests were performed to determine the optimum Microcel™ setpoints needed to achieve process development goals for the Indiana VII coal. Unfortunately, the product quality goal of 2.0 lb ash/MBtu was not achieved during these tests. It was suspected that a buildup of frother in the clarified water system resulted in the 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 surface of the water in the clarified water storage tank and also at spray water locations on the screens.

Somewhat less frother was used for the production run completed during July, 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, 77 tons were processed during the run for a weight yield of 75.2 percent, Btu recovery of 82.0 percent, and a clean coal residual ash of 2.33 lb/MBtu. The nominal feed rate was 3,200 pounds per hour.

Flotation of Hiawatha Coal

The operation of the PDU with Hiawatha coal was very successful with project goals achieved on numerous occasions [**R-21**].

Because the Hiawatha coal had not been evaluated during Subtask 4.4 (Hiawatha replaced Sunnyside), no data were available for indicating expected performance. For this reason, laboratory release analysis tests were performed on the coal. Two slurries, one having D80=54 µm and a second with D80=49 µm were evaluated. The residual ash specification and target Btu recovery were met at both particle size distributions. The D80=49 µm was targeted for the parametric testing, and the same grinding circuit configuration was employed as employed for the Taggart coal [**R-20**].

Overall, the clean coal yield varied during the parametric testing from 12.3 percent to 94.0 percent while Btu recovery and residual ash contents varied from 13.8 percent to 98.7 percent and 1.43 to 2.87 lb/MBtu, respectively. The grade-recovery data are plotted in Figure 18 which also includes the results of the follow-up optimization tests and the production run.

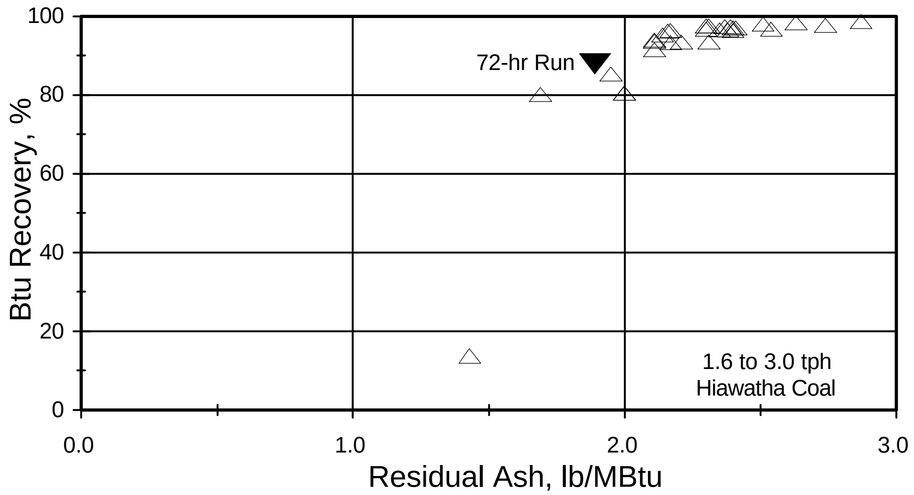


Figure 18. Grade-Recovery Curve for Flotation of Hiawatha Coal in PDU

Stepwise multiple regression analyses were also performed on the parametric test data again producing equations that fit the data quite well. The most important operating variables affecting residual ash were the recirculation rate and wash water usage. However, the operating variable that have the most significant impact on yield was the frother dosage.

Based upon the results of the regression analysis, eight optimization tests were performed and the residual ash goal of 2 lb/MBtu was achieved during two of the tests. A 72-hour extended production run on Hiawatha coal was successfully completed using the set points derived from the optimization testing. There were no interruptions during the run. Overall, 155 tons of coal were processed with a weight yield of 81.8 percent, Btu recovery of 88.0 percent, and a clean coal residual ash content of 1.89 lb/MBtu. Forty-four bulk bags of the clean coal were shipped to Pennsylvania State University for future combustion testing.

Microcel™ Scale-up Test Work

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 [**R-21**]. Test work performed on all three test coals ground in the PDU 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 larger (2 mm) than those in the 6-foot column (1 mm). Large bubbles typically result in a lower carrying capacity (low yield) and better selectivity (higher quality) than smaller bubbles. The difference appeared to be due to a lower recirculation velocity through the in-line mixer of the 12-inch unit than through the four in-line mixers of the 6-foot unit.

- The retention time of the 12-inch column was less than in the 6-foot column (9.0 minutes versus 12.4 minutes). This low retention time may have resulted in the rejection of middlings to the tailings stream from the smaller column. Had the retention time been longer the middlings might have reported to the clean coal stream increasing the yield and product ash.

Despite these differences, the carrying capacities of the two sizes of the Microcel™ column were comparable on a cross-sectional area basis when floating comparable coal slurries ground in the PDU [**R-21**]:

	Carrying Capacity, lb/hour/square foot		
Column	Taggart	Indiana VII	Hiawatha
12-inch	129	74	116
6-foot	127	86	125

Lessons Learned from Operation of Flotation Module

Based on the test work and operation of the PDU and Flotation Module, a number of lessons were learned which should be considered when planning similar endeavors in the future [**R-21**]. Some of the lessons which particularly apply to the use of column flotation for advanced physical fine coal cleaning are listed below:

- Grinding mills should be reviewed for proper loading and ball size.
- Grinding circuits should be designed to handle 40 to 50 percent solids coal slurries and include classification equipment for closed-circuit operation.
- Magnets should be installed to remove fragments of the grinding media from the mill discharge streams to avoid plugging lines and pumps.
- Clean coal product sumps should be located immediately adjacent to the flotation column and furnished with large-diameter feed pipes to catch and contain the large volume of froth overflowing the column.
- A simple ball float proved to be the most reliable method for locating the froth/slurry interface level. Continuous data acquisition should be considered with such a unit.
- A variable-speed recirculation pump was invaluable for proper operation of the Microcel™ column since it provided tremendous flexibility in varying bubble size and the operating position on the grade / recovery curve.

Conclusions of PDU Flotation

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 method for cleaning coal to high quality levels.

The capacity of the column depended upon the properties of the coal being processed and particularly upon the grind size required for liberation of the ash minerals. Frother dosage, aeration rate, wash water addition rate, and the recirculation rate though the in-line aerator were found to be significant operating variables impacting performance of the system. Performance goals could generally be met by adjusting these operating variables to reach product yield and quality specifications. Flotation performance scaled up from a 12-inch column to a 6-foot column in a consistent manner.

6. SELECTIVE AGGLOMERATION STUDIES

As discussed in Chapter 2, selective agglomeration was an effective method for rejecting mineral impurities from ground coal. The process consisted of mixing an oily bridging liquid into an aqueous coal slurry. The insoluble oil selectively coated coal particles, and during a series of high- and low-shear mixing steps the oil-coated particles clumped together into enlarged pellets which could be separated from the dispersed slurry of unoiled mineral particles by size separation (screening) or by froth flotation.

BRIDGING LIQUIDS AND ACTIVATORS

Normal pentane (n-pentane, C_5H_{12}) and n-heptane (C_7H_{16}) were employed as bridging liquids for most of the work as recommended by the Agglomerating Agent Selection Topical Report [**R-12**]. This recommendation was based upon the following criteria:

- Personnel and environmental safety
- Performance as an agglomerating agent for coal
- Ease of coal-water slurry preparation
- Cost

A number of light and heavier hydrocarbons were considered before making the recommendations, and a ranking procedure was followed which resulted in the selection of pentane and heptane as the agglomerating agents to be evaluated further for preparation of premium fuel. Cyclohexane ranked next on the list of candidates.

Usage cost was an important reason for favoring light volatile hydrocarbons such as pentane and heptane over heavier hydrocarbons such as fuel oil and kerosene since the light hydrocarbons can be conveniently stripped from the product with steam and reused. The main difference between n-pentane and n-heptane was their boiling points, 36º and 98º C (97º and 209º F), respectively.

The pentane and heptane tested were commercial products offered by various oil refineries. The n-pentane sold in bulk quantities for industrial use had a purity of about 98 percent and closely resembled the reagent grade. Several grades of heptane were sold for industrial use. One was “pure” grade containing about 98 percent n-heptane which sold for about $6.50/gallon. The less expensive grades were distillation products with boiling ranges near that of pure n-heptane. As a result, they were mixtures of C_7H_{16} isomers along with other C_6 and C_7 hydrocarbons, including up to 4 percent toluene and similar aromatics. Products were also offered which had been hydrotreated or hydrogenated to reduce their aromatic content. Aromatics were not wanted in the bridging liquid because of their odor, potential toxicity, and possible effect upon the efficiency of the agglomeration separation.

The selection report [**R-12**] suggested that diesel fuel and kerosene would be the preferred bridging liquids if the liquid was not to be recovered for reuse. Non-recovery systems may be appropriate when capital costs must be kept low such as the case for near-term applications where additional fine coal is to be recovered at an existing preparation plant.

In most cases light oils such as pentane and heptane displaced water effectively from coal surfaces and spread over the particles to begin the agglomeration sequence. However, the displacement and spreading were slow for oxidized coal and for lower rank coals that were less hydrophobic than the higher rank coals. Agglomeration "activators" or aids were added to the oil in such cases in order to shorten the phase inversion time. Activators contained polar compounds which reduced the interfacial tension between the oil and water and between the oil and the coal surface. The activators may also have absorbed onto the coal to present a more hydrophobic surface to aid spread of the bridging liquid. Ethylhexanol (more properly 2-ethyl-1-hexanol) was routinely used for this purpose during the liberation studies. Asphalt, which may also have acted as a binder, performed better for this purpose when agglomerating subbituminous coal. An anionic emulsion was a convenient method for adding asphalt during agglomeration since it could be added directly to the coal slurry instead of to the bridging liquid.

LABORATORY PROCESS OPTIMIZATION

The laboratory process optimization was to determine the operating conditions required to best meet the target premium fuel coal quality and Btu recovery specifications. An important objective was to compare the performance of selected bridging liquids, and especially pentane and heptane, so that one of the two might be selected for use during the bench-scale testing to develop design parameters for the PDU. A further objective was to compare the performance of an innovative unitized reactor design combining high- and low-shear mixing in one vessel with the usual plant design employing a series of separate vessels for each shear regime.

The key factors of interest during laboratory-scale selective agglomeration were the efficiency of the separation (recovery of the heating value in the coal and rejection of the ash) and the time required for formation of the agglomerates (inversion time). Inversion time depended upon a number of factors: coal and oil concentrations, particle size, oil properties, mixing conditions, and most importantly, upon how rapidly the bridging liquid spread across the coal particles. Oily bridging liquids readily spread on hydrophobic, high-rank coals so inversion occurred rapidly for most fresh bituminous coals. Inversion was slower for oxidized coals and for less hydrophobic, lower-rank coals such as the high volatile C and subbituminous classifications so an activator was used with these coals. The oil to coal ratio had some impact of upon the speed of the agglomeration, but the ratio had a greater impact upon the physical properties of the coal agglomerates which formed during inversion. Excess oil led to loose sticky masses which entrained impurities, while the agglomerates that formed with insufficient oil failed to grow into large enough pellets to be captured on a screen.

The laboratory work was conducted at Arcanum and Amax R&D and reported fully in a Topical Report [**R-13**]. Waring blender batch tests were done at both locations. The continuous unitized reactor was tested at Amax R&D and the conventional continuous two-stage mixer approach was tested at Arcanum. In most cases, the agglomerated coal was collected by washing the agglomerated coal on 48- or 100-mesh test sieves.

The five bituminous coals responded quite well to laboratory scale selective agglomeration with pentane and heptane bridging liquids. Target residual ash and Btu recovery specifications were easily met for the Taggart, Elkhorn No. 3, Sunnyside, Indiana VII and Winifrede coals ground to the fineness projected from the liberation studies. The target sulfur specification was met when cleaning the Taggart, Sunnyside and Indiana VII coals. The subbituminous Dietz coal did not respond as well and did not meet the target residual ash specification consistently. Heating value recoveries were consistently over 93 percent with heptane and pentane bridging liquids and commonly exceeded 98 percent from the Taggart, Indiana VII, Sunnyside and Elkhorn No. 3 coals.

The heptane to coal and pentane to coal bridging liquid ratios were approximately the same for good agglomeration with the two hydrocarbons. The ratios ranged from 0.18 gram hydrocarbon per gram coal on up to 0.36 gram per gram for the bituminous coals and 0.50 for the subbituminous coal. The more finely ground slurries, such as the Winifrede slurry, required more bridging liquid. Indiana VII slurry also required about 16 lb/st asphalt along with the bridging liquid in order to activate agglomeration. Ethylhexanol was a less effective activator than asphalt and was not needed for agglomerating Taggart, Elkhorn No. 3, Winifrede and Sunnyside coals.

Agglomeration proceeded satisfactorily in both of the continuous systems under a variety of operating conditions (percent solids, impeller speeds, feed rates, etc) until the feed rate was increased to the point where Btu recovery fell sharply and agglomeration ceased. The capacity of the units were at their maximum and expenditures for mixing energy were at their least when feed rates were just below the level where the recovery fell sharply. Changes in operating conditions had little impact upon the amount of residual ash left in the agglomerated clean coal.

The separation performances of the unitized reactor and the two-stage system were similar. The available data suggest, though, that the two-stage system required less high-shear mixing energy for agglomerating fine coal. Since the unitized reactor system did not offer any power-saving advantages, the two-stage system was recommended for the bench-scale testing because its development was further along and more scale-up experience was available for it from the prior DOE project performed by Arcanum and Bechtel. Minimum high-shear mixing energy consumptions in the two-stage system were in the 11.8 to 23.6 kWh/ton range when agglomerating minus 325 mesh bituminous coal.

Pentane, pure heptane, commercial heptane and dearomatized (hydrotreated) commercial heptane bridging liquids appeared to be equally capable of agglomerating the ground test coals while effectively rejecting ash minerals. Because of its low boiling temperature, the feed slurries required precooling when pentane was used as the bridging liquid. Dearomatized commercial heptane was the preferred bridging liquid because of its good performance, low cost, mild odor and low concentration of aromatic compounds.

Comparison tests were also made using diesel fuel and kerosene to agglomerate minus 325 mesh coal. Oil dosages as high as 10 to 30 percent of the coal weight were evaluated, but heating value recoveries never rose over 65 percent compared to the 95 plus percent recovery seen with heptane and pentane. Because of the quantities required, diesel fuel and kerosene were unlikely to be cost-effective bridging liquids for preparation of premium fuel. On the other hand, volatile hydrocarbons such as heptane and pentane were likely to be cost-effective bridging liquids since they can be stripped from the clean coal and reused.

The Dietz coal required addition of a considerable amount of asphalt to activate agglomeration, and acidification to pH 3 or 4 as well, before the high-shear step. Because of these added costs, agglomeration did not appear to be a cost-effective method for cleaning the coal. Therefore Dietz coal was dropped from further consideration during the selective agglomeration program.

BENCH-SCALE AGGLOMERATION AND STEAM STRIPPING

The bench-scale testing had three main objectives:

1. Design, construct, and operate a continuous selective agglomeration system of about 25 lb/h capacity to demonstrate the feasibility of the process.
2. Optimize process conditions to minimize product ash contents and to reduce process costs.
3. Generate reliable design data to insure successful scale-up of the process to the 2-t/h PDU scale.

A detailed account of the design and operation of the bench-scale system was provided in a topical report [**R-15**] which also presented the results of the testing.

Parametric testing was conducted on the five bituminous test coals plus the Hiawatha coal also selected for processing in the PDU. The coals had been ground to the appropriate particle size in the 4-ft ball mill with additional size reduction in the 40-liter stirred ball mill if need be. A few of the coals were also ground in the 2 t/h PDU grinding circuit during the column flotation PDU testing. Samples from selected parametric tests were analyzed to determine the distribution of the toxic trace elements during selective agglomeration (Chapter 7).

Bench-Scale System

The bench-scale system was assembled by Amax R&D from component designs based on the laboratory-scale testing results [**R-14**] and included provisions for recovering and reusing the heptane bridging liquid. The system was designed to produce 25 to 50 lb/h of product on a dry basis and represented a 3- to 10-fold scale-up of the laboratory equipment. Figure 19 is a block flow diagram of the system as it finally evolved. The mixing vessels, impellers and stripping columns were especially designed for this project and were fabricated locally. Although capable of operating together, the grinding, agglomeration, steam stripping, and final product dewatering steps were normally carried out separately.

The high-shear unit operation was designed to provide the following:

- Complete dispersion of the heptane agglomerant
- Sufficient heptane/coal contact to coat the coal with heptane
- Sufficient particle-to-particle contact, insuring formation of micro-agglomerates (phase inversion).

The high-shear vessel was 6 inches in diameter by 6 inches high. It was fully baffled and contained about 2.8 liters of slurry. This design was based on the requirements for the Indiana VII coal, the coal needing the longest residence time (2 to 3 minutes). The shearing in the high-shear vessel was provided by a single radial flow impeller located in the center of the vessel and driven by a 1-1/2 hp variable-speed motor. Impeller tip speeds evaluated were in the 5 to 18 m/s range. Heptane was added prior to the high-shear mixing. When necessary to promote agglomeration, SS1H asphalt emulsion was also added to the feed slurry.

The low-shear vessel was designed to provide a residence time of 5 minutes, insuring agglomerate growth to pellets of sufficient size for screening. The actual vessel was 8 inches in diameter and 18 inches tall and held about 18 liters. It was also fully baffled. A horizontal baffle divided the vessel into two low-shear mixing zones. Shearing was provided by two radial flow impellers on a single shaft, one located at the center of each mixing zone.

The agglomerated coal was recovered on a 10-inch x 16-inch inclined vibrating screen. The inclination of the screen could be adjusted and either a 48- or a 100-mesh screen deck was used. Spray water was applied to the screen to wash residual tailings slurry from the agglomerates. The tailings (screen underflow) discharged into a 4-inch diameter by 18-inch froth skimming column designed to collect any heptane-bearing carbonaceous material floating in the tailings stream.

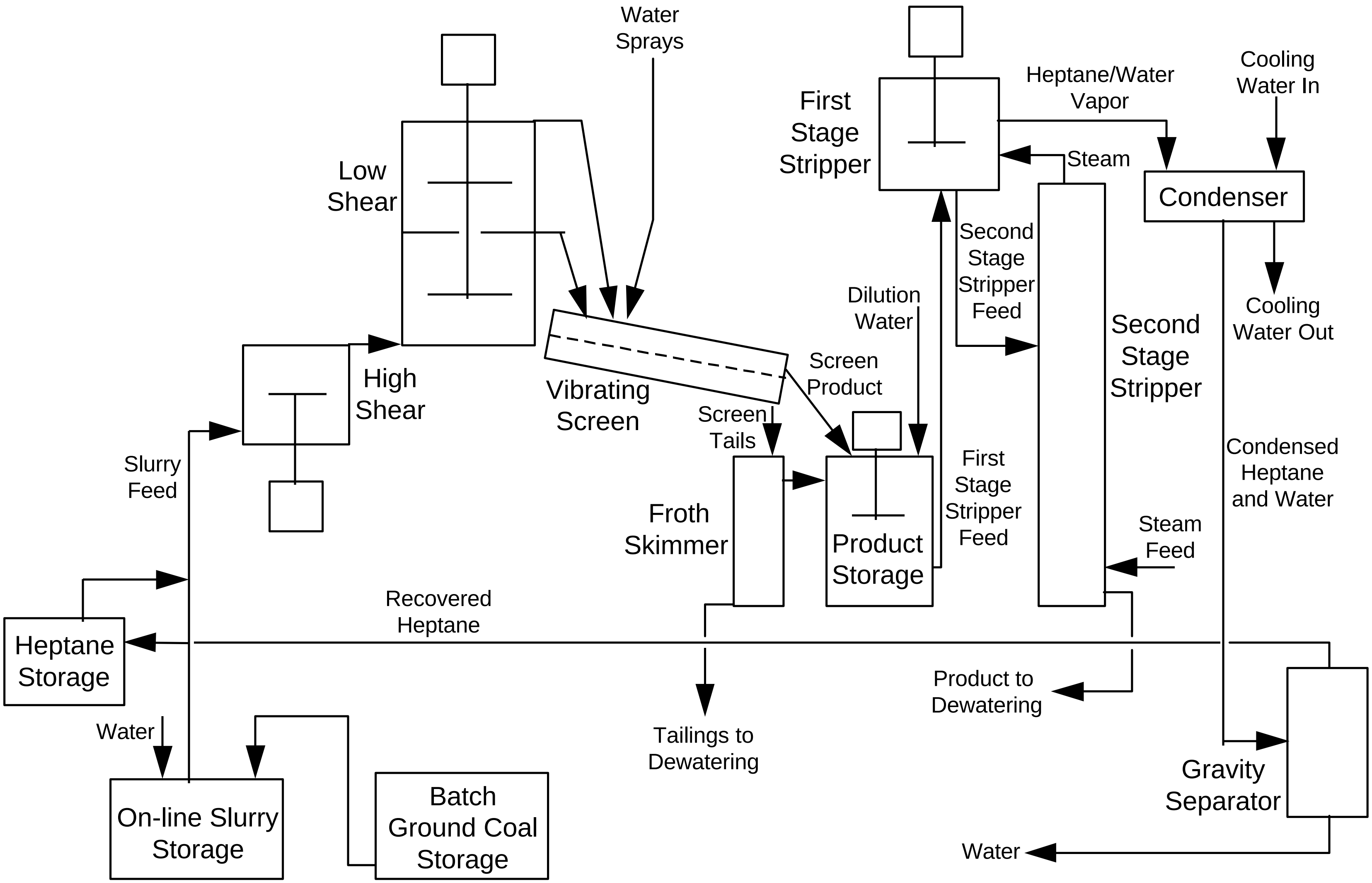


Figure 19. Bench-Scale Block Agglomeration Flow Diagram

Heptane was removed from the agglomerated product by direct steam stripping in one or two stages. A single stage of steam stripping was used during initial testing. The stripping vessel was a 4-inch diameter column approximately 52 inches tall. A portion of this column was filled with packing (5/8-inch stainless steel pall rings) to distribute flows and prevent excessive turbulence and back-mixing. Steam entered the column below the packing and the vapor (heptane and water) exited from the top at near-ambient pressure, while the agglomerated feed entered the top of the column and the product slurry discharged from the bottom through a U-tube for level control. This provided a steam flow counter-current to the process slurry flow.

The stripping circuit was modified later to include a new vessel which received the agglomerated coal ahead of the above described column. This allowed the bulk of the heptane to be removed in the new first-stage stripper followed by additional heptane removal in the original column, which was modified to operate at elevated pressures (to 15 psig) and temperatures. This two-stage circuit better simulated the developing 2-t/h PDU stripping circuit design which also used a counter-current steam flow scenario. Figure 20 is a diagram of the two-stage system.

The exiting vapor stream from the steam stripping circuit was condensed in a tube coil submersed in a water bath. The bath was serviced with sufficient tap water to cool the condensed heptane/water stream to about 38º C. Once condensed and cooled, this heptane/water mixture was separated by gravity in a column where the heptane (s.g.=0.7) overflowed from the top and the water from the bottom via a U-tube.

An on-line data acquisition system was included in the selective agglomeration test unit. This system allowed real-time data acquisition of operating temperatures and provided a data log into which manually obtained operating conditions were entered.

Agglomeration Reagents

The primary agglomerating reagent was the heptane added prior to the high-shear mixing. Both a commercial dearomatized grade heptane (about 28% n-heptane) and a pure grade heptane (>99% n-heptane) were utilized. Asphalt, in the form of an SS1H anionic emulsion, was also used at times since it was found to be an effective activator for agglomerating oxidized or difficult to agglomerate coals such as the hvC Indiana VII. No difference was seen between the performance of the two grades of heptane or between fresh and recycled heptane.

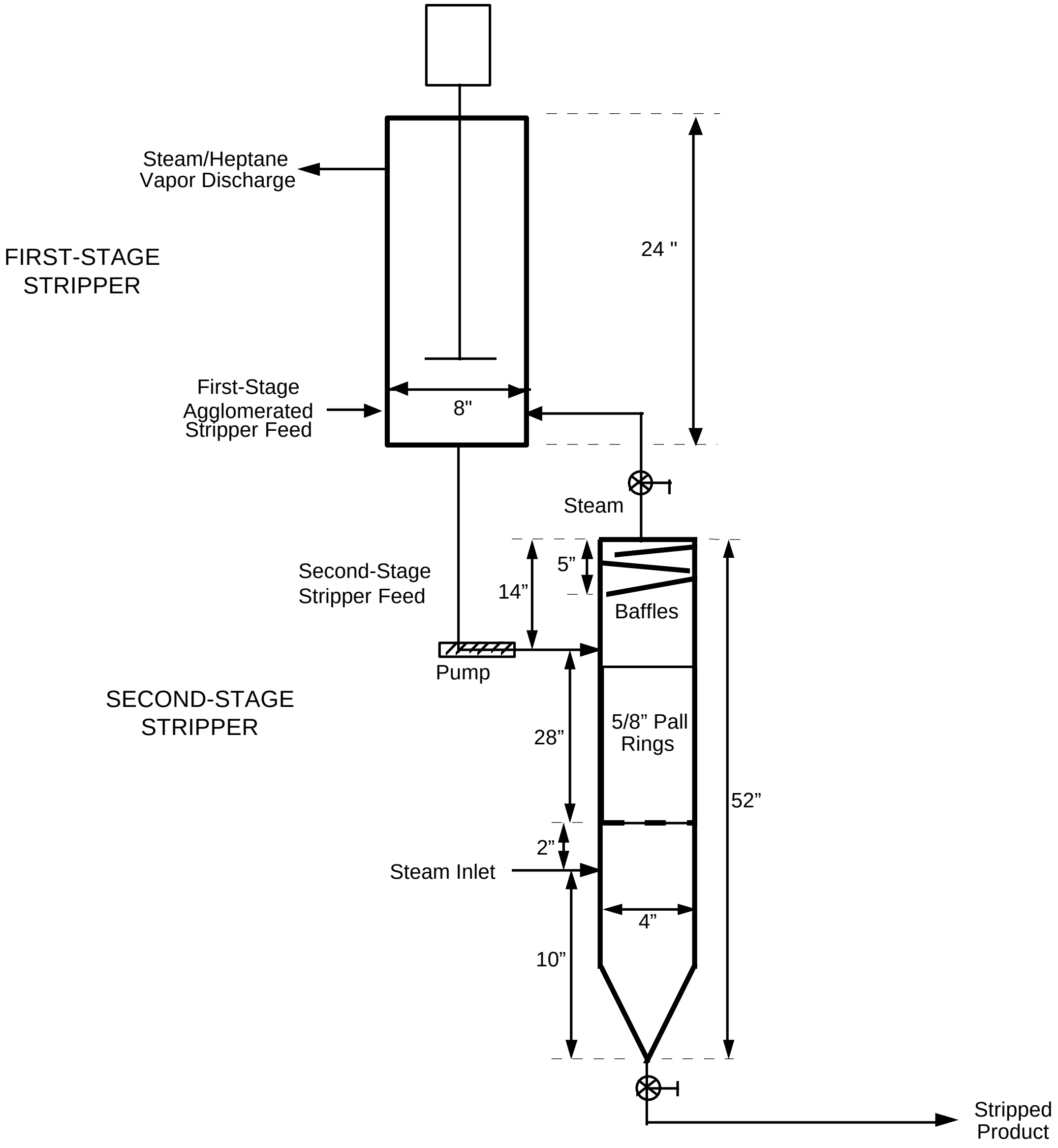


Figure 20. Details of Bench-Scale Steam Stripping Circuit

Bench-Scale Agglomeration Summary

The results of the bench-scale agglomeration test work indicated that the product ash specification of 1 to 2 lb/MBtu, as well as the Btu recovery goal of at least 80% on a run-of-mine coal basis, were met for all six of the coals tested. Of paramount importance in achieving these product ash levels was the particle size distribution to which the coal was ground. As for any physical coal cleaning process, if sufficient mineral-matter liberation were not achieved, the desired product grade could not be attained, except at the expense of significant heating value losses to the tailings stream. The coarsest particle size distribution to which each coal was ground while still achieving the product ash specifications are listed below. Typical product ash and Btu recovery values attained when operating at optimized conditions for the grind sizes shown are included in this tabulation:

	PSD, µm		Ash	Btu Recovery, %	
Coal	D80	MMD	lb/MBtu	Agglomeration	Run-of-Mine
Taggart	32.8	23.0	0.95	99.1	93.5
Sunnyside	59.6	34.3	1.79	98.3	88.6
Indiana VII	21.9	14.5	1.95	99.0	89.6
Elkhorn No. 3	68.0	39.4	1.69	96.8	91.6
Winifrede	12.4	7.1	1.91	99.2	88.8
Hiawatha	65.2	40.9	1.85	99.6	99.6

Various operating conditions were used to achieve these typical agglomeration results, i.e., several combinations of residence times, energy inputs, and heptane levels could ultimately achieve similar results. It was found however that the following guidelines should be followed to insure that consistent results are achieved:

1. Sufficient heptane must be used during high shear to achieve phase inversion and the formation of microagglomerates. Typically, the amount of heptane required increased with decreasing coal particle size and decreasing coal rank.
2. Sufficient energy or impeller tip speed (10 to 18 m/s) must be applied during high shear mixing to achieve complete dispersion of the heptane and enough particle-to-particle contact to form microagglomerates.
3. Sufficient residence time (typically 30 to 60 seconds) must be provided during high shear mixing to form microagglomerates (that is, to achieve phase inversion). Residence time requirements depended primarily on coal fineness and rank but were reduced by higher tip speeds.
4. The use of higher solids concentration during high-shear mixing reduced energy input requirements.
5. For lower rank and oxidized coals, an activator such as asphalt may be required to achieve phase inversion during high-shear mixing.
6. Sufficient heptane must be provided to allow agglomerate growth to the 2 to 3 mm size range during low-shear mixing. However, too much heptane resulted in poor agglomerate formation.
7. The production of consistent size and shape agglomerates in the 2 to 3 mm diameter range was paramount to achieving good recovery on the washing screen and low residual ash levels in the product. Figure 21 illustrates the relationship between residual ash content and the size of the agglomerates.

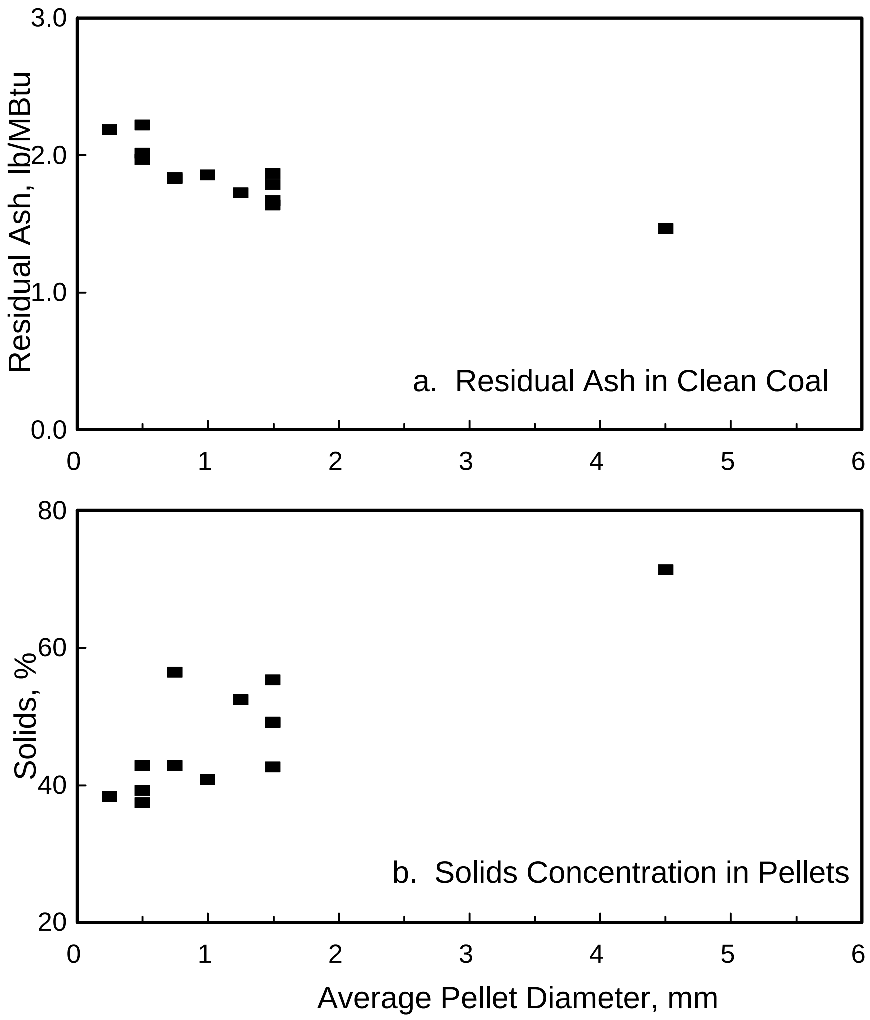


Figure 21. Effect of Agglomerate Growth During Low-Shear Mixing on the Residual Ash and Water Content of the Drained Agglomerates

8. Impeller tip speed (agitation intensity) during low shear mixing needed to be in vicinity of 5 m/s for the growth of well formed agglomerates of sufficient strength for recovery on the vibrating screen. Too mild or too intense agitation resulted in poorly formed agglomerates and higher residual ash contents.
9. Residence time during low-shear mixing was found to have little effect on agglomerate growth since ultimately, agglomerate formation was controlled by heptane dosage and low-shear agitation intensity. However, residence times no greater than 2 to 3 minutes were recommended since agglomerate growth was very difficult to control over longer residence times.
10. The discharge of the low-shear mixing vessel should be at the same elevation as the impeller. This configuration insured that continual

discharge occurred under all operating conditions of the low shear mixer.

11. Higher solids concentrations during low-shear mixing resulted in higher residual ash levels in the products and made agglomerate growth difficult to control.
12. The vibrating screen used for agglomerate recovery must have sufficient forward linear motion to transport the agglomerates across the screen deck.
13. Screen spray water was required to wash the mineral-matter bearing process water off of the agglomerated coal.
14. A froth skimmer on the screen underflow recovered any clean coal that passed through the screen.

Overall, the selective agglomeration process was very robust. It either worked well or did not work at all. Therefore, as long as the coal grinding provided sufficient mineral-matter liberation and a consistent low-shear product of the appropriate pellet size was produced, the desired product grade was achieved along with consistently high Btu recovery.

Heptane Recovery

In an effort to better quantify the residual heptane concentrations remaining with a stripped agglomerated product, a number of batch stripper tests were carried out. Several different types of tests were completed using commercial and pure grades of heptane:

- Boiling of recovered agglomerates in an open stirred beaker for a set period of time at 94º C.
- Thermal drying at 110º C for a set period of time.
- Autoclave treatment at 115 to 120º C for a set period of time.

The following is a summary of these initial batch stripper test results:

- Lower residual hydrocarbon concentrations were achieved as the stripping residence time increased.
- Thermal drying achieved much lower residual heptane levels than boiling. This is believed to be due to a combination of longer residence times, higher temperatures, and removal of virtually all of the water present.
- Storage of the clean coal for 2 days prior to stripping resulted in higher residual hydrocarbon concentrations than immediate stripping.
- Presence of asphalt in the stripper feed resulted in lower residual hydrocarbon concentrations in the stripper product.
- Stripping at increased temperatures and pressures resulted in reduced residual hydrocarbon concentrations.

Stripper Testing

The continuous steam stripping was usually carried out independently of the agglomeration circuit. Agglomerates were diluted to 10 to 15 percent solids in a well mixed feed tank. When operating the two-stage stripper circuit, the feed to the system was delivered intermittently to avoid the plugging that occurred at lower feed rates. As such, the feed was cycled on and off as required to maintain a first-stage stripper temperature between 90 and 95º C. Feed to the second-stage steam stripper was set at a fixed volumetric flow rate to control the combined system throughput. The pressure and temperature in the second-stage stripper were controlled manually to maintain a target operating pressure and operating temperature, typically 10 psig and 117º C, respectively, by adjusting the slurry and steam flows. The slurry level in the second-stage stripper was also controlled via a manual discharge valve. The initial testing with the single-stage stripping system indicated that the column could handle a coal feed rate approaching 20 lb/h without plugging.

Heptane Analyses

The residual concentrations of heptane in the stripper products were determined by gas chromatography of a methylene chloride extract using procedures developed with the assistance of Huffman Laboratories of Golden, Colorado. The moisture in slurry and solid samples was fixed with anhydrous sodium sulfate during the extraction procedure so that water would not enter the chromatography column. The actual residual analyses were for n-heptane only. An appropriate factor was used to convert the n-heptane to total hydrocarbon concentrations when commercial grades of heptane were used for the agglomeration. The factor was derived from detailed gas chromatography/mass spectroscopy analyses done by Phoenix Laboratory on the commercial heptane and on the residual hydrocarbons remaining in selected slurries and filter cakes.

Bench-Scale Steam Stripping Summary

In general, steam stripping to remove heptane from agglomerated products was a straight forward operation. The steam was applied directly to the reslurried agglomerates to evaporate the heptane, and the ratio of heptane to water in the exiting vapor phase from the stripping circuit was minimized to insure that steam consumption was kept as low as possible.

A two-stage system should be used due to the advantage of carrying out steam stripping at elevated pressures and temperatures (lower residual heptane concentrations). In this scenario, the first-stage stripper was agitated to keep the buoyant agglomerates dispersed and operated at only a little above atmospheric pressure to facilitate pumping the agglomerated coal slurry. The evaporation of most of the heptane in the first-stage disintegrated the agglomerates, so slurry could then be pumped more easily through the higher pressure and temperature second-stage stripper. The second stage was a plug-flow packed column for better mass transfer efficiency that removed additional heptane from the coal. A counter-current steam flow was used with fresh steam feeding the second stripper and the vapor from the second stripper feeding the first stripper. The first-stage stripper vapor was condensed and the water and heptane separated by gravity.

Continuous two-stage stripper testing was carried out at operating temperatures of approximately 92º C and 115º C in the first- and second-stage strippers, respectively. Residence times were 5 and 10 minutes for the first- and second-stages, respectively. Under these conditions, residual hydrocarbon concentrations on the order of 1,000 to 3,000 ppm (0.1 to 0.3 percent) on a dry coal basis were achieved in the final product. These residual concentrations appeared to be independent of the coal tested and the type of heptane used, that is, either pure grade or commercial grade.

The effects of various steam stripping operating variables on the residual heptane content of the stripped products are summarized as follows:

1. No benefits were gained by providing residence times greater than five minutes when steam stripping heptane from agglomerated products at ambient-pressure boiling temperatures. (Ambient pressure at Golden is about 12.1 psia.)
2. Steam stripping at elevated temperatures, as achieved by increased operating pressures, resulted in lower residual heptane concentrations.
3. Two stages of steam stripping achieved lower trace heptane concentrations than a single stage of steam stripping due to the increased temperature in the second stage.
4. No benefits were gained by use of large flows of excess steam.
5. Increasing the solids concentration at which the steam stripping was carried out had no detrimental effect on residual heptane concentrations.
6. The presence of asphalt (used as an activator during agglomeration) resulted in lower residual hydrocarbon concentrations under otherwise similar stripping conditions.
7. Regardless of whether a commercial or pure grade of heptane was used during agglomeration, total residual hydrocarbon concentrations were similar.

Beyond the problems faced while feeding agglomerated coal to the stripping circuit, no major operational difficulties were encountered during the bench-scale stripper testing. Vapor condensation and liquid cooling were achieved in a tube coil submersed in a water bath serviced by utility water. Complete condensation was consistently achieved with minimal carryover of coal from the stripping circuit. Separation of the condensed water and heptane was easily accomplished in a gravity separator column with the heptane overflowing from the top and the water exiting the bottom. This separation was complete with only minimal solubility (<10 ppm) of heptane into the water phase.

Tailings Heptane Analysis

A set of agglomeration tailings samples (froth skimmer underflow) was analyzed for residual heptane content in order to plan the design of tailings disposal system for the PDU and a commercial plant. The samples were from an Elkhorn No. 3 coal agglomeration test and the tailings contained about 50 percent ash on a dry solids basis. Samples submitted included as-produced tailings, tailings filter cake, tailings filtrate, and tailings samples that had been boiled for 5, 10, and 20 minutes. No more than 10 ppm of n-heptane was detected in any of the samples, except for the filter cake, which contained 567 ppm heptane on a dry solids basis. There was less than 1 ppm of heptane detected in the tailings filtrate. These results indicated that tailings disposal in conventional waste disposal sites should not be a problem.

PDU OPERATIONS

The conceptual design of the Agglomeration Module for the PDU was a collaborative effort between Bechtel, Entech and Arcanum. Much of the early effort was devoted to consider the kind of bridging liquid to be used, the type and scale-up of the high-shear and low-shear mixing vessels and the flowsheet for the bridging liquid recovery operation. [**R-17, R-23**]. Once these issues were resolved, the detailed design of the Agglomeration Module was performed by Bechtel Corporation with support from Arcanum and Entech Global engineers. The detailed design [**R-18**] was for a plant that would fit into the limited space remaining in the building at the Amax R&D Center which housed the PDU and Flotation Module already in place. Fortunately some of the larger components could remain outside of the main building.

The construction contract for the Agglomeration Module was awarded to Mech El Inc of Aurora, Colorado. Construction began on March 11, 1996 and was completed in November 1996. Start-up and shake-down proceeded according to a test plan [**R-22**] approved by DOE project management and was completed in time for the parametric testing to begin in January 1997.

Process and Plant Description

As described in the detailed design [**R-18**] and topical reports [**R-22**, **R-23**], the Area 300 Selective Agglomeration Module contained several areas which will be described separately:

- Area 300 - Agglomeration
- Area 300 - Steam Stripping
- Area 300 - Heptane Recovery and Recycle

In addition to these specific areas, the module included a nitrogen gas blanket system, equipped with oxygen sensors to contain the heptane vapor and prevent formation of potentially explosive mixtures of heptane vapor and air. The module also contained provisions for supplying steam and chilled water to the components. Hydrocarbon vapor detectors and alarms were installed in the rooms in and around the agglomeration areas. All of these auxiliary parts of the Selective Agglomeration Module are described in the Test Plan [**R-22**] and Topical Report for the task [**R-23**]. The equipment in Area 300 was sized for production of 2.0 st/h of Sunnyside clean coal. The PDU could process Taggart coal to produce 1 lb/MBtu ash at the same capacity but had a lower capacity for Indiana VII coal.

The ground coal slurry for selective agglomeration was produced by the existing Area 100 Grinding and Classification circuit described in Chapters 4 and 5 and the clean coal and tailings were dewatered in the existing Area 400 thickener and filters. Since the existing clarified water system also had to be modified for the selective agglomeration, start-up of the Selective Agglomeration Module could not begin until the column flotation testing was finished.

As with the column flotation, all areas of the PDU were operated from a central control room utilizing the integrated graphic data-logging and control system furnished by Honeywell. Generally there were one control room operator and three area operators working on each shift. The operations were supervised by an engineer and supported by two technicians performing analytical duties.

Area 300 - Agglomeration

As shown in Figure 22, the ground coal was stored temporarily in one of two tanks before being mixed with heptane and proceeding to agglomeration. There were two variable-speed high-shear mixing vessels for beginning agglomeration. One had a volume of 35 gallons and the other 75 gallons. They were arranged so that they could be used individually, in parallel or in series, in order to provide the requisite high-shear retention time to achieve inversion. From there the slurry proceeded to a 400-gallon low-shear mixing tank and on to 48-mesh inclined vibrating screen where the agglomerates were collected and rinsed with spray water (Figure 23). The fine refuse tailings passing through the screen deck flowed to a froth skimming tank before being pumped to the Enviro-Clear thickener in Area 400. Any coal which passed through the screen deck floated to the surface of the slurry and was collected by the skimmer.

Area 300 - Steam Stripping

A process flow diagram of the steam stripping part of Area 300 is shown in Figure 23. The circuit generally followed the two-stage system studied during the bench-scale testing. In this part of the module, the agglomerated coal from the screen and the froth from the froth skimmer were mixed with preheated water and pumped to Stripper A where the mixture was heated by the hot vapor leaving Stripper B. The hot slurry from Stripper A in turn was pumped to Stripper B where it was further heated by injection of 25 psig steam. The stripped slurry leaving Stripper B was pumped though a heat exchanger to the Area 400 clean coal filters.

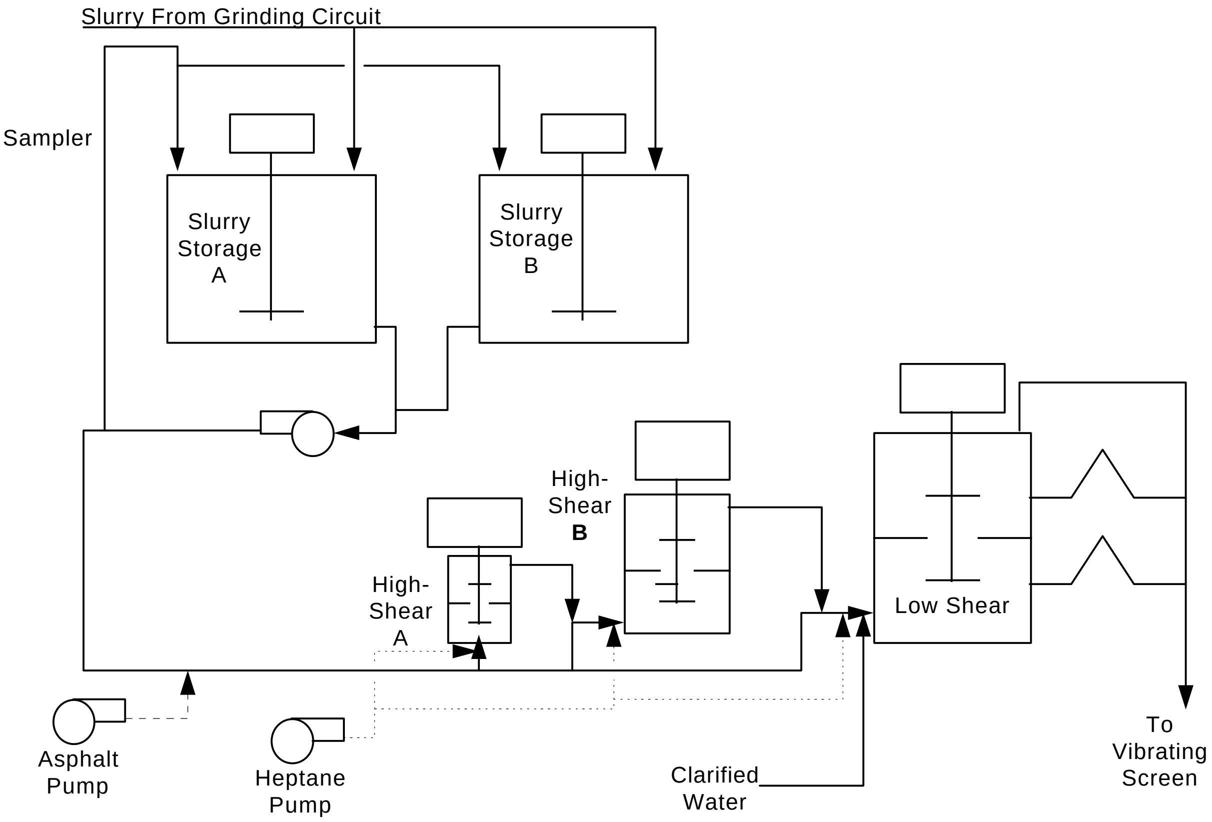


Figure 22. PDU Area 300 - Agglomeration

Area 300 - Heptane Recovery and Recycle

The heptane in the vapor exiting the top of Stripper A was condensed in an air cooler for reuse in the circuit as shown in Figure 24. The water in the condensate was removed by a gravity separator and passed through a carbon filter so that it could either be reused or discarded into the local sewer system.

Selective Agglomeration Module Operation and Test Work

The operation and testing of the Flotation Module was in accordance with the test plan [**R-22**] approved by the DOE. Beginning with Hiawatha coal, the basic strategy was to conduct a planned series of 3 sets of parametric tests, one set to evaluate agglomeration, one set to evaluate agglomerate recovery (screening and froth skimming), and a final set to evaluate the stripping circuit. These would be followed by optimization tests and completed with a 72-hour production run on each coal.

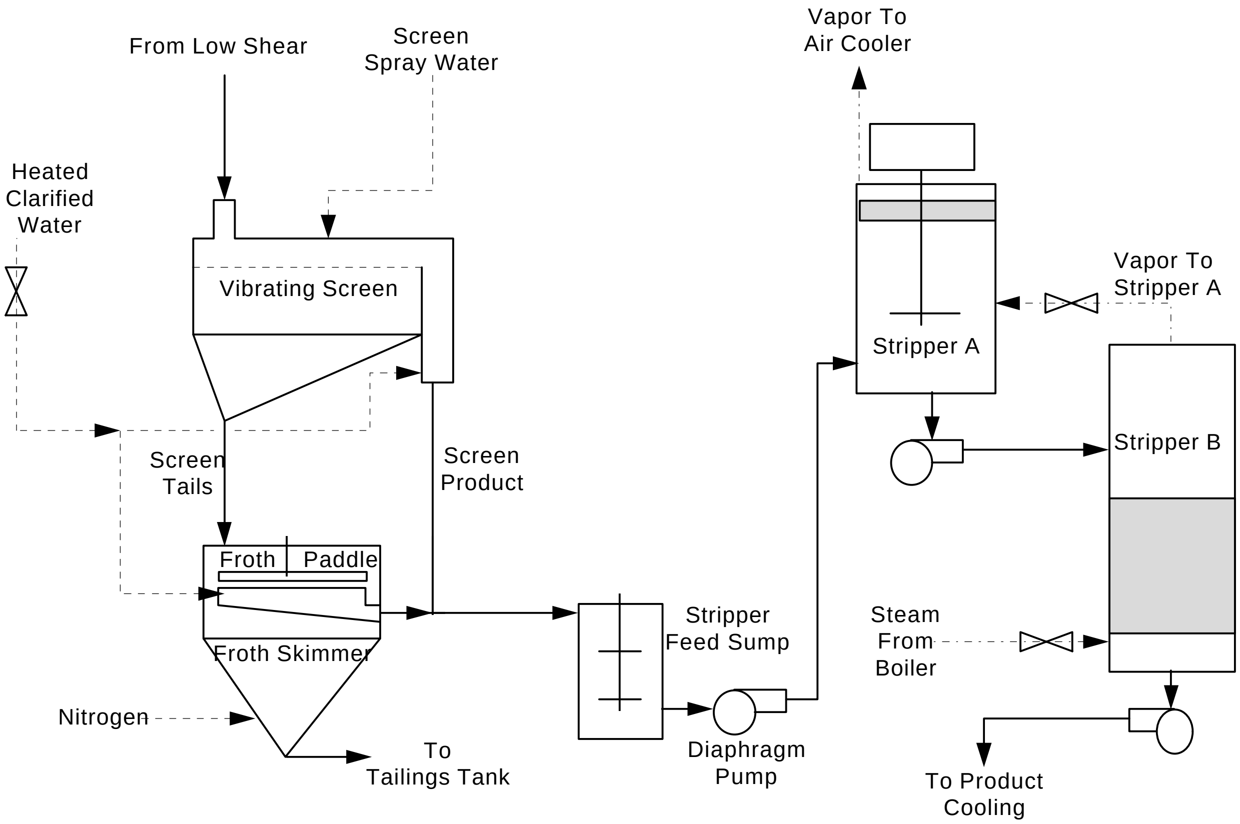


Figure 23. PDU Area 300 - Screening and Steam Stripping

The test work began during January 1997 with Hiawatha coal and was finished with Indiana VII coal during July 1997. About 1,000 tons of coal were processed through the PDU during operation of the Selective Agglomeration Module.

Agglomeration of Hiawatha Coal

A considerable effort was devoted to the establishment of proper operating conditions in the high-shear and low-shear mixers [**R-23**]. The parameters which were studied included heptane/coal ratio, impeller tip speeds and retention times. Good results were generally obtained with heptane/coal ratios between 0.24 and 0.30 to one for Hiawatha coal ground to D80=40 µm. Good agglomeration was achieved at retention times between 40 and 80 seconds at tip speeds of 11 to 15 m/s in the high-shear mixer. Some instability was noted under some operating conditions in the low-shear mixer while attempting to grow 2- to 5-mm agglomerates. The most consistent operation appeared to be with a 5 m/s tip speed.

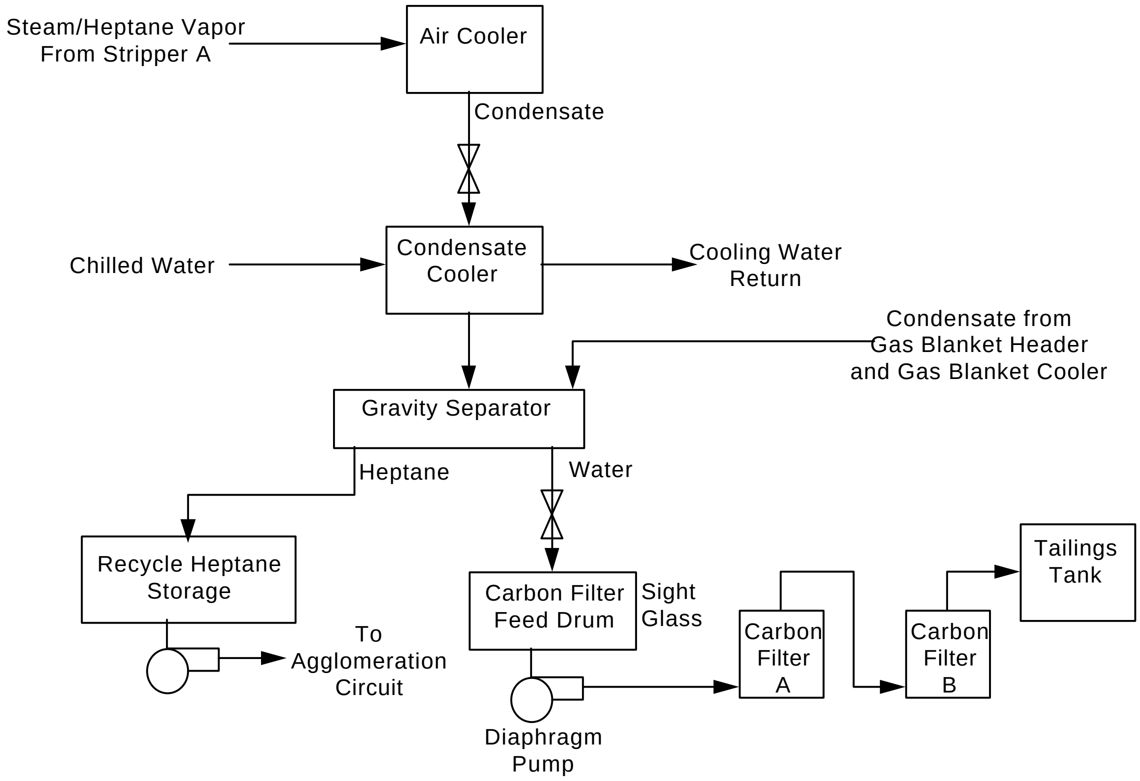


Figure 24. PDU Area 300 - Heptane Recovery and Recycle

Combinations of retention times and temperatures were investigated for the two stripping stages. Relatively low residual heptane concentrations were reached consistently. Typical values were in the 2,000 to 3,000 ppm range, and no clear trend was seen relating temperature and retentions to lower residual heptane concentrations. The heptane was recovered from the vapor leaving Stripper A without difficulty.

The variable most effecting performance was the particle size distribution. This is illustrated in Figure 25 where all of the residual ash values from the parametric, optimization and production runs are plotted against the D80 particle size of the feed slurry. A D80 of less than 40 µm appears to be essential for meeting a 2 lb/MBtu ash specification. Btu recoveries were consistently over 98 percent during these tests.

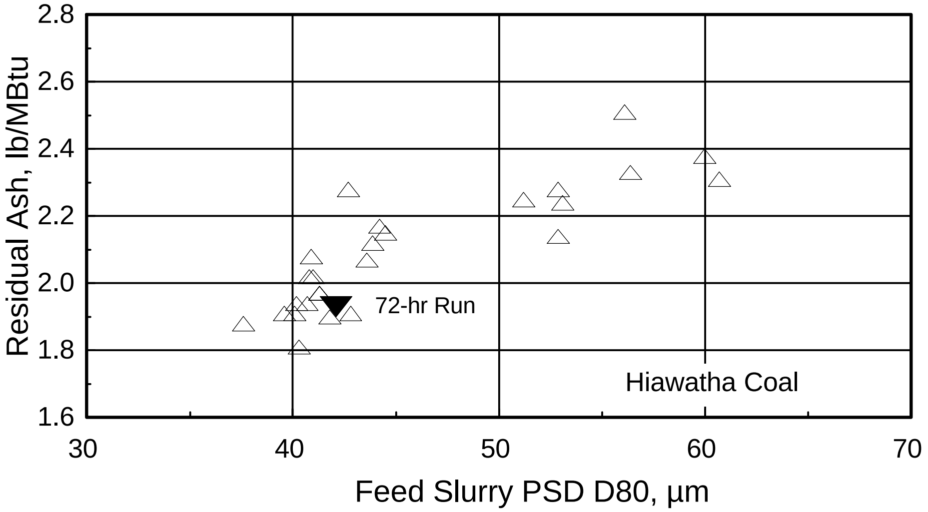


Figure 25. Residual Ash in Agglomerated Hiawatha Coal

The Hiawatha production run was completed on April 17, 1997 and successfully met expectations. The average feed rate was 3,839 lb/h (dry basis) and the grind was D80=42 µm. There were two periods of downtime due to a pump failure. Overall, 106.5 tons were processed during the run for a clean coal yield of 92.8 weight percent and the following operating results:

• Heptane/Coal Ratio	- 0.30 to 1
• Mixing Energy	- 17 kWh/st feed coal
• Steam Consumption	- 1,320 lb/st clean coal
• Residual Heptane:	
Clean coal	- 2,951 ppm dry coal basis
Tailings	- 1,470 ppm total solids basis
• Residual Ash	- 1.93 lb/MBtu (2.78 percent)
• Residual Sulfur	- 0.35 lb/MBtu (0.50 percent)
• Btu Recovery	- 98.9 percent

Sixty bulk bags of cleaned Hiawatha coal were shipped to Pennsylvania State University for future combustion testing.

Agglomeration of Taggart Coal

A similar set of parametric tests were conducted on the ground Taggart coal, and the relationship between operating variables and performance seen for agglomeration and stripping were similar to the relationships seen during the Hiawatha testing [**R-23**].

Again, the variable most effecting performance was the particle size distribution. Some effort was devoted toward identifying the D80 value that would allow production of clean coal containing less than 1.0 lb of ash per million Btu. Apparently this was something less than D80=30 µm in the case of the Steer Branch Taggart coal as shown in Figure 26 where all of the residual ash values from the various tests are plotted against the D80 particle size of the feed slurry. Btu recoveries were consistently over 97 percent during these tests.

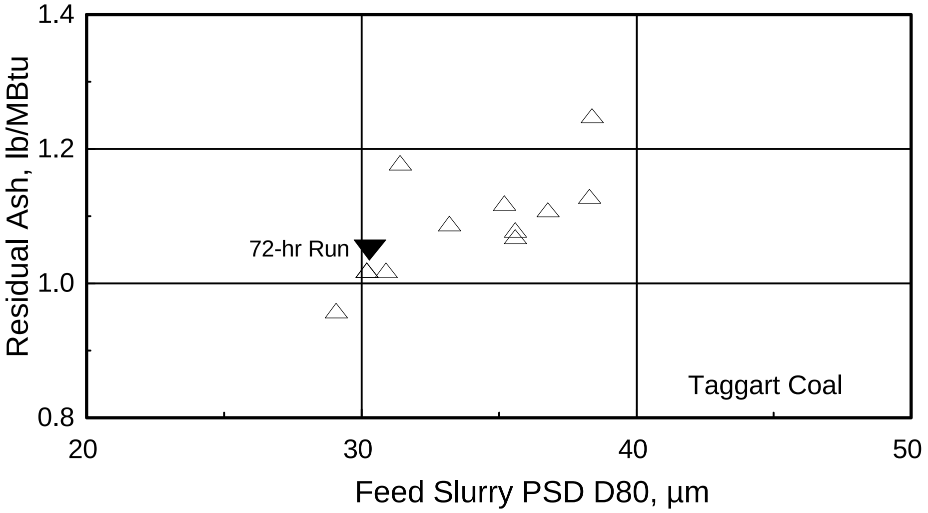


Figure 26. Residual Ash in Agglomerated Taggart Coal

The Taggart production run was completed successfully on May 22, 1997. It was conducted at an average feed rate of 3,305 lb/h (dry basis) and a grind of D80=30 µm. There was a short downtime due to a pump failure. Overall, 115.7 tons were processed during the run for a clean coal yield of 96.7 weight percent and the following operating results:

• Heptane/Coal Ratio	- 0.39 to 1
• Mixing Energy	- 16.1 kWh/st feed coal
• Steam Consumption	- 1,553 lb/st clean coal
• Residual Heptane:	
Clean coal	- 5,115 ppm dry coal basis
Tailings	- 4,094 ppm total solids basis
• Residual Ash	- 1.06 lb/MBtu (1.59 percent)
• Residual Sulfur	- 0.42 lb/MBtu (0.63 percent)
• Btu Recovery	- 99.2 percent

Forty bulk bags of cleaned Taggart coal were shipped to Pennsylvania State University for future combustion testing.

Agglomeration of Indiana VII Coal

A similar set of parametric tests were conducted on the ground Indiana VII coal as conducted on the two previous coals, and the relationship between operating variables and performance seen for agglomeration and stripping were similar to the relationships seen before [**R-23**]. However, there was one difference. Between 5 and 10 lbs of asphalt in the form of an emulsion were required to activate agglomeration of the Indiana VII coal because of its lower rank.

The Indiana VII coal required very fine grinding (D80=20 µm) in order to reach a residual ash content of less than 2.0 lb/MBtu in the clean coal as shown in Figure 27. Despite the fine grinding, Btu recoveries consistently exceeded 99 percent and the fine refuse tailings ordinarily contained between 88 to 91 percent ash.

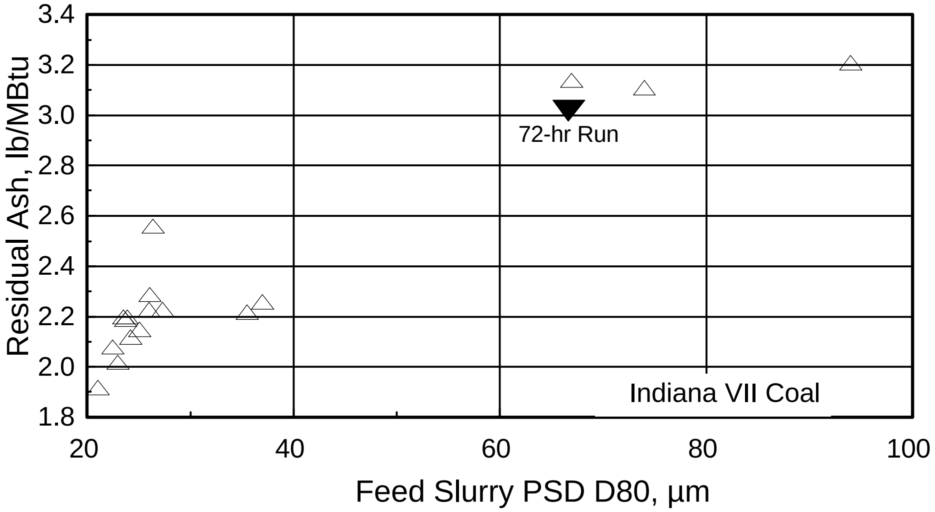


Figure 27. Residual Ash in Agglomerated Indiana VII Coal

Poor filter performance was an unwanted side effect of the fine grinding required for the Indiana VII coal. Since there was not enough storage capacity in the PDU for the clean coal coming from the stripper over very many hours, it would not have been possible to operate the agglomeration circuit for anywhere near 72 hours at one time. For this reason the decision was made to operate the PDU at a coarser grind so that the reliability and robustness of the selective agglomeration process could be demonstrated over a longer running time. This was done for the Indiana VII production run that was completed on July 31, 1997. The run was conducted at an average feed rate of 3,491 lb/h (dry basis) and a grind of D80=67 µm. There were two short downtimes due to the failure of a tailings filter and a control valve. Overall, 113.5 tons were processed during the run for a clean coal yield of 93.5 weight percent and the following operating results:

- Heptane/Coal Ratio - 0.35 to 1
- Mixing Energy - 36.6 kWh/st feed coal
- Steam Consumption - 1,778 lb/st clean coal
- Residual Heptane:
    Clean coal - 3,967 ppm dry coal basis
    Tailings - 472 ppm total solids basis
- Residual Ash - 3.02 lb/MBtu (4.19 percent)
- Residual Sulfur - 0.31 lb/MBtu (0.43 percent)
- Btu Recovery - 99.9+ percent

The exceptionally high Btu recovery from Indiana VII coal was a result of the asphalt added to activate agglomeration of coal and also to the residual heptane in the clean coal. Ten bulk bags of cleaned Indiana VII coal were shipped to Pennsylvania State University for future combustion testing.

Discussion of Selective Agglomeration Module Operating Results

In general, it was found that changes in most operating variables had only small effects on the amount of residual ash in the clean coal. However, it was important that the coal be ground fine enough for liberation of the ash minerals since the particle size distribution had far more effect on residual ash than any other variable. Generally, growth of the agglomerates to the 2- to 5-mm range provided the best operation. Similarly, once set within the proper range for inversion, agglomerate growth and screening efficiency, small changes in operating variables such as heptane dosage, shear rates, retention times and spray water usage had little effect upon the percentage recovery of coal. Under these conditions product recovery was very good, typically over 98 percent Btu recovery.

Asphalt was required to activate agglomeration of the Indiana VII coal, which was of high volatile C rank rather than the high volatile A rank of the other two coals tested in the PDU. An excessive amount of asphalt (perhaps over 10 lb/st) tended to result in slightly larger amounts of residual ash in the clean coal. Despite activation with asphalt, the Indiana VII coal still required a longer retention time in the high-shear mixing vessel than the Hiawatha and Taggart coals.

The stripping and heptane recovery system was equally robust in its operation. About 99 percent of the heptane was stripped from the coal, and there was no loss of performance or change in the operation when the condensed heptane was reused for agglomerating ground coal. The main operating problems encountered with the system were episodes of carry-over of coal into the condensation system. These episodes became far less frequent as the operators became more experienced handling the equipment.

The design of the Selective Agglomeration Module provided a good work environment. All of the flows were fully contained so there was no odor from the heptane or high humidity from escaping steam. Neither was there any noticeable odor from the clean coal and fine refuse filter cakes. As seen while operating the Flotation Module, the Area 400 product dewatering step, especially clean coal filtration, was the leading bottleneck restricting production of clean coal.

DISPOSITION OF COAL AND PDU

As provided in the contract between the DOE and Amax, the entire PDU was dismantled upon completion of the selective agglomeration task and the equipment packed and shipped to FETC Pittsburgh [**R-24**]. The refuse and left-over clean coal from the operation was consigned to a land-fill for disposal.

7. REJECTION OF TOXIC TRACE ELEMENTS

In response to provisions of the 1990 Clean Air Act Amendments (CAAA), the reduction in the concentration of specified toxic trace elements was monitored during the bench-scale and PDU advanced cleaning. Samples from representative tests conducted in each circuit were analyzed to determine the relative effectiveness of advanced cleaning for reducing the amounts of 12 trace elements found in run-of-mine (ROM) and washed coal. The elements of interest were antimony, arsenic, beryllium, cadmium, chromium, cobalt, lead, manganese, mercury, nickel, selenium and chlorine which could become hazardous air pollutants during combustion.

TRACE-ELEMENT ANALYSES

The trace-element analyses were conducted on solutions generated by perchloric acid decomposition of the samples. Mercury was determined by cold-vapor spectroscopy, chlorine by total halides coulometry, and the remaining ten elements by ICP spectroscopy. The analyses are tabulated in Appendix Tables A-4 and A-5. Additional results and mass balances are contained in the topical reports for bench-scale flotation and selective agglomeration cleaning [**R-9**, **R-16**] and in the topical reports for the PDU operations [**R-21**, **R-23**]. Cadmium was detected in only a few of the ROM and test coals. It was detected in one of the clean coals, though, and in a number of the fine refuse samples. Lead was not detected (< 2 ppm) in any of the Sunnyside or Hiawatha samples. Except for cadmium and for instances where elements were not detected in the clean coals, product mass balances for the trace elements were usually within 20 percent of the amounts indicated by the ground coal feed analyses. One really cannot expect any better mass balance closures since many of the trace element analyses are reported to only one or two significant figures at best.

The amount of trace elements found in the samples varied from coal to coal, and the residual amounts in the clean coals after cleaning were dependent upon the source coal. Generally, when prepared from the same test coal, the advanced flotation clean coal and the selective agglomeration clean coal contained about the same amounts of the trace elements.

The residual mercury and selenium analyses were of particular interest. Mercury was not detected (< 0.01 ppm) in some clean coal samples (Sunnyside, Indiana VII and Wentz Mine Taggart) and ranged up to 0.03 ppm in others (Winifrede coal cleaned by flotation). The fine refuse samples contained between 0.01 and 0.12 ppm mercury. Selenium analyses ranged from 0.41 ppm up to 5.7 ppm in the clean coals and from 0.30 ppm up to 7.1 ppm in the various fine refuse samples. Further details are provided in Appendix Tables A-4 and A-5.

RESULTS OF ADVANCED CLEANING

The reductions in the concentrations of the impurity ash, sulfur and trace elements on a heating value (lb/MBtu) basis from the amounts in the test coals and ROM parent coals were included in the topical reports [**R-9**, **R-16**, **R-21**, **R-23**], a presentation to the Pittsburgh Coal Conference [**P-5**] and a paper in *Coal Preparation* [**P-1**].

Since the PDU data represented operations over a longer period of time, the percentage reduction in the concentrations of the impurity ash, sulfur, and trace elements on a heating value basis (lb/MBtu) from the amounts in the test coals and in the ROM parent coals are shown in Figure 28 for these tests. Similar data were presented earlier for the bench-scale results [**P-5**].

There were substantial reductions (25 to 75 percent) in the concentrations of some impurities, especially ash, arsenic and manganese, on a heating value basis from the amounts in the as-received test coals. On the other hand, there was little or no reduction (less than 25 percent or negative) in the amounts of antimony, beryllium, cobalt, nickel, and selenium in the as-received test coals on the same basis. Little or no reduction in the concentration of an impurity means that the impurity is closely associated with or actually part of the carbonaceous components of the coal, whereas a substantial reduction signifies an association with the mineral matter in the coal. The reduction of other impurities, such as total sulfur, pyrite sulfur, cadmium, chromium, mercury, lead and chlorine varied from coal to coal. Reductions from the trace-element concentrations found in the ROM parent coals were generally greater than the reductions from the as-received test coals on a heating value basis.

Of particular interest, the PDU fine coal cleaning was effective for reducing the lb/MBtu concentration of mercury in the Taggart and Indiana VII coals by 39 percent or more. Similar reductions had been seen earlier for the bench-scale cleaning of Winifrede, Taggart, Sunnyside, Indiana VII, and Elkhorn No. 3 coals. The Hiawatha coal contained less mercury to begin with than the other coals, and the advanced fine coal cleaning had less impact upon the final concentration of mercury in the clean product from that coal than it did on the mercury concentration in the other coals.

The reduction in the concentrations of the trace elements was confirmed by comparing the analyses of the clean coals and the corresponding fine refuse products. In particular, the concentration of mercury in refuse samples were two to four times as high as the concentrations in the ROM parent and the PDU test coals, even in the case of the Hiawatha coal. The concentrations of chromium and manganese were very high in some of the fine refuse samples due to metal worn off the balls in the grinding mills, particularly when the stirred ball mill was used for the bench-scale grinding.

SUMMARY

The two advanced cleaning procedures -- column flotation and selective agglomeration -- appeared to be equally effective for reducing the concentrations of impurities in coal and equally effective for cleaning coal to premium fuel specifications. For certain coals, physically cleaning to premium fuel specifications substantially reduced the concentrations of some of the hazardous air pollutant trace elements, especially arsenic, chromium, cobalt, lead, manganese, mercury, selenium, and chlorine, in the coals. As such, fine coal cleaning can be a useful part of a hazardous air pollutant control strategy for coal-fired utilities.

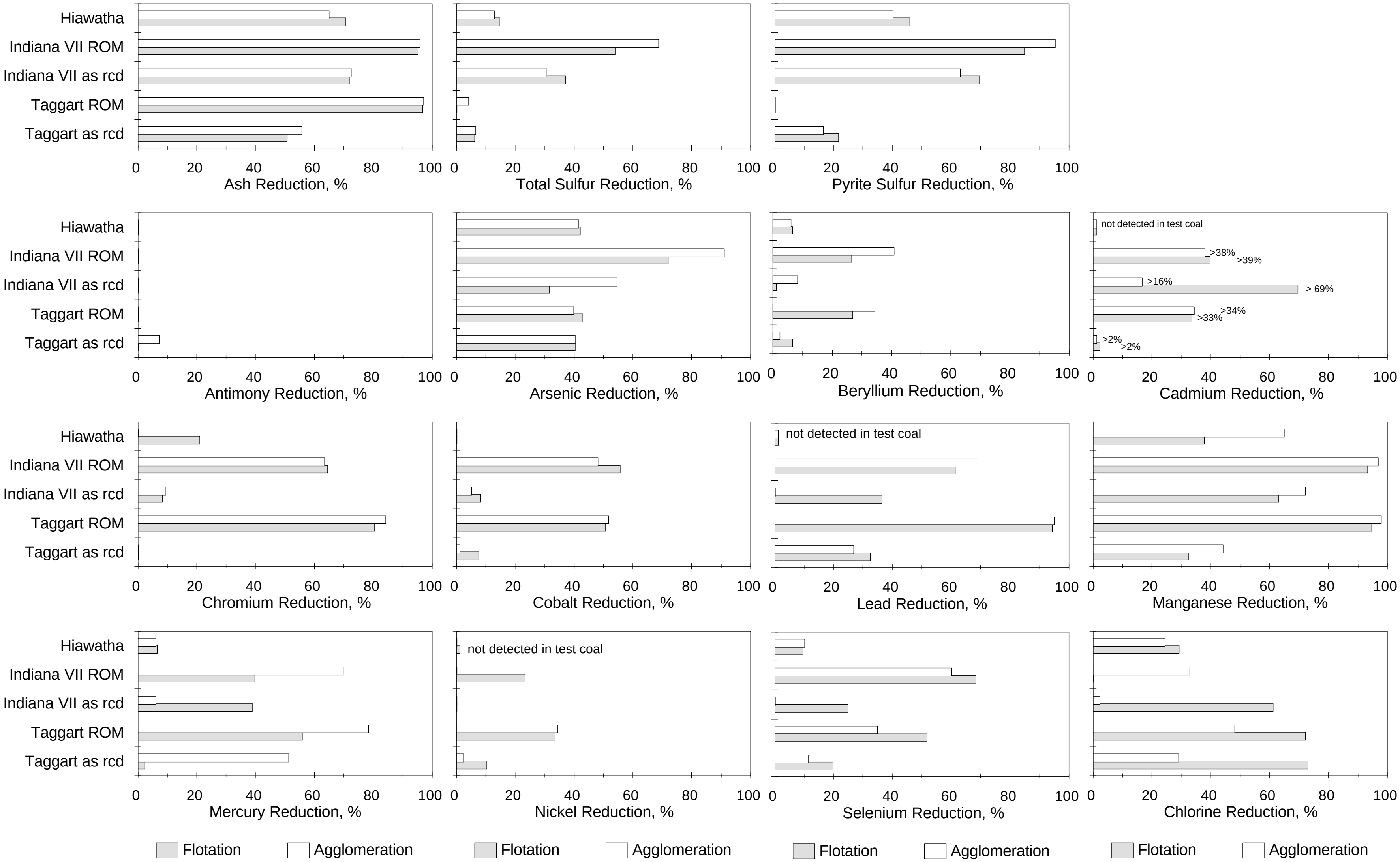


Figure 28. Percentage Reduction in the Trace Element Concentrations Achieved by Advanced Physical Fine Coal Cleaning.
(Reductions are on a Heating Value Basis. Negative values are not shown on these charts.)

8. PREPARATION OF COAL WATER SLURRY FUEL

The form in which the fuel is delivered to and handled by the end-user is an important consideration when marketing premium fuel prepared from coal. A pumpable, highly-loaded slurry of coal and water (coal-water slurry fuel or CWF) appears to be a highly attractive option. CWF technology is generally regarded as ready for large-scale commercial use in a class of oil-fired boilers liberally designed to be “coal-capable.” Using commercially ready technology, the conversion of more typical large oil-designed boilers is predicted to result in significant derating. Available information from DOE fuel development studies indicate that the derating is driven by three factors: (1) the size of the particles undergoing combustion, (2) the ash content of the CWF, and (3) the water content of the CWF. All three factors must be minimized in order avoid derating the boiler by an excessive amount. During this project, particle size and ash content issues were largely fixed by the efficiency of the coal cleaning process. The water content issue was addressed by laboratory investigations of the properties CWF prepared from the clean coal [**P-4**, **R-8**, **R-15**].

To be a viable substitute for oil and natural gas, CWF must be fluid enough that it may be pumped and atomized efficiently yet it must not contain any more water than necessary to achieve such fluidity. For ordinary boiler firing, a suitable balance between loading and fluidity seems to be at 60 to 65 percent coal loading and 200 to 500 cP viscosity. Higher loadings are more desirable, though, so parametric tests were conducted on the clean coals from the bench-scale circuits to optimize the CWF formulations. The stability of the formulations was also examined during the optimization since separation of hard-packed sediments can lead to handling problems when using CWF.

CWF PREPARATION AND EVALUATION

CWF was produced in the laboratory by blending water and reagents with partially dried clean coal filter cakes as described in the subtask topical reports [**R-8**, **R-15**]. Slurry preparation tests were made on the filter cakes directly and on filter cakes after manipulation of the particle size distribution (PSD) to improve the packing of the particles in the slurry. The viscosity of most of the CWF samples was reduced by adding a commercial naphthalene sulfonate reagent called A-23M to the mixture in order to disperse flocculated coal particles. A slurry was considered to have formed when the mixture in the blender achieved a uniform texture and sufficient fluidity to flow when the container was tipped. Particle size manipulation was accomplished by regrinding a portion of the clean coal to a finer size distribution and blending the reground portion back with the unground portion. In this manner, the PSD was altered to a bimodal distribution which provided better packing of the particles and improved the loading of the slurry at a given viscosity.

Following an initial characterization, additional successive dilutions were often made with small quantities of water. Viscosity measurements were repeated after each dilution. In this manner, the relationship between solids loading and viscosity were developed for each clean coal PSD and reagent level. At times, additional increments of dispersant and/or stabilizer were added to evaluate their effect on solids loading, viscosity, and stability.

A Fann rotating-cup and bob viscometer was used to determine the rheology of slurry fuel samples, and a probing procedure was used to rate the stability of the slurries [**R-8**, **R-15**].

EFFECTS OF CWF PREPARATION VARIABLES

The effects of varying dispersant additions and adjustments of the particle size distribution were investigated by the parametric testing described in topical reports for coal cleaned by advanced flotation [**R-8**] and coal cleaned by selective agglomeration [**R-15**]. Certain tests on clean coals from the bench-scale circuits provided CWF samples with near-optimum formulations. Properties of CWFs from these selected tests are presented in Table 5. From these results, and from individual parametric tests, one may project the effects that coal rank, PSD and PSD adjustment, cleaning procedure, and stabilization have on CWF loading and reagent requirements.

Table 5. Physical Properties of Premium CWF Slurries

	Taggart	Winifrede	Elkhorn No. 3	Indiana VII	Sunnyside	Hiawatha
Cleaned by Advanced Flotation:						
MMD of coal, µm	44.2		36.6	10.7	28.2	
A-23M, % of coal	0.5		0.5	1.0	1.0	
Coal Loading, wt %	67.2		60.4	50.9	61.9	
Viscosity, cP at 100 s^{-1}	515		220	860	335	
HHV, Btu/lb slurry	10,241		8,827	7,180	8,899	
Cleaned by Selective Agglomeration:						
MMD of coal, µm	21.0	7.4	37.9	14.8	24.9	30.1
A-23M, % of coal	0.5	1.5	0.5	1.0	1.0	1.0
Coal Loading, wt %	62.2	48.8	60.0	52.5	61.9	59.5
Viscosity, cP at 100 s^{-1}	450	10	220	630	225	205
HHV, Btu/lb slurry	9,485	7,085	8,762	7,405	8,902	8,514

Dispersant

The A-23M dispersant served to reduce the viscosity of the slurry thereby allowing formulation of higher loading CWF. Figure 29 is an example of the effect of varying additions of A-23M on the loading of CWF prepared from Taggart coal. The addition of A-23M allowed preparation of Taggart slurries containing 60 to 65 percent coal rather than 52 percent coal. Note that there was little improvement in the loading of Taggart CWF when the A-23M additions increased above 0.5 percent by weight of the coal in the slurry. Similar patterns were seen for the other clean coals, and the amounts of A-23M used in the tests listed in Table 5 were usually for the point where the loading began to plateau as in Figure 29.

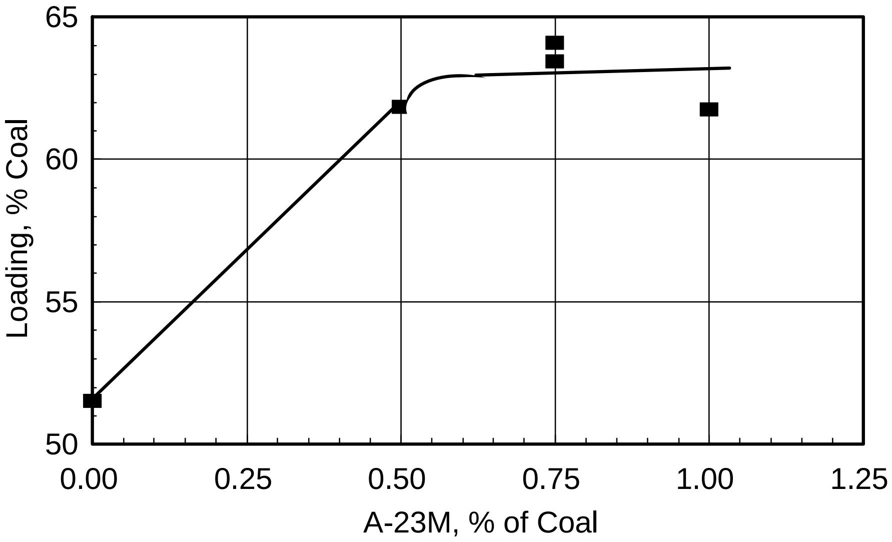


Figure 29. Effect of Dispersant Addition on Loading of Taggart CWF (Loadings Normalized to 500-cP Viscosity)

Rank of Coal

The test coals were all high volatile bituminous coals but varied enough within this rank (as defined by the ASTM D-388 moist, mineral-matter free heating value) that a difference was seen in the loadings of optimized slurries during the parametric testing. Figure 30 shows the relationship between the loadings listed in Table 5 and rank as defined by the heating value. Taggart, the highest ranking coal, provided the highest slurry loading, and Indiana VII, the lowest ranking coal, provided the lowest loading. The high ranking Winifrede coal did not follow the pattern shown by the other coals because it had been ground much finer than the other coals – to minus 20 µm.

The equilibrium moisture of the parent coal was an alternative indication of the rank of a high-volatile coal. It varied from 1 percent in the Taggart coal on up to 14 percent in the test coal from the Indiana VII seam. Thus, the lower loadings of CWF from the lower ranking coals can be attributed, in part, to the moisture which soaked into the coal particles and was not available to provide fluidity to the slurry.

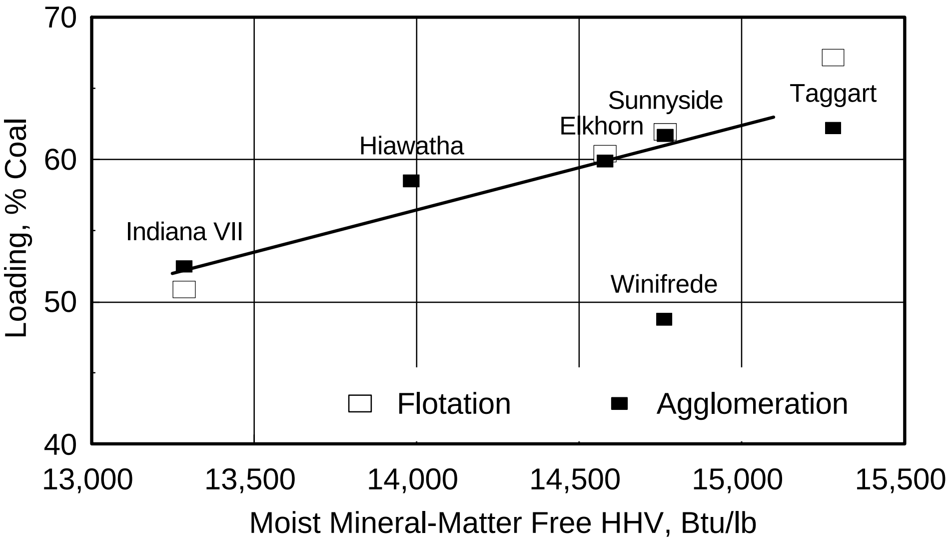


Figure 30. Effect of Coal Rank on CWF Loading (Fully dispersed with A-23M)

Note: ASTM high volatile bituminous coal rank designation increases with moist, mineral-matter free heating value

Particle Size

It was necessary to grind the coals to differing degrees of fineness in order to achieve the target ash rejection during cleaning. The fineness of grinding had a significant impact upon the loadings of the optimized slurries as shown in Figure 31. There was some overlap in Figure 31 with the effect of varying rank because Taggart coal, the coal which did not require as fine a grind as the other coals, also had the highest rank of the six coals. A multiple regression analysis of the data from the test program showed that the particle-size effect was a more significant effect than the effect of the rank of the parent coal.

The fineness of the grind also had an impact on the amount of A-23M required for producing CWF. This is shown in Figure 32. In this case, a multiple regression analysis showed little or no effect of coal rank on the amount of dispersant required for the slurry.

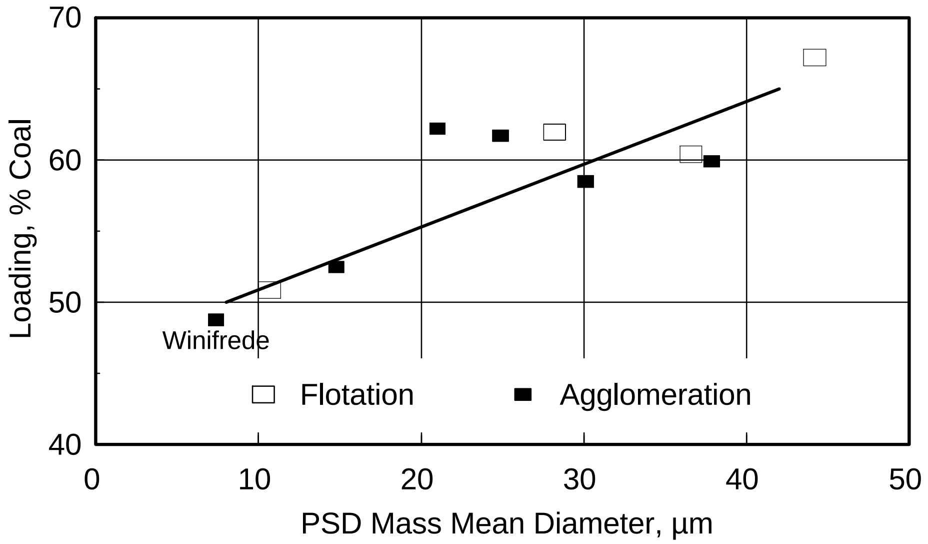


Figure 31. Effect of Particle Size on CWF Loading (Fully dispersed with A-23M)

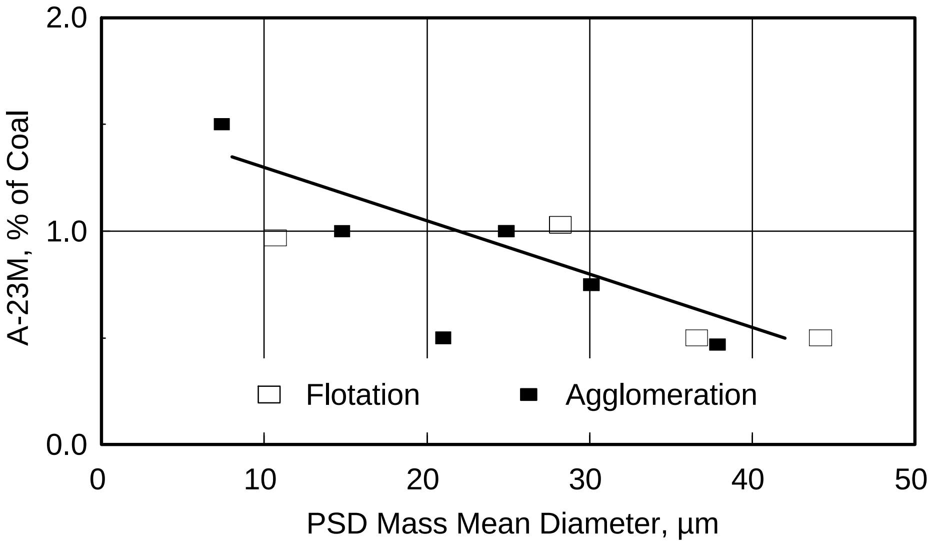


Figure 32. Effect of Particle Size on Dispersant Requirement

Adjustment of the Particle Size Distribution

The natural PSDs of the clean coals were quite uniform with little concentration of particles in any particular size range. A number of tests were made on the Taggart, Elkhorn No. 3, Sunnyside, and Hiawatha coals cleaned by selective agglomeration to determine whether adjustment of the distributions would allow preparation of higher-loading CWF. Since the PSD adjustments were accomplished by grinding a portion of the clean coal this meant that the overall distribution also became finer. The volumetric loadings of these test slurries are compared in Figure 33 to the volumetric loadings of comparable slurries prepared with the natural PSDs. It does, indeed, appear that PSD adjustment improved the loading of the CWF samples but only by a few percent. Because of its cost and complexity and limited benefit, the PSD adjustment does not appear to be a commercially viable approach.

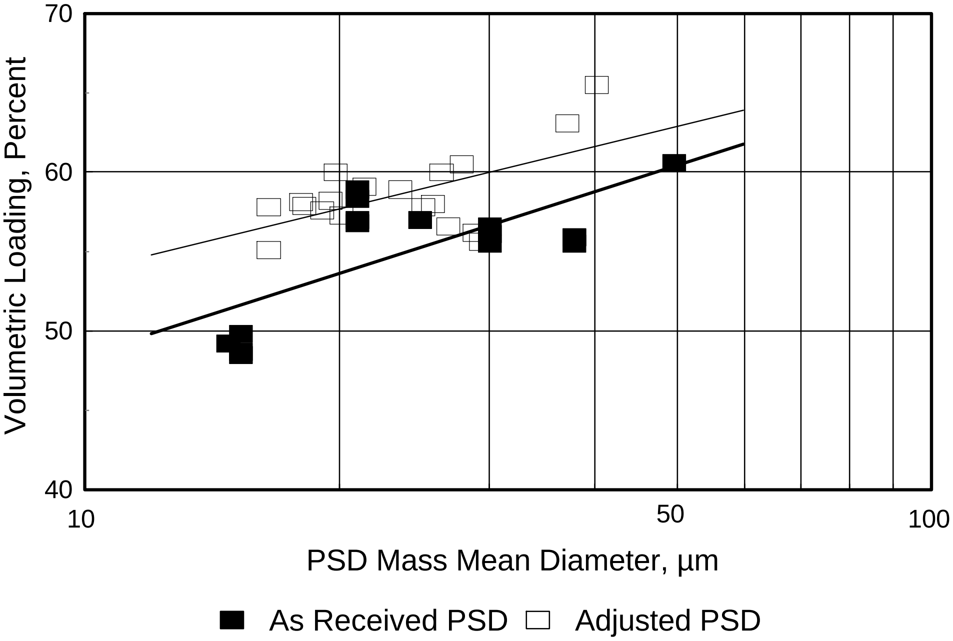


Figure 33. Effect of PSD Adjustment on CWF Loading (Fully dispersed with A-23M)

Coal Cleaning Process

The CWF formulation data for coals cleaned by flotation and by agglomeration were plotted separately in Figures 30 through 32. It can be seen from these plots that it made little or no difference how the coals were cleaned. Particle size and coal rank were the main factors affecting their slurryability. The clean coal from the selective agglomeration had been stripped with steam to recover the heptane bridging liquid and the small amount of residual heptane did not appear to effect the slurryability of the clean coal.

Stabilization

Hard-pack cake formed in CWF samples formulated with A-23M dispersant when they were stored overnight. Xanthan gum (Flocon) was investigated as a stabilizing reagent to inhibit such sedimentation. A soft easily-remixed sediment formed when 800 ppm Flocon 4800C gum was added to the CWF, even when the samples were stored for a week or more. Unfortunately, the xanthan gum also increased the viscosity of the slurries, so the coal loadings had to be reduced in order to maintain the desired fluidity. The following data for 500-cP Sunnyside slurries were typical of the effects seen for the 800-ppm stabilizer additions:

	w/o Flocon	w/Flocon
Coal Loading, wt %:	61.4	60.0
Heating Value, Btu/lb	8,830	8,630

Because of the cost of the Flocon (several dollars per lb) and because of its uncertain effectiveness over long storage periods (months rather than weeks), it is recommended that CWF should be prepared without using a stabilizer and that it should be kept in mixing tanks and burned soon after preparation.

COMMERCIAL SPECIFICATIONS OF PREMIUM CWF

This investigation has shown what specifications can be expected for commercial production of premium CWF [**P-4**, **R-15**]. Earlier, the bench-scale advanced cleaning [**R-9**, **R-16**], and the subsequent PDU operations [**R-21**, **R-23**], had shown that selected, but readily available, coals can be cleaned to less than 2 lb ash per million Btu. Fine grinding was required, but one could still prepare CWF from at least two Eastern and two Western coals that would contain more than 8,500 Btu/lb and meet the premium fuel ash specification. All four of these coals were from high-volatile A bituminous seams where the ash minerals could be liberated by grinding the coal to between minus 62 mesh and minus 150 mesh. CWF prepared from a lower-rank (high-volatile C) Midwestern coal had heating values between 7,000 and 7,500 Btu/lb. In the case of the Midwestern coal, the lower loading of the CWF was partly due to the finer grinding required for ash mineral liberation from that coal.

CWF slurries prepared from high volatile A coals contained 60 to 62 percent coal and were formulated with A-23M dispersant to have viscosities of less than 500 cP at 100 s^{-1}. They were intended for use soon after preparation, and provisions would be needed in the fuel system for frequent mixing and for draining fuel lines when not in use. If desired, the fuel could be formulated with a stabilizer such as Flocon, and perhaps with less dispersant, to alleviate some of the need for remixing and line drainage by the user, but the loading and heating value specifications of the CWF would have to be reduced if one wished to maintain a viscosity of less than 500 cP.

As a cost-saving alternative, CWF from the high volatile A coals could also be formulated without dispersant. Such fuel would contain about 52 percent coal and have a higher heating value in the 7,200 to 7,500 Btu/lb range.

9. PROPERTIES OF PREMIUM FUEL PRODUCED IN PDU

The clean coal produced during the extended production runs in the PDU provides examples of the quality of the premium fuel which can be produced by advanced physical cleaning of fine coal. The properties of these clean coals are presented in this chapter and a comparison made between the two technologies.

As discussed below, particularly good quality fuel was prepared from the Taggart and Hiawatha coals in the PDU. However, earlier laboratory and bench-scale testing had also shown that Sunnyside and Elkhorn No. 3 coals may also be good source coal candidates for preparation of premium fuel.

COMPOSITION AND YIELD OF CLEAN COALS AND CWF

The compositions of the clean coals from the PDU extended production runs (three by flotation and three by selective agglomeration) are presented in Table 6. The sulfur specification of less than 0.6 lb/MBtu was met in all six instances, and the ash specification of less than 2.0 lb/MBtu was met for the Taggart and Hiawatha coals. It should be pointed out that the extended production runs were not necessarily conducted at the operating conditions where the ash specifications would have been met. Specifically, less than 1.0 lb/MBtu ash coal was produced during certain flotation and selective agglomeration parametric tests on the Taggart coal. Similarly, less than 2.0 lb/MBtu ash coal was produced during certain flotation and selective agglomeration parametric tests on the Indiana VII coal albeit with some difficulty operating the grinding and, in particular, the dewatering circuits of the PDU.

The clean coal yields and Btu recoveries are also listed in Table 6. Btu recoveries met project goals in each instance except for the flotation cleaning of the Indiana VII coal where the recovery would have fell short of the project goal of 80 percent Btu recovery from the ROM coal.

There were no slurry preparation tests conducted on the filter cakes from the PDU extended operations. However, there was sufficient information available from the slurry preparation testing done on the bench-scale production [**R-8**, **R-15**] to allow projection of the slurry properties of the CWF that can be prepared from the clean coals. These projections are shown in Table 7.

Table 6. Composition and Yield of Production Run Clean Coals

(Analyses are for bone-dry coal by Hazen Research Inc)

	Taggart Clean Coal		Hiawatha Clean Coal		Indiana VII Clean Coal	
	Agglomeration	Flotation	Agglomeration	Flotation	Agglomeration	Flotation
Ultimate Analysis, %:						
Carbon	87.62		82.35		81.24	
Hydrogen	5.14		5.51		4.82	
Nitrogen	1.53		1.56		1.79	
Sulfur	0.63	0.72	0.50	0.63	0.43	0.59
Ash	1.64	1.83	2.73	2.70	4.27	3.23
Oxygen	3.44		7.35		7.45	
Total	100.00		100.00		100.00	
HHV, Btu/lb:	15,072	15,045	14,302	14,296	13,836	13,849
Proximate Analysis, %:						
Ash	1.64	1.83	2.73	2.70	4.27	3.23
Volatile Matter	35.09		43.54		35.12	
Fixed Carbon	63.27		53.73		60.61	
Total	100.00		100.00		100.00	
Ash, lb/MBtu	1.09	1.22	1.91	1.89	3.08	2.33
Sulfur, lb/MBtu	0.42	0.48	0.35	0.44	0.31	0.43
Yield, %	96.7	95.3	92.8	81.8	93.5	75.2
Btu Recovery, %	99.2	96.9	98.9	88.0	99.9	82.9

Table 7. Projected Slurry Properties of Premium Coal Water Fuels

	Taggart Clean Coal		Hiawatha Clean Coal		Indiana VII Clean Coal	
	Agglomeration	Flotation	Agglomeration	Flotation	Agglomeration	Flotation
Loading, % Coal	60	60	60	60	52	52
Viscosity, cP	500	500	500	500	500	500
HHV, Btu/lb slurry	9,043	9,027	8,581	8,578	7,195	7,201
Ash, % (slurry basis)	0.98	1.10	1.64	1.62	2.22	1.68
, lb/MBtu	1.09	1.22	1.91	1.89	3.08	2.33
Sulfur, % (slurry basis)	0.42	0.48	0.35	0.44	0.31	0.43
, lb/MBtu	0.42	0.48	0.35	0.44	0.31	0.43
Top Size, mesh	140	100	70	140		270
PSD, D80 µm	30	71	42	48	50	23

ASH PROPERTIES OF FUELS

Hazen Research Inc, Golden CO, determined the ash chemistry and fusion properties of the feed coal and the clean coal samples from the extended production PDU advanced flotation and selective agglomeration runs on the Taggart, Indiana VII and Hiawatha coals [**R-21**, **R-23**]. It was found that the 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 cleaned by column flotation [**R-21**]. A similar pattern was seen for these two coals when cleaned by selective agglomeration except that the decline was not as great in the case of the Indiana VII coal [**R-23**]. It should be noted that the shipment of Indiana VII coal cleaned by selective agglomeration seemed to have had a more siliceous ash than the ash in previous shipments. This may be the reason that the fusion temperatures of the ash in the Indiana VII were not as much affected by the agglomeration cleaning as was the ash in the Indiana VII coal cleaned by flotation. The fine coal cleaning did not have much impact upon the fusion temperatures of the ash in the Hiawatha coal. The complete set of fusion temperatures are listed in Table 8.

The compositions of the ash in the clean coals produced during the production runs are presented in Table 9. Except for titanium dioxide, and iron oxide in the case of Taggart coal, the concentrations of the ash constituents, including the alkali metals, were significantly reduced from the amounts in the feed coals on a heating value (lb/MBtu) basis by both the flotation and the selective agglomeration in the PDU [**R-21**, **R-22**]. The slagging and fouling characteristics of the ashes were little changed by the cleaning and remained in the low and medium categories.

COMPARISON OF TECHNOLOGIES

A comparison of the results presented in Tables 6 through 9, and also of the results on toxic trace element rejection presented in Chapter 7, suggests that column flotation and selective agglomeration were equally effective methods for cleaning coal to premium fuel specifications. Undoubtedly the results were coal-specific but there were indications from the Btu recovery comparisons (see Table 6) that selective agglomeration provided a somewhat higher product yield. This difference in yield may be most noticeable when the flotation response of the coal was poor or when very fine grinding was needed to liberate the ash minerals as true for Indiana VII coal.

Table 8. Fusion Temperatures of Ash in Test Coals Before and After PDU Advanced Cleaning

	Taggart Coal		Indiana VII Coal		Hiawatha Coal	
	Before Cleaning	After Cleaning	Before Cleaning	After Cleaning	Before Cleaning	After Cleaning
			Cleaned by Advanced Column Flotation			
Oxidizing Atmosphere, °F:						
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, °F:						
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
			Cleaned by Selective Agglomeration			
Oxidizing Atmosphere, °F						
Initial	2570	2485	2540	2482	2170	2290
Softening	2657	2618	2583	2541	2230	2306
Hemispherical	2695	2630	2600	2560	2300	2319
Fluid	2710	2680	2625	2590	2445	2333
Reducing Atmosphere, °F						
Initial	2286	2236	2300	2270	2084	2120
Softening	2552	2396	2479	2362	2145	2181
Hemispherical	2600	2475	2489	2400	2255	2195
Fluid	2664	2600	2500	2455	2346	2220

Table 9. Ash Composition of Test Coals After Cleaning

	Taggart Coal		Indiana VII Coal		Hiawatha Coal	
	Flotation	Agglomeration	Flotation	Agglomeration	Flotation	Agglomeration
Constituent, %:						
SiO_2	41.43	46.24	46.44	58.96	40.28	38.54
Al_2O_3	27.72	28.81	23.04	24.68	20.05	21.87
TiO_2	1.65	1.92	3.09	2.20	2.02	2.22
Fe_2O_3	17.92	14.12	11.92	6.56	9.36	7.29
CaO	1.95	2.05	3.71	1.63	9.22	8.49
MgO	0.61	0.60	1.03	0.67	1.20	0.78
Na_2O	0.77	0.81	1.21	0.71	3.90	4.30
K_2O	2.17	2.27	3.43	3.20	0.71	0.56
P_2O_5	0.29	0.29	0.23	0.19	0.75	1.03
SO_3	0.99	1.52	1.68	1.34	11.10	10.20
Ash Type	High Rank	High Rank	High Rank	High Rank	Lignite	Lignite
Slagging Type	--	Low	--	Low	Medium	Medium
Fouling Type	Medium	Medium	Medium	Low	Medium	Medium

10. COST OF COMMERCIAL PREMIUM FUEL PRODUCTION

An important goal of this project was to develop the two advanced cleaning processes so as to produce premium fuel from coal commercially at a cost of less than $2.50 per million Btu including the mine-mouth cost of the raw coal. For this reason, Bechtel conducted commercial production cost studies for two conceptual plants producing premium CWF by advanced physical fine coal cleaning [**R-25**]. One plant utilized column flotation for the cleaning technology, and the other plant utilized selective agglomeration for the cleaning technology. Plant design and operating parameters for the two conceptual commercial facilities were based on the results of the bench-scale and PDU testing described in this report.

CONCEPTUAL PLANT

The conceptual premium CWF production plants would be located in an industrial area of an Ohio valley state and near potential customers for the fuel. The plants would produce 2.5 million short tons of CWF per year containing 1.5 million short tons of coal. Feed stock for the plants would be purchased from mines in the central Appalachian area that produce coal that upgrades in a manner similar to the Taggart, Elkhorn No. 3, Sunnyside and Hiawatha test coals used for the bench-scale and PDU testing. The CWF would contain 60-62 percent coal (8,900-9,400 Btu/lb), less than 2.0 lb ash per million Btu, less than 0.6 lb sulfur per million Btu, and would have a viscosity of 500 cP.

Bechtel assembled capital and operating data for the study and developed equipment flowsheets and levellized cost projections for producing premium CWF by fine grinding and application of the two advanced cleaning technologies. The conceptual plants included sections for coal receiving and storage, crushing and grinding, advanced physical cleaning, clean coal dewatering, CWF preparation, storage and load-out, tailings handling, and recycle water clarification. Details are provided in the report of the study [**R-24**]. The selective agglomeration advanced cleaning section included facilities for heptane recovery and reuse. For the base case, the plants would operate 24 hr/day, 7 days/week for a scheduled 7,600 hours of production per year. Figure 34 is a layout for the conceptual plant employing the advanced fine coal cleaning technologies for commercial CWF production.

An advanced flotation premium fuel production plant was found to be less expensive to place into service than a selective agglomeration plant:

	Estimated Cost, $millions	
	Flotation	Agglomeration
Construction and Start-up Cost	69.6	97.2
Working Capital	10.0	11.0
Total	79.6	108.2

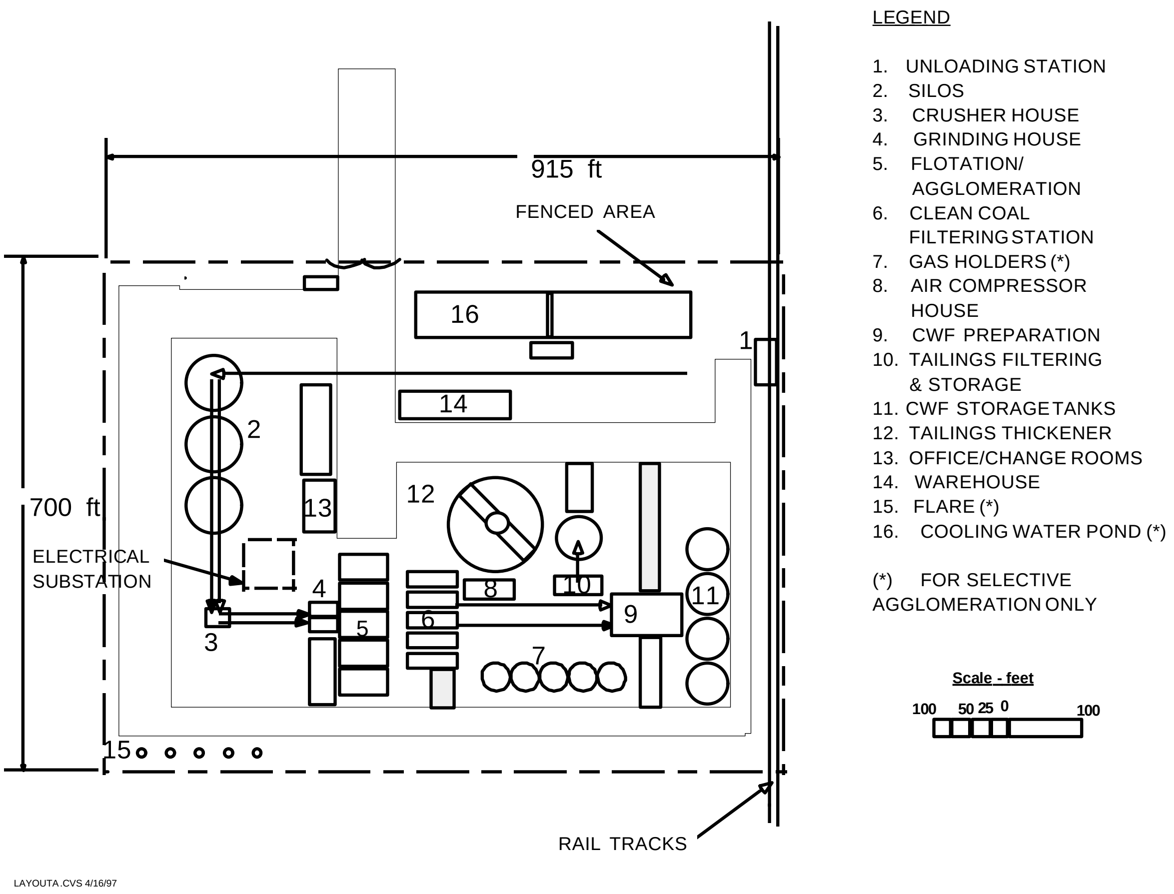


Figure 34. Plant Layout for Advanced Technologies Commercial CWF Production

Total fixed and variable operating and maintenance (O&M) costs for producing CWF by the two cleaning methods were estimated to be $2.15/MBtu and $2.42/MBtu for advanced flotation and selective agglomeration processes, respectively. The cost of the feed coal delivered to the premium fuel plants (at $1.24/MBtu or about $32.40/st) was included in these estimates. The cost of the feed coal included preparation and loading costs at the mine and also included $5.20/st ($0.20/MBtu) for freight to the premium fuel production plant. In each case, the cost of producing premium CWF compared favorably with the cost of No. 6 fuel oil (about $3.35/MBtu or $0.50/gallon at the time) and met the project goal of less than $2.50/MBtu.

A breakdown of the annualized O&M costs is presented in Table 10. As shown in the Table, the A-23M dispersant added to reduce the CWF viscosity was the second most costly item among the O&M costs. A savings of $0.23/MBtu would be accomplished by formulating the CWF without A-23M. Such CWF would contain 54 percent coal instead of 60-62 percent and have a heating value of 8,100 Btu/lb instead of 8,900-9,400 Btu/lb.

Table 10. Projected Variable and Fixed O&M Costs and Total Cost of Premium CWF

	Advanced Flotation			Selective Agglomeration		
	$/st Coal*	$/MBtu	Percent	$/st Coal*	$/MBtu	Percent
Capital Charges including Interest on Working Capital**	7.94	0.27	12.3	10.94	0.37	15.1
CWF Additive (A-23M)	7.00	0.23	10.8	7.00	0.23	9.7
Labor	4.05	0.13	6.3	5.20	0.17	7.2
Electric Power	3.35	0.11	5.2	4.50	0.15	6.2
Flotation Reagents and Flocculants	2.05	0.07	3.2	0.83	0.03	1.1
Heptane and Steam				4.82	0.16	6.7
Btu Losses	1.50	0.05	2.3	0.38	0.01	0.5
Others	1.47	0.05	2.3	1.53	0.06	2.1
Total O&M Costs	27.36	0.91	42.4	35.20	1.18	48.6
Coal (Delivered to Plant, dry basis)	37.21	1.24	57.6	37.21	1.24	51.4
Total Projected Cost of CWF	64.57	2.15	100.0	72.41	2.42	100.0

* per short ton dry clean coal in the CWF.

** Construction and start-up costs amortized over 20 years at 15 percent return. 8 percent interest on working capital.

The results of sensitivity studies on process economics are illustrated in Figures 35 and 36. As one would expect, variations in the price of the feed coal had a significant impact upon the cost of the CWF since the cost of the feed coal accounted for half of the production cost of the CWF. Variations in the annual production rate, either due to variations in equipment performance or to variations in the scheduled number of hours of operation, also had a significant impact upon unit production cost. A reduction of the operating schedule to 10 shifts per week, in particular, instead of the design 19 shifts per week had a serious negative impact upon production cost. Variations in other sensitivity parameters – ash in the feed coal, labor, electricity, reagent, and steam costs, and construction and start-up costs – had lesser effects upon the annualized cost of producing CWF. The relative sensitivity of production cost to the various operating and investment parameters was about the same for each of the two cleaning technologies.

CONCLUSIONS AND RECOMMENDATIONS OF THE COST STUDY

The estimated cost of commercial production of premium CWF using either column flotation or selective agglomeration processes was encouraging. Column flotation was particularly promising. This process, in spite of its lower yield and Btu recovery, was found to be more economical than selective agglomeration while offering a comparable quality product. Total O&M processing cost with column flotation, at $0.91/MBtu, was significantly lower than the cost of $1.18/MBtu estimated for the plant using selective agglomeration.

As found during the CWF cost sensitivity analysis, one of the significant factors that could vitally affect production costs was the annual sustainable production rate. Product costs would escalate drastically if the annual production rate of 1.5 million short tons cannot be achieved in plants built according to the conceptual designs presented in the study. Two significant technical factors that could adversely affect production are (a) reduced plant availability due to worse than anticipated plant operability or maintenance requirements and (b) feed coal that is either harder to grind or requires finer grinding than expected. The latter possibilities would reduce the grinding capacity of the plants and have an adverse effect upon the annual production of CWF.

These technical uncertainties are best resolved by operating experience with a larger scale plant and for a longer term than was possible with the PDU. It was noticed during the conceptual design stage that plants with capacities of 1.5 million short tons per year of ultra-clean coal will need 12 parallel trains of commercial-size flotation equipment or 10 parallel trains of commercial-size agglomeration equipment. This would suggest installation of a demonstration/production plant using a single train of commercial equipment with a capacity in the 125,000 to 150,000 short ton per year range.

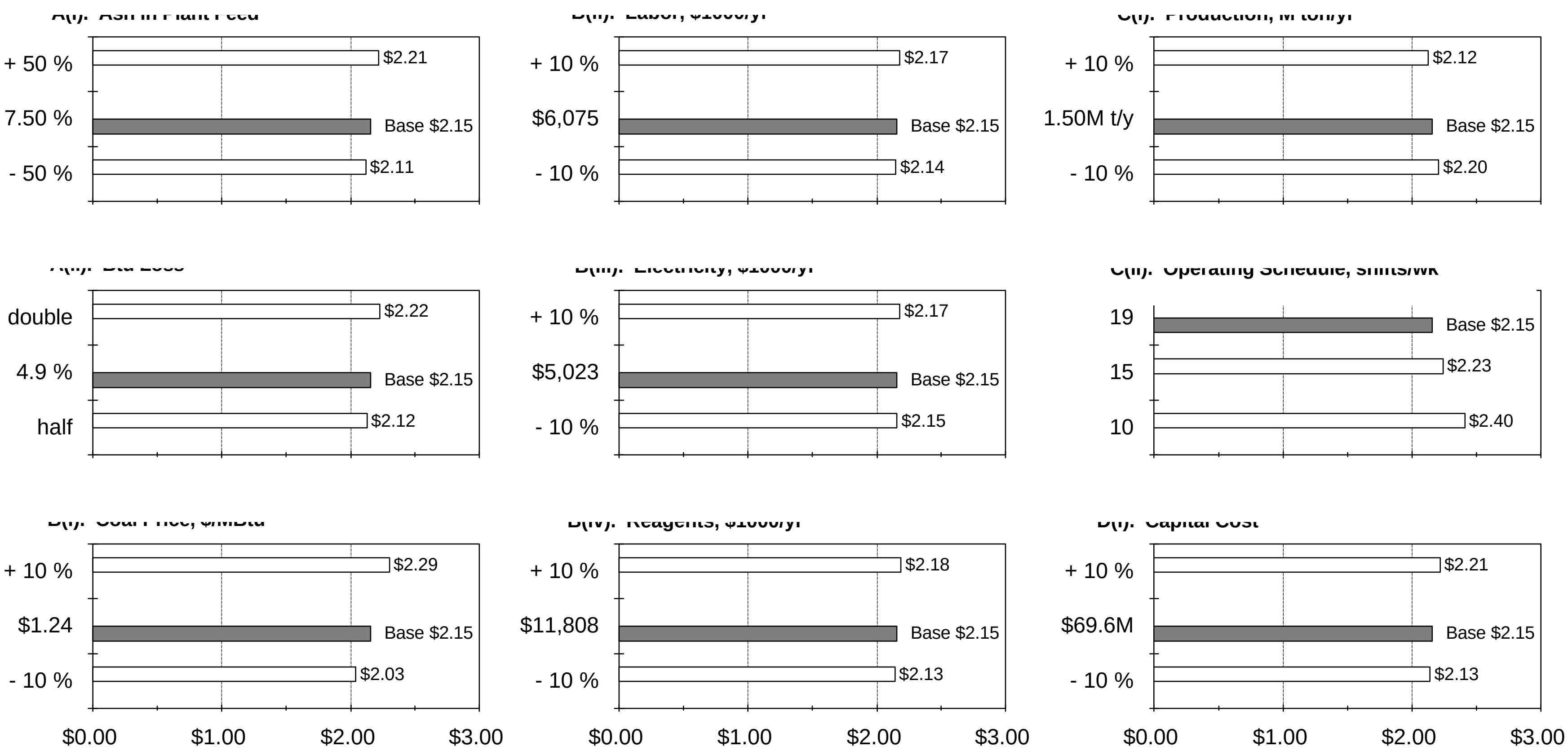


Figure 35. Sensitivity of Estimated Flotation CWF Production Cost ($/MBtu) to Changing Operating and Investment Parameters

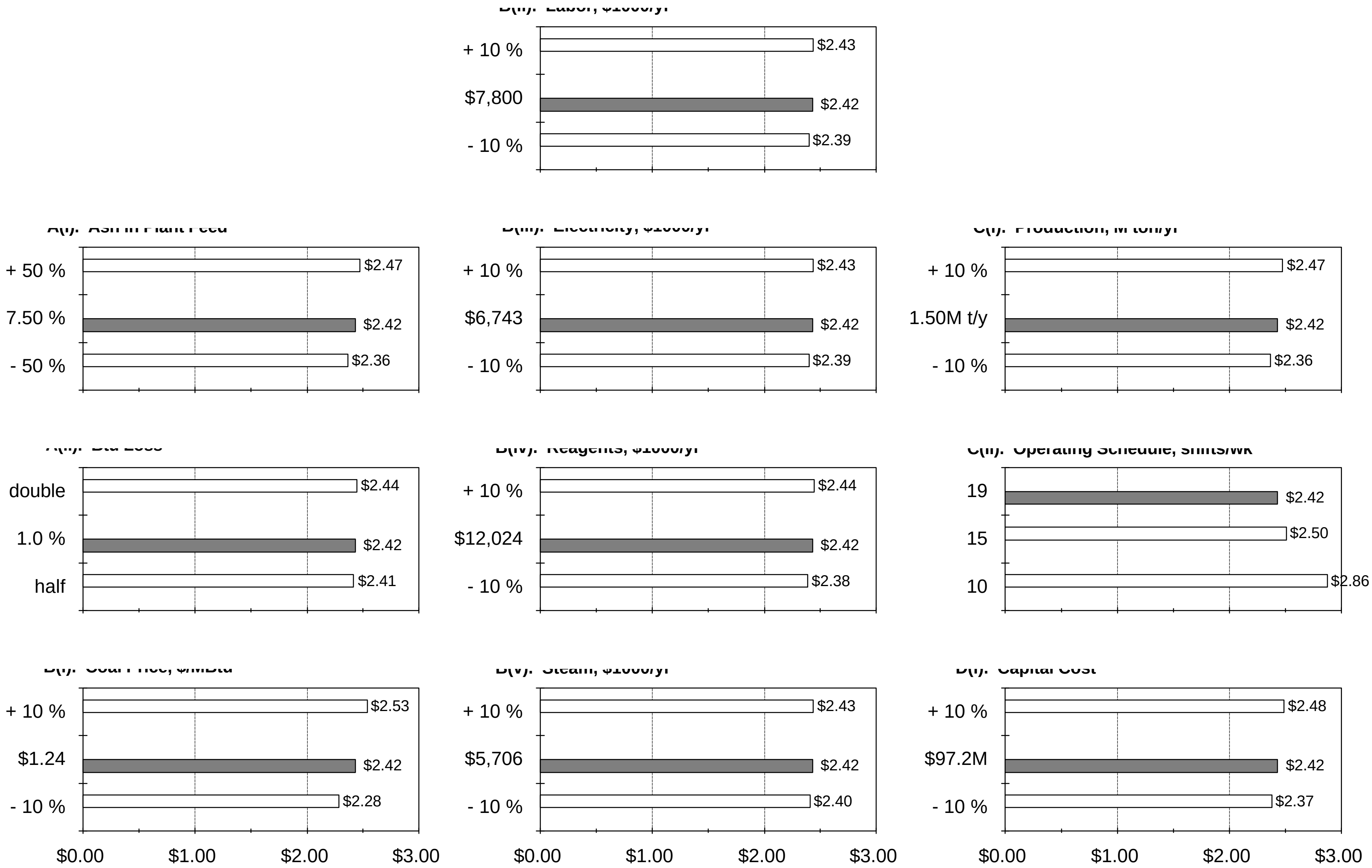


Figure 36. Sensitivity of Estimated Agglomeration CWF Production Cost ($/MBtu) to Changing Operating and Investment Parameters

Another issue of uncertainty is the acceptability of the product. Before long-term commitments can be made, all potential customers would need verification, by meaningful plant-scale testing, of the suitability of premium CWF for their applications. A single-train demonstration/production plant of the capacity suggested here will enable production of adequate amounts of premium CWF for this purpose.

Preferably, the demonstration/production plant would be built close to a coal mine so that the costs associated with feed coal transportation, refuse disposal, and rail car unloading can be minimized or eliminated.

11. NEAR-TERM APPLICATION OF ADVANCED TECHNOLOGIES

Near-term applications were an extension of the premium fuel project to specifically address the use of advanced flotation and selective agglomeration processes for recovering 28-mesh and finer coal lost in existing coal preparation plants. The goal was to produce clean coal which could be sold in existing markets by one or both of the following strategies:

- Increase the percentage recovery of marketable coal from the ROM coal.
- Improve the quality and value of the marketable coal (heating value, sulfur or ash content, and handling characteristics) in a cost-effective manner.

Such applications would represent immediate near-term benefits from the project and would complement the long-term benefits gained from the production of premium fuel from coal.

A five-step approach was followed to accomplish the goals of this task. The steps were as follows:

1. Survey, with close cooperation from division-level operating management, Amax Coal Company properties to determine which preparation plants were candidates for application of the new technologies. The factors to be considered included the extent of the fine coal losses in the various plants, the accessibility of the waste coal streams for study, and the likelihood of a major renovation or expansion of the plant.

2. Perform laboratory column flotation and selective agglomeration amenability tests on samples collected from the candidate preparation plants in order to determine operating conditions and potential product quality and recovery.

3. Design conceptual plants integrating the advanced flotation and selective agglomeration technologies into the existing plants and estimate the capital and operating cost for the additional production from the integrated plants. From these data, recommend confirmation testing, if any, to the DOE and the coal company.

4. Confirm laboratory projections by continuous pilot-scale testing of the recommended application at the host preparation plant. Also, further optimize process conditions to obtain design parameters so that the coal company may assess feasibility of a plant conversion to the advanced cleaning process.

5. Determine dewatering, CWF preparation, and briquetting properties of fine clean coal from the pilot operation so that the marketing prospects of the additional coal production can be included in the coal company assessment.

Detailed accounts of how the steps were accomplished were presented in topical reports to the DOE [**R-2**, **R-3**, **R-4**] and in presentations to the CoalPrep 96 and CoalPrep 97 meetings [**P-3**, **P-7**, **P-8**]. Laboratory studies on an innovative hydrophobic dewatering process were also conducted to support the near-term effort [**R-5**].

Both of the physical fine-coal cleaning technologies being developed for production of premium fuel (advanced column flotation and selective agglomeration) were considered for near-term applications. Column flotation technology was considered to be ready for commercial fine coal recovery applications. However, only non-recovery systems with diesel fuel, kerosene or heating oil bridging liquids were considered for near-term applications of selective agglomeration since the technology for recycling volatile bridging liquids had not yet been developed much beyond the conceptual laboratory stage at the time this task began [**R-13**].

LOCATIONS FOR NEAR-TERM APPLICATIONS

As a first step, team-member Amax Coal Company was asked to suggest locations for application of the new technologies. Two locations were selected by the project team, the Ayrshire Preparation Plant in Indiana and the Lady Dunn Preparation plant in West Virginia. Later, when it was learned that the Ayrshire Mine would be closing, the Wabash Preparation Plant in Illinois was also included as a study site for the advanced technology.

Amax R&D and Bechtel engineers visited each of the locations to assess its suitability as a host site for eventual pilot-scale testing. At the same time, fine coal samples were collected for laboratory amenability tests at Amax R&D, Arcanum, CAER and CCMP. Bechtel engineers also obtained the existing plant layout and operating data that they would need for the conceptual-design and economic-feasibility studies.

Ayrshire Preparation Plant

The Ayrshire Mine and Preparation Plant northeast of Evansville produced surface mined coal from the Indiana VI seam. The preparation plant was a 1,200 st/h jigging operation originally placed into service in 1973. The minus 28-mesh underflow from the clean coal dewatering screens was cycloned, and the cyclone overflow discarded to a slurry pond. The cyclone underflow was dewatered with basket centrifuges and combined with the clean coal from the jig plant. A significant amount of clean coal was lost in the cyclone overflow, and the quality of the overall plant production was degraded by the excessive amounts of moisture and ash retained in the centrifuge cake. Product quality was an important consideration at Ayrshire since low sulfur coals were being purchased at the time to blend with the plant production in order to meet customer specifications.

The main focus of the near-term application was the 80 st/h (dry basis) of fine refuse going to the slurry pond. It was viewed as a potential source of low sulfur coal which could replace some of the coal being purchased as blending stock. In addition some attention was given to improving the quality of the centrifuge cake, perhaps by including a grinding step ahead of advanced cleaning.

Lady Dunn Preparation Plant

The Lady Dunn Preparation Plant east of Charleston, West Virginia, received Stockton and Eagle seam coal from a nearby underground mine. The plant had a heavy-media vessel/shaking table/mechanical-cell flotation circuit and had a capacity of 550 st/h when the task began in 1992. A multiphase expansion to 1200 st/h, involving replacement of the shaking tables with heavy-media cyclones and spiral separators was on the planning board at the time.

An initial evaluation of the plant operation indicated that the Lady Dunn plant could benefit from the use of advanced cleaning technology to treat the additional fines that would be generated by the expansion. Since the plant was being expanded anyway, the advanced cleaning units could be easily incorporated into the circuit. In addition to the production of a larger quantity and a better quality clean coal for steam coal customers, the advanced technology could also produce low-sulfur specialty fuels at that location. Manufacture of briquettes as a premium stoker fuel for industrial boilers would be a particularly attractive option. Other options would be clean coal sale as a powder fuel or a CWF for firing industrial boilers in the area.

The overflow stream from the classifying cyclones in the expanded plant was expected to contain 35 tons of dry solids per hour. At the time, plant management envisaged installation of additional mechanical flotation cells to clean this stream. Based upon past experience, they expected to achieve 50 percent recovery, at most, of the combustible material in the cyclone overflow. Vorsiv underflow slurry feeding the existing mechanical-cell flotation circuit was collected for amenability testing. Since the contemplated feed to the expanded plant included streams of coarser material than the minus 48-mesh Vorsiv product, a 28x100-mesh raw coal fraction was also prepared for separate testing.

Wabash Preparation Plant

The Wabash Mine and Preparation Plant near Keensburg, Illinois, produced coal from an underground mine in the Illinois No. 5 seam. The preparation plant was a 1,500-st/h heavy-media vessel/heavy-media cyclone operation that had been placed into service a few months earlier. The minus 1-mm fines from the heavy-media cyclone feed were cycloned with the plus 0.15-mm oversize going to spiral separators and the minus 0.15-mm overflow going to the refuse thickener and disposal. It was proposed to clean the latter stream by an advanced technology since coal recovered here would increase the overall production of saleable coal from the mine. Recent test data developed to support an unfunded Clean Coal Technology V proposal were available in the coal company files so it was not necessary to obtain new samples from the preparation plant for testing.

LABORATORY AMENABILITY TESTING

Portions of slurry samples collected at the Ayrshire and Lady Dunn Plants were distributed to team members for the initial amenability testing. Typical properties of these samples and of the Wabash fines are summarized below:

	Ayrshire		Lady Dunn		Wabash
	Fine Refuse	Centrifuge	Vorsiv U'flow	28x100m	-65m Fines
As Rec'd Solids, %	15.10	86.29	19.66	98.94	
Ash, % dry	64.47	26.79	34.39	30.56	23.90
Total Sulfur, % dry:	1.26	2.93	0.67	0.85	1.92
Particle Size, wt %:					
Passing 28 mesh	98.8	65.5	100.0	100.0	
Passing 48 mesh	96.3	26.8	>95.0		100.0
Passing 100 mesh	90.6	7.8	83.1	0.0	86.5
Passing 325 mesh	78.4	1.7	60.4	0.0	

Three-product washability tests were made on the Ayrshire and Lady Dunn samples [**R-4**], and significant amounts of good coal containing less than 11 percent ash were found in each sample. In addition, there was significant rejection of pyritic sulfur to the sink products, particularly in the case of the two Ayrshire samples.

Froth Flotation of Ayrshire Fines

Batch and continuous-flow laboratory flotation tests were performed on the Ayrshire fines to provide a quantitative basis for a feasibility analysis of an advanced column flotation application at that plant. An important objective of this testing was to determine the likely yield of clean coal and its quality and to obtain operating and plant design data to quantify parameters having an impact on process economics.

Denver cell tree-flotation tests yielded 90 percent recovery of the higher heating value in the respective samples. The clean coal from the fine refuse contained 10 percent ash and the clean coal from the centrifuge cake contained 12 percent ash. Grinding the centrifuge cake shifted the ash content of the resulting clean coal to the left to 8 percent ash. Flotation also reduced the SO_2 emission of the fine coal down to 3 lb/MBtu. The emission of the centrifuge cake was also reduced, but only to about 4 lb of SO_2 per MBtu.

CAER performed continuous flotation tests on the Ayrshire fine refuse using a 2-inch diameter generic Ken-Flote™ column with an internal aeration system. Parametric tests were conducted to investigate the effects of varying aeration, wash water and feed rates on Btu recovery and the ash content of the clean coal. The consensus of the laboratory flotation results was that a 10 percent ash product can be prepared from the Ayrshire fine refuse slurry at 95 percent recovery of the heating value in the slurry.

Selective Agglomeration of Ayrshire Fines

Arcanum performed batch laboratory-scale selective agglomeration separations on the Ayrshire coal fines using kerosene and diesel fuel as bridging liquids. Asphalt was mixed with the bridging liquid at times to activate agglomeration and serve as a binder.

At least 9.3 percent bridging liquid, on a MAF coal basis, was required to form pellets from the fine refuse that were large enough to be retained on a 100-mesh sieve. However, the microagglomerates which formed when adding 4.5 percent bridging liquid could be recovered by froth flotation. The clean agglomeration products prepared from the refuse stream contained between 4.24 and 7.53 percent ash, which was lower than the ash content of the comparable column flotation products.

The agglomerated product recovered from the centrifuge cake by froth flotation contained 10.23 percent ash. The agglomerated centrifuge products recovered by screening contained considerably more ash because coarse mineral matter was retained on the test sieve along with the pellets of clean coal.

It did not appear from these results that further selective agglomeration testing was warranted on the Ayrshire products. Because of the cost of the bridging liquid, the only combinations which seemed at all practical were those where the use of less than 5.0 percent bridging liquid was combined with froth flotation for collecting the agglomerated coal.

Froth Flotation of Lady Dunn Fines

Batch and continuous-flow laboratory flotation tests also were performed on Lady Dunn coal fines to provide a quantitative basis for feasibility studies on a near-term application at that plant. As with the Ayrshire application, it was important during this testing to project the likely yield of clean coal and its quality and to obtain operating and plant design data for parameters having an impact on process economics.

Denver cell flotation recovered around 90 percent of the heating value in the Vorsiv underflow feeding the existing mechanical cells. The ash content was reduced to 8 percent. Flotation also reduced the sulfur content of the coal by a small amount.

CAER performed continuous laboratory column flotation tests on the Lady Dunn Vorsiv undersize using the 2-inch diameter generic Ken-Flote™ column. Parametric tests were conducted on the slurry to investigate the effects of varying aeration, wash water and feed rates on Btu recovery and the ash content of the clean coal. The consensus of the column test results was that a 6 to 8 percent ash product could be prepared from the Lady Dunn Vorsiv underflow at 95 percent recovery of the Btu value in the slurry (or about 90 percent MAF coal recovery).

Two years later, and after the project team had evaluated the initial laboratory studies described above and found the proposed application to have merit, further laboratory testing was done on Lady Dunn cyclone overflow similar to the Vorsiv underflow described above. These tests were conducted at CCMP in a 2-inch Microcel™ column, the type of column proposed for installation during the plant expansion. A grade-recovery plot of the Microcel™ results showed 90 percent Btu recovery of coal containing slightly under 10 percent ash and generally agreed with the CAER observations.

Selective Agglomeration of Lady Dunn Vorsiv Underflow

Arcanum performed batch laboratory-scale selective agglomeration separations on the Lady Dunn Vorsiv underflow using kerosene and diesel fuel as bridging liquids. The Lady Dunn coal responded to selective agglomeration better than did the Ayrshire coal, so it was not necessary to add asphalt during these tests. Over 97 percent of the heating value was recovered from the 2.0 percent bridging liquid froth recovery tests, and the clean coals contained between 7.30 and 8.00 percent ash.

Froth Flotation of Wabash Fines

The decision to include advanced flotation of the natural fines in Illinois No. 5 coal as a near-term application was based upon laboratory and bench-scale testing that had been done a year earlier to support a Clean Coal Technology V submission. The amenability of these fines to froth flotation had been demonstrated at CCMP using 2-inch and 8-inch Microcel™ columns. The fine coal responded very well to the column flotation. Over 90 percent combustible recovery was achieved during one test where the resulting clean coal contained only 5.0 percent ash. A 55 percent rejection of pyrite, a particularly important consideration at Wabash, was especially noteworthy.

ECONOMIC AND TECHNICAL FEASIBILITY OF PROPOSED APPLICATIONS

Bechtel performed an engineering analysis of the economic and technical feasibility of the proposed near-term applications, that is, column flotation and selective agglomeration at Ayrshire and Lady Dunn and column flotation at Wabash [**R-3**]. They considered three marketing options for the clean coal to be produced by each application, namely 1) dewatered centrifuge cake blended with the existing production, 2) dry powder fuel produced from the centrifuge cake, and 3) briquettes produced from the dry powder fuel.

Capital and Processing Costs

During their analysis [**R-3**], Bechtel found that between 21 and 98.8 st/h of good quality clean coal would be produced by the proposed applications. This would be new production in the Ayrshire and Wabash cases. As part of the economic analysis, Bechtel estimated the cost of installing the advanced circuits in the three plant locations and projected the total processing costs for operating the circuits, including capital charges. The projected capital costs, in 1993 dollars, for installing the circuits in each plant were as follows:

	Ayrshire Plant		Lady Dunn Plant		Wabash
	Flotation	Agglomeration	Flotation	Agglomeration	Flotation
Advanced Cleaning	$3.66M	$6.80M	$1.50M	$1.60M	$9.16M
Thermal Drying	5.36M	4.22M	3.4M	1.7M	5.4M

Specific installation costs for briquetting circuits were not determined for the three plants since there was no test data available for specifying equipment selection parameters. An allowance for capital charges was included in the subsequent briquetting processing cost, though. Figure 37 compares the combined capital charges and operating and maintenance costs for producing clean coal at each location for each of the three marketing options.

Producing centrifuge cake for blending with current plant production was the lowest-cost option at each location. The cost of producing centrifuge cake after column flotation cleaning ranged from $5.63/st at Lady Dunn up to $8.73/st at Wabash. Column flotation was also less expensive than selective agglomeration at Ayrshire and Lady Dunn, although the difference was only $3.18/st at Lady Dunn. Most of the difference in cost between column flotation and selective agglomeration was due to O&M charges, especially for electric power and the fuel oil used as the bridging liquid. There was a larger difference between the two cleaning costs at Ayrshire because it was more difficult to agglomerate Ayrshire coal than it was to agglomerate Lady Dunn coal.

Drying added between $7.36/st and $10.65/st to the total processing cost at the three locations. From published reports and past Bechtel experience, it was estimated that briquetting the dried coal would add between $6.17/st and $7.26/st to the processing cost. This led to total processing costs between $24.02/st and $36.06/st for briquetted clean coal on a bone-dry basis. On a heating value basis, the total processing cost, including briquetting, ranged from $0.84/MBtu for flotation clean coal at Lady Dunn on up to $1.32/MBtu for agglomeration clean coal at Ayrshire.

Discussion of Economic Comparisons

The projected processing costs at the three locations, Ayrshire, Lady Dunn and Wabash, were not really comparable to each other since they reflected differences in site conditions, utility rates and feed material characteristics which were very site-specific. For example, the solids content of the feed slurry had a significant effect on the flotation cell volume and was a major factor affecting the capital and operating costs of the flotation circuits. To maintain an acceptable retention time in the columns, the required volume of the columns increased as the solids content of the feed slurry decreased.

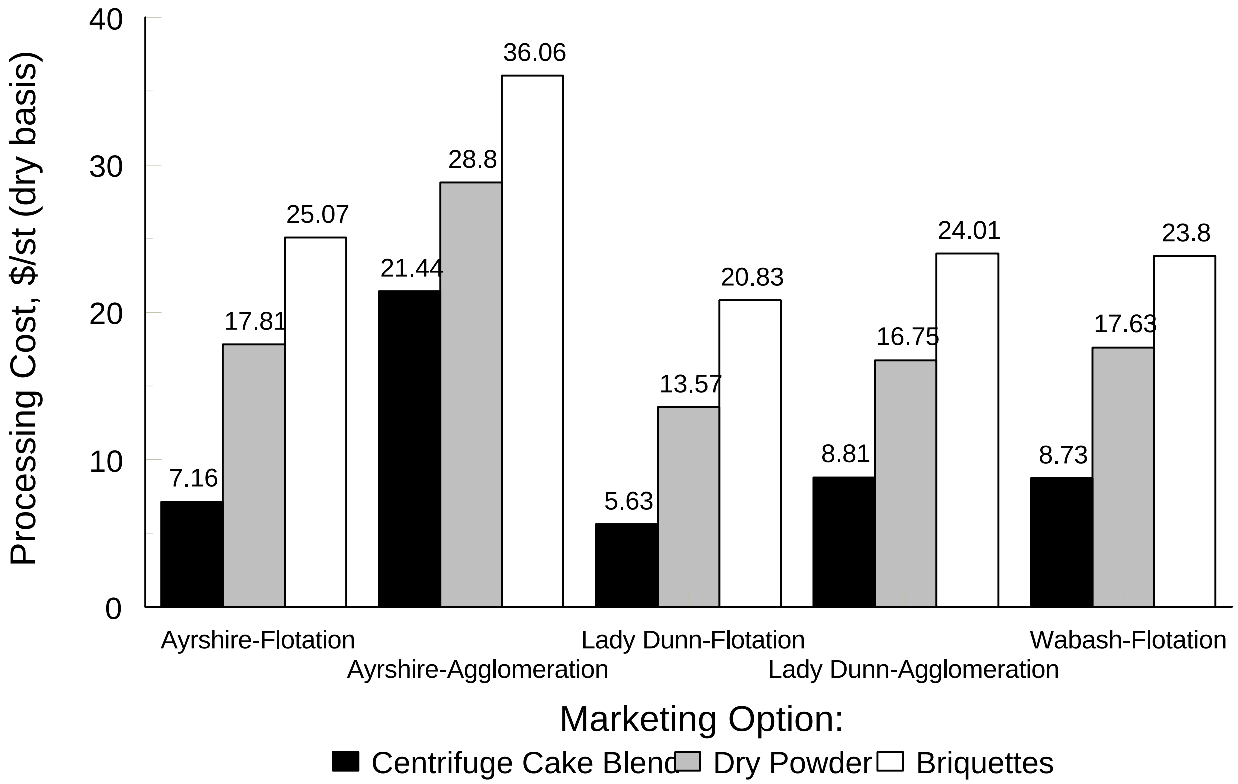


Figure 37. Processing Cost Summary for Plant Location and Marketing Options

(1993 Prices, Total Processing Cost includes Capital and O&M Charges but not the Cost of the Coal)

Screen-bowl centrifuges for dewatering the fine coal accounted for a major part of the capital charges. The Lady Dunn Plant had spare centrifuge capacity, and also thickener capacity, for use in the column flotation and selective agglomeration circuits. This was part of the reason that the Lady Dunn applications tended to be more attractive than similar applications at the other two plants. On the other hand, the Wabash column flotation application benefited from the larger scale of the operation, particularly with respect to the drying and briquetting circuits.

Flotation and agglomeration characteristics of the coal were other factors which affected processing costs. The Ayrshire fine refuse coal had poorer flotation and agglomeration properties than the Lady Dunn coal. This difference meant that extra flotation and shearing/mixing times were needed. Also, twice as much bridging liquid was required for agglomerating the coal in the Ayrshire fine refuse as needed for the Lady Dunn fines. For these reasons the Ayrshire applications were somewhat less attractive cost-wise than the Lady Dunn and Wabash applications.

Recommendation of Team

Bechtel also performed a technical assessment of the processing options. They generally found that the benefits of the applications, particularly of the column flotation options, were quite attractive compared to the risks involved. In view of the encouraging economic and technical assessment of the column flotation near-term applications, the project team with strong support from Cyprus Amax Coal Company recommended larger-scale column flotation testing at the Lady Dunn Preparation Plant.

COLUMN FLOTATION TESTING AT LADY DUNN PREPARATION PLANT

In response to the favorable assessment of column flotation by the project team, pilot testing of a 30-inch diameter Microcel^{TM} column began at the Lady Dunn preparation plant in June, 1995 in order to confirm the laboratory results and to obtain additional scale-up information. The recovery of the coarser particle sizes of coal, that is, coal particles in the 0.25 to 0.75 mm range, was of particular interest during this work. A detailed presentation of the 30-inch column testing is contained in the topical report for this task [**R-4].**

The Lady Dunn flotation feed typically contained around 40 percent ash and had a high percentage of minus 325 mesh coal and clay in the slurry. Column performance was evaluated from the percentage recovery of the coal in various particle-size fractions and from the ash content of the products. Operating parameters such as feed rate, aeration rate, frother and collector dosage were varied to determine their effects on the recovery of the various particle sizes of coal. The plant had existing mechanical flotation cells so the test results could be directly compared to conventional technology.

A 30-inch diameter column was chosen for the study because that column diameter would provide a reasonable froth travel distance to allow time for coarser particles to drop out of the froth zone back down into the slurry as one would expect in a full-size column.

The Lady Dunn Plant

At the time of the pilot testing, the Lady Dunn Plant was mid-way through an expansion program. The flowsheet consisted of heavy-media vessels for coarse coal (plus 1/4 inch), heavy-media cyclones and Deister tables for 1/4-inch x 100-mesh coal, and conventional flotation on the minus 100-mesh overflow from desliming cyclones. A stream of minus 1-mm raw coal screen undersize stream was selected for the bulk of the parametric testing.

30-inch Column Circuit Description and Operation

The 30-inch Microcel™ test column had a capacity of 0.5 to 1 tph of clean coal for most applications. Microbubbles were created by injecting air into tailings slurry pumped through in-line mixers back into the bottom of the column. Wash water was added at the top of the column and also as push water in the overflow launder. The system was fitted with instrumentation for measuring and controlling wash water, air and slurry flows and the pulp level in the column. The instrumentation also provided an indication of the air fraction in the column. A sight glass provided a view of the pulp/froth interface area for assessment of turbulence, approximate bubble size, and excess air flow.

Testing Results and Discussion

Information was gathered from preliminary testing and from two series of parametric tests [**R-4**]. The feed for the preliminary testing was from the classifying cyclone overflow stream. Results were excellent and compared well with the earlier laboratory results as seen in Figure 38.

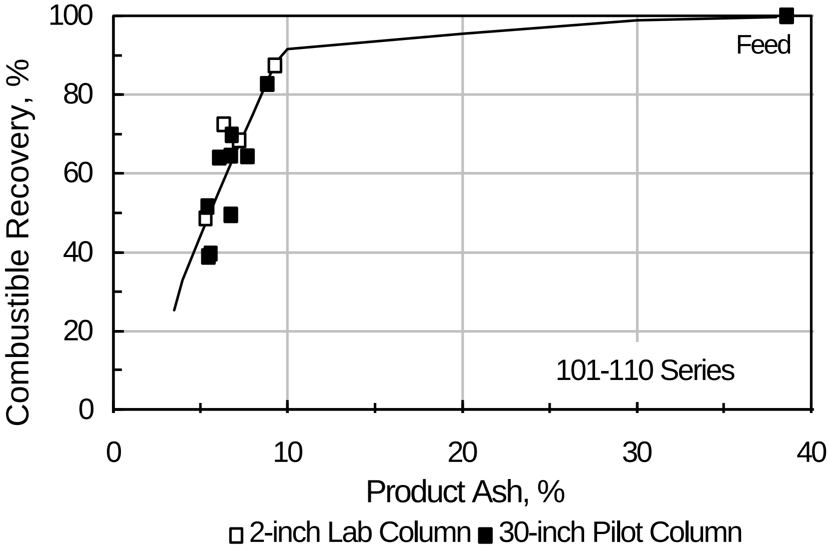


Figure 38. Comparison of 2-inch and 30-inch Microcel™ Column Flotation Results

Parametric Tests - First Series

The intent of the initial series was to vary key operating variables from low to high in order to determine likely operating points for the second series. In effect though, the first set of results provided a more consistent data set than the second. Good correlations were found and definite trends were seen when the main parameters (i.e. frother dosage, collector dosage, and feed rate solids) were analyzed.

Nearly all of the results from the first series fell along a single grade-recovery curve for each particle size range (Figure 39). Results move along an existing grade-recovery curve due to variations in the loading of the bubbles (i.e., space available for attachment). Changes in the specific characteristics of the coal particles (i.e., degree of liberation and hydrophobicity) result in new grade-recovery curves. The close fit to a

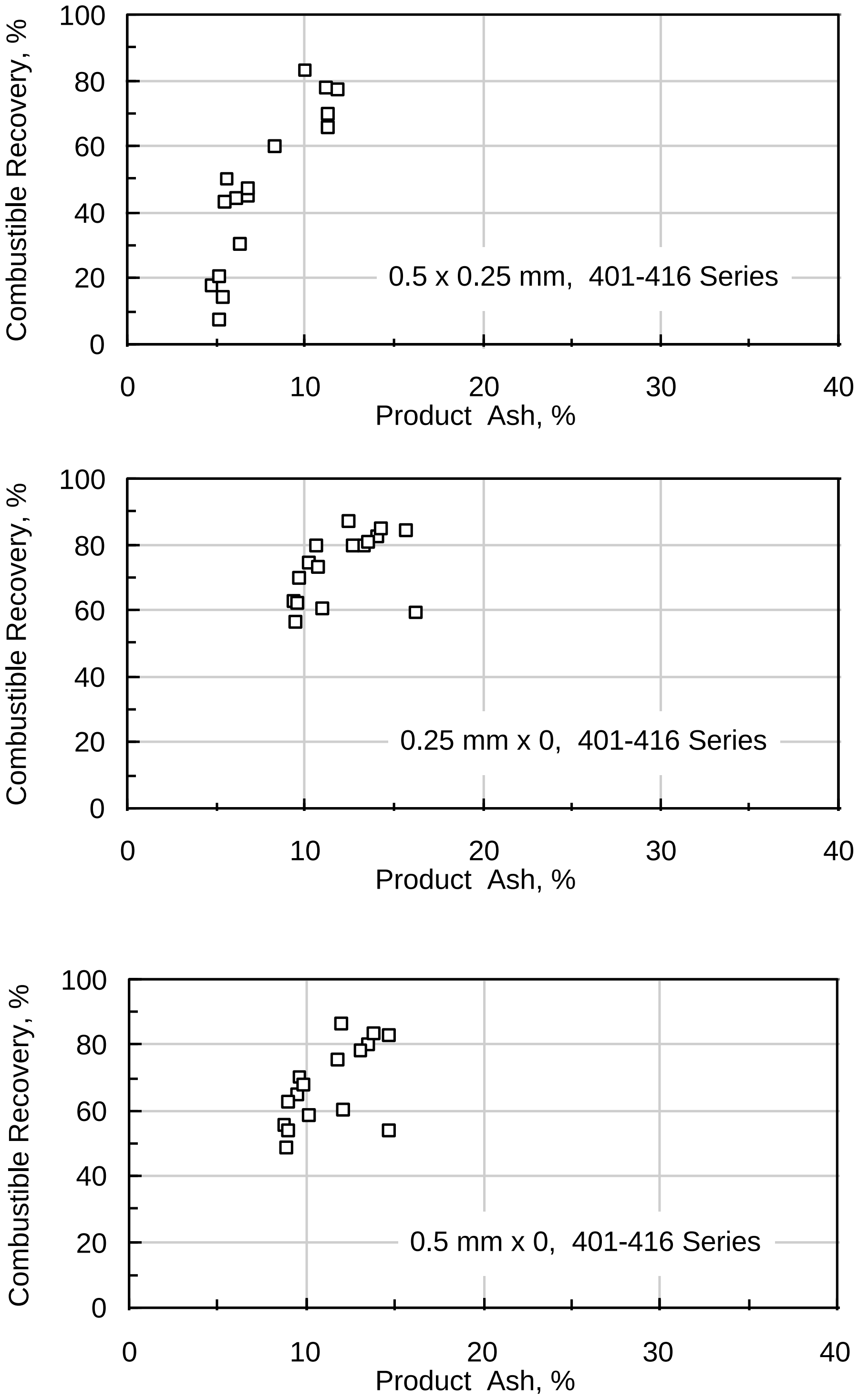


Figure 39. Grade Recovery Plots for Parametric Flotation of Lady Dunn Fines

common grade-recovery curve indicated that entrainment of non-floatable material in the froth was not a problem during this series of tests. In other words, the wash water flow was sufficient to remove entrained high-ash particles from the froth zone.

The best recovery seen during each test was for the 0.150 x 0.045 mm (100 x 325 mesh) size fraction, and the 0.045 mm x 0 recovery was always slightly less as shown in Figure 40. Most often combustible recovery dropped off at the coarser sizes. The parameters that had the most impact on combustible recovery were feed rate, frother dosage, and diesel fuel dosage.

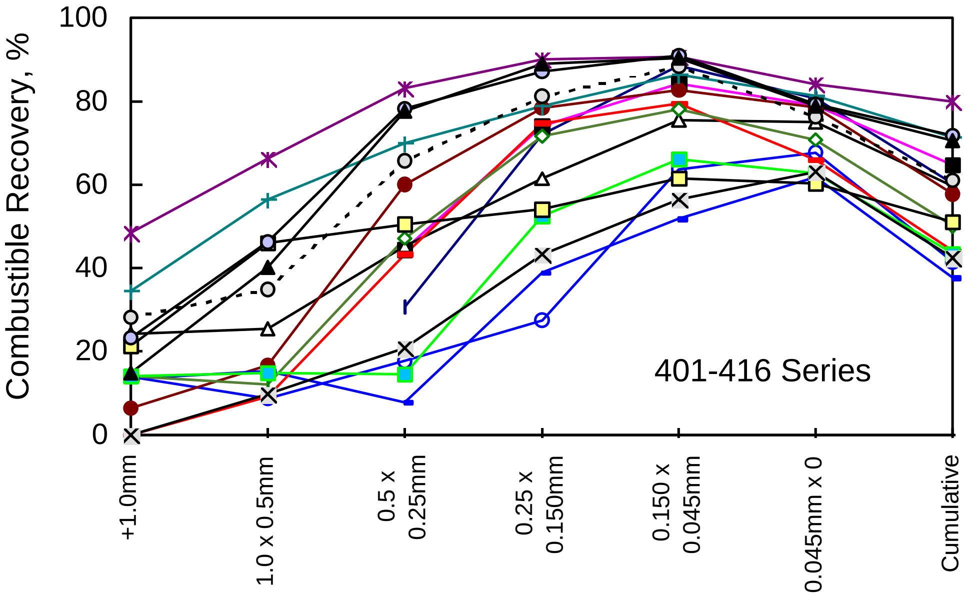


Figure 40. Combustible Recovery by Particle Size

The data analysis showed that at low frother dosages, increased feed rate reduced combustible recovery from the 0.25 mm x 0 fraction. This was expected since the bigger bubbles which formed under those conditions were quickly overloaded since their limited surface area restricted their carrying capacity for coal particles. The same performance was shown for the medium frother dosage except that at the higher feed rate, recovery improved over that seen with the lower frother dosage. At a high frother dosage little change in recovery was noticed with changes in feed rate, indicating sufficient bubble surface area for carrying the full range of particle sizes available in the feed slurry.

Variation in the diesel fuel dosage had little effect on the flotation of 0.25 mm x 0 coal except for some improvement in recovery at higher frother dosages. When taken together the smaller bubble size and the increased collector dosage improved the flotation rate constant and provided the extra carrying capacity needed to collect middlings particles that did not previously have an opportunity to float.

There were differences in the flotation of the coarser 0.50 x 0.25 mm particle size range coal. At the lower frother dose, combustible recovery was highest at the low feed rate just as for the flotation of the smaller particles. Unlike the finer sizes, however, diesel fuel dosage had a major effect upon the recovery of the coarse coal. At low frother and low feed rates, the recovery actually dropped with increased diesel fuel addition. This probably was because the excess diesel fuel depressed froth formation which resulted in larger bubbles with less surface area. In such cases, the fine coal particles preferentially stuck to the bubbles blocking the coarser particles from access to the bubbles.

At the medium frother dosage and a low diesel fuel dosage, the relationship between feed rate and recovery was similar to that of the low frother dosage; that is, increased feed rate meant lower recovery. At the low feed rate, increasing the diesel fuel dosage lowered recovery due to the decreased effectiveness of the frother. At the highest diesel dosage the recovery increased again due to the increased particle hydrophobicity brought about by the large amount of collector available in the slurry.

At a high frother dosage, combustible recovery was affected only by the diesel fuel dosage. At the low diesel fuel dosage, the recovery of coarse coal was depressed by the excess frother. Maximum recovery was projected, though, at higher diesel fuel dosages with the high frother dosage.

Overall, it was shown that 0.50 x 0.25 mm coal can be recovered at specific conditions nearly as well as the finer coal. The actual size-by-size results also show why flotation is seldom utilized to recover coal above 0.50 mm in particle size. Even with the best combination of parameters, the combustible recovery began to drop off rapidly as the size of the particles increased above 0.50 mm.

Parametric Tests - Second Series

The intent of the second series of parametric testing was to further determine the effect of bubble size and air fraction on coarse coal recovery. To do this, air volume and frother dosage were varied. Feed rate was a third variable.

The intent of the second series was to remove diesel fuel dosage as a variable by holding it relatively constant. Due to variations in plant operation, screen wear in the feed preparation system, and raw coal pumping surges, though, the actual percent solids in the column feed varied considerably and had an unintended impact on the actual diesel fuel dosage. Although the volumetric dosage of diesel fuel was held constant for a given feed flow, the pounds per ton of feed dosage varied with the percent solids changes. Since the diesel fuel tended to coat the fine coal particles first, with only the remaining oil being available to coat coarser particles, any variation in the fraction of finer coal in the feed caused the amount of diesel fuel available for coating the coarse particles to vary considerably. As a result, the original results of the test series did not appear to be consistent with earlier results.

After extensive review and cross plotting of the operating parameters, the question of the inconsistent results from the second series was resolved. The major problem stemmed from the variation in the percent solids of the feed slurry. Above a threshold dosage of diesel fuel (around 1200 g/T for this system), the air fraction dropped rapidly. The decrease in the air fraction from the 10 to 13 percent range down to below 4 percent indicated formation of much larger air bubbles with less surface area for attachment of particles. Since the fine coal particles were more strongly attached to the bubble surfaces than were the coarse particles, the coarser particles were the first to be lost at high bubble loadings. The larger bubbles may also have caused increased turbulence that resulted in the coarse particles becoming detached from the bubbles.

The data analysis was repeated using diesel fuel dosage, frother dosage, and feed rate as variables. Changes in 0.25 mm x 0 coal recovery due to differences in frother dosage were again found to be small. The best performance was at the medium frother dosage while the lowest recovery was found at the extremes of high diesel fuel dosage, high feed rate, and low frother dosage. At the low feed rate, the diesel fuel dosage accounted for a slight increase in recovery at all but the lowest frother dosage.

Combustible recovery from the 0.50 x 0.25 mm fraction was shown to have a much broader range of response in the revised evaluation. A change in recovery at low diesel fuel dosages was the most significant variation observed. Recovery dropped considerably at all feed rates with the increasing frother dosage. At the higher frother dosages, increasing the diesel fuel dosage improved the recovery of the coarse coal by overcoming the effects of the excess frother. An unexpected response was the increase in recovery with increasing feed rate and low diesel dosage.

Conclusions of Pilot Testing

The test work in the 30-inch column illustrated very well the potential for coarse coal flotation in a properly operated system. Particles up to 0.25 mm in size floated consistently well. Coarse coal up to 0.50 mm in size also floated well, but coal recovery dropped off rapidly above that size. Since it is difficult to avoid misplaced material when separating fine particle sizes, making a nominal 0.25-mm cut and sending the minus 0.25-mm fraction to a flotation column should work well in most coal processing plants. As long as the misplaced coarse material in the feed slurry is smaller than 0.50 mm, the column can provide very good recovery of coal with a low ash content.

In traditional coal processing plant applications, the particle-size cut ahead of flotation is made at a nominal 0.150 mm (100 mesh). The difficulty in making such a fine cut results in a considerable amount of fine material (usually high in ash) remaining in the coarser fraction. In all gravity separation devices designed for cleaning plus 0.150-mm material, much of this minus 0.150-mm material reports to the clean coal launder without cleaning (i.e., as high-ash coal). Thus, the difficulty in removing fines from clean coal streams results in higher ash final products. By utilizing wash water, a flotation column can remove the high ash slimes that otherwise would be entrained in the froth. Therefore, it can handle slimes better than other cleaning devices readily available to preparation plant operators, yet it can still clean plus 0.25-mm particles which do not respond well to conventional flotation.

All indications were that column flotation would perform well at the Lady Dunn Plant. The original mechanical flotation cells produced an average of 14 to 16 percent ash clean coal at a 20 percent combustible recovery. Results from testing the 30-inch diameter column, on the other hand, indicated that clean coal containing 10 to 11 percent ash can be obtained from the 0.25-mm x 0 fines at a combustible recovery of 75 percent.

The success of this test work was made tangible by the installation of three Microcel^{TM} flotation columns, each four meters in diameter, in the Lady Dunn Preparation Plant [**P-3**]. These are the largest known flotation columns for cleaning coal. Cyprus Amax Coal Company installed the columns in the plant on the basis of the good results achieved by the test work described in this report. The new columns have been successfully cleaning 0.25-mm x 0 coal and producing results that fit on the ash/recovery curves seen during the pilot testing [**R-4**].

AUXILIARY OPERATIONS

The pilot scale flotation investigation was supplemented with laboratory and bench-scale studies to dewater the clean coal froth from the 30-inch column and also to improve its marketability by conversion to CWF slurry fuel and by briquetting to a lump fuel [**R-4**].

Dewatering

Clean coal slurry from the 30-inch column testing was shipped to the Federal Energy Technology Center at Pittsburgh for centrifuge dewatering tests using their GranuFlow process. The GranuFlow process involved mixing an asphalt emulsion called Orimulsion with the coal slurry before dewatering in order to reduce the amount of moisture remaining in the cake. The handling properties of the fine coal were also improved. Performance of screen-bowl and solid-bowl centrifuges were compared, and cakes with the following percentage moisture contents were obtained:

	No Additive	6 - 8 % Orimulsion
Screen Bowl	39.4	35.2-35.7
Solid Bowl	34.8	31.0

The Orimulsion additions were also found to reduce the potential dustiness of the fine coal as measured by the amount of minus 100-µm material released when sieving dried centrifuge cakes. The Orimulsion additions improved solids recovery in the screen-bowl centrifuge as well.

In addition to the centrifuge testing, 122 laboratory vacuum filtration leaf tests were conducted on froth from the 30-inch column by Westech Engineering Inc. personnel. The objective of the leaf testing was to project the capacity and performance of both top-feed horizontal belt filters and bottom-feed drum filters. The laboratory evaluation included testing the benefits of layering spiral concentrate (available from a separate project at Lady Dunn) onto a horizontal filter ahead of the froth slurry.

Because of the residual clay in the Lady Dunn clean coal slurries, preflocculation was required to achieve good filtration performance. Severe filter cloth blinding occurred after a few tests so it was necessary to include a cloth washing step in the filtration cycles.

There were some ambiguities among the capacity and cake moisture projections which may have been due to the differing amounts of flocculant required for each situation. Filtering coarse spiral concentrate along with the froth slurry was found to offer little advantage with respect to capacity or moisture removal. A horizontal belt filter did appear to offer a somewhat higher capacity on a lb/h/sq ft basis than a drum belt filter, but the moisture contents of the resulting cakes were about the same, that is, in the 34 to 43 percent range. Because these cake moistures were similar to the centrifuge cake moistures, Lady Dunn management decided to continue with their original plan to use a screen-bowl centrifuge for dewatering the column flotation froth after the plant expansion.

Hydrophobic Dewatering

The hydrophobic dewatering is an innovative process for dewatering fine coal that is being developed at the Center for Coal and Mineral Processing at Virginia Tech. During the process liquid butane is mixed with a coal slurry such as the froth from column flotation [**R-5**]. The butane displaces the water from the particle surfaces so that the butane and coal particles float to the surface of the slurry where they may be separated from the water phase. Evaporation of the butane from the floating solids left coal containing as little as 1 percent moisture during the laboratory testing [**R-5**]. It is expected that the butane vapor would be recovered for reuse so that the process would be economically attractive alternative to the thermal drying of fine coal.

Slurry Preparation

Marketing clean coal from near-term column flotation as slurry fuel rather than as filter cake or centrifuge cake was considered. Slurry preparation tests were performed on froth slurry from the Microcel™ testing at the Lady Dunn plant. The tests were on the froth slurry alone and on froth slurry blended with coarser slurry prepared by stage grinding spiral concentrate to minus 48 mesh. It was found that at a projected viscosity of 500 cP, slurry loadings of 62, 63 and 68 percent coal could be achieved for blends containing 0, 10 and 40 percent, respectively, of the ground spiral concentrate. In each case, the slurry contained one percent A-23M dispersant on a dry coal basis.

These results indicate that if a niche market were found in the Charleston area, one might sell the fine coal from the Lady Dunn plant as a slurry fuel containing about 60 percent coal. However, it appears at this time that dewatering the fines in a centrifuge and blending the cake with the normal plant production is the better alternative in terms of cost and marketability.

Briquetting

A portion of the clean coal from the 30-inch column testing was submitted to TraDet Inc. for binderless briquetting tests. Good quality specimens of the briquette production were returned by TraDet, who reported that the briquetting was done at near-ambient temperature on the flotation product after it had been air-dried to between 1.0 and 2.4 percent moisture. The briquettes contained 11.8 percent ash and 34.2 percent volatile matter and had a heating value of 12,900 Btu/lb.

A model B-100A Komarek laboratory roll-press machine was used. The rolls were preheated to equilibrium operating temperature by briquetting waste material before switching to the test coal. Parametric tests were made at three roll speeds and at five hydraulic roll pressures between 1,300 and 2,800 psig on batches of the coal that had been dried to four differing moisture levels. At the product temperatures of 128° to 178° F, these pressures deform coal particles and fuse them together.

The crush strengths of the briquettes were between 50 and 200 lbs, and these strengths correlated well with the amount of energy transferred to the briquettes (between 8 and 29 kWh/ton) as shown in Figure 41. TraDet considers any strength over 100 lbs to be acceptable for briquettes such as these. The best briquettes were made when the feed coal had been dried to 1.0 percent moisture. The briquetted products from all 58 tests had acceptable moisture reabsorption, weathering and degradation properties. Based upon these results, TraDet suggested follow-up optimization testing in a pilot-size machine to allow scale-up of the laboratory briquetting performance to commercial production units.

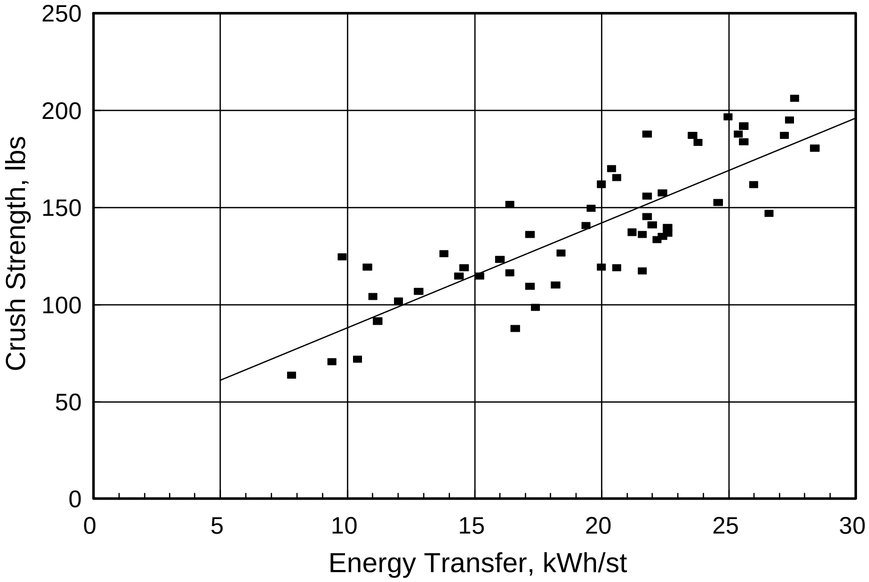


Figure 41. Strength of Briquettes vs Energy Transfer During Compaction

Note: Energy transfer equals energy input to feed screw and roll drive less energy consumed by the evaporation of moisture during compaction.

CONCLUSIONS AND RECOMMENDATIONS FOR NEAR-TERM APPLICATIONS

The conceptual engineering analysis of the laboratory column flotation and selective agglomeration test results and the confirmation bench-scale and pilot testing of column flotation showed that advanced physical fine-coal cleaning processes can be advantageously integrated into existing coal preparation plants. The following observations were made regarding this work:

- Column flotation can recover a lower-ash clean coal than the usual mechanical-cell flotation and at a higher recovery of combustibles. The following example is for the Lady Dunn application:
    - Microcel™ column – 10 to 11 percent ash clean coal, 75 percent recovery
    - Mechanical cells – 14 to 16 percent ash clean coal, 20 percent recovery
- Column flotation can be effectively applied to streams containing coal as coarse as 0.5 mm and, less effectively, as coarse as 1.0 mm.
- High-pressure binderless bench-scale briquetting was effective for reconstituting the clean coal.
- Selective agglomeration performance projected from laboratory testing was similar to or somewhat better than the performance of column flotation.

- Projected near-term application costs for producing dewatered clean coal by column flotation of raw coal fines were in the $5.60 to $8.70 per dry short ton range.
- Projected near-term application costs for producing dewatered clean coal by selective agglomeration with a non-recoverable bridging liquid such as diesel fuel were significantly higher than the projected cost of recovering the clean coal by column flotation. Selective agglomeration was particularly less competitive when cleaning midwestern Ayrshire coal which did not agglomerate as easily as the eastern Lady Dunn coal.
- Thermal drying of the clean coal for blending with the existing plant production or for separate sale as powder fuel adds $7.60 to $10.60 per short ton to the production cost of the coal recovered by advanced cleaning.
- The total projected cost of producing briquetted fuel (but not including the cost of the raw coal fines) was less than $25.10 per short ton for four of the five near-term applications evaluated.

The following recommendations are offered to operators of coal preparation plants:

- Advanced physical fine coal cleaning options should be considered for installation in new plants and when refurbishing or expanding existing plants. It is likely that additional revenue can be generated over the revenues from the “no fine coal cleaning” or the “mechanical-cell flotation” options.
- In order to reduce costs, agglomeration with recoverable bridging liquids such as heptane and pentane, should be explored as alternatives to fuel oil and diesel fuel.

Methods for improving the marketability of the recovered fine coal, such as GranuFlow processing, conversion to CWF, powder fuel, and especially binderless briquetting, should be developed further.

12. CONCLUDING REMARKS

This project is an important milestone on the way to the commercial production of coal-based premium fuel as a replacement for oil and gas fired in some utility and industrial boilers. Much has been accomplished by this project and much learned, particularly with regard to grinding coal and operation of advanced flotation and selective agglomeration circuits. The recovery and reuse of the selective agglomeration bridging liquid had not been done before at this scale. These accomplishments allow one to reach some important conclusions:

- There are coals available in the United States backed by large reserves that can be finely ground and cleaned to meet the premium-fuel specifications of less than 2 lb ash per million Btu and less than 0.6 lb sulfur per million Btu.
- The advanced column flotation and selective agglomeration physical fine coal cleaning processes are capable of recovering 80 to 90 percent of the heating value in available ROM coal while producing clean coal meeting the premium-fuel ash and sulfur specifications.
- The column flotation and selective agglomeration equipment and processes are robust and reliable for producing the target yield and quality of clean coal as demonstrated by processing over 2,100 tons of coal from three different mines through the 2 t/h integrated process development unit (PDU).
- If desired, the clean coal can be formulated into usable coal water slurry fuel with a heating value of 8,500 to 9,100 Btu/lb.
- Advanced physical fine coal cleaning rejects certain toxic trace elements from coal and could be part of a strategy to control hazardous air pollutant emissions from coal burning boilers.
- The production of premium fuel from coal in a Midwestern industrial area by either cleaning technology would cost less than $2.50/MBtu, in other words, the cost would be competitive with the cost of buying fuel oil.
- Advanced column flotation can and has been applied effectively in existing preparation plants for recovering minus 28 mesh fine coal that would otherwise be lost to refuse.

13. PROJECT REPORTS AND PRESENTATIONS

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

PROJECT REPORTS TO U.S. DEPARTMENT OF ENERGY

PHASE I Engineering Analysis and Laboratory and Bench-Scale R&D

TASK 2: Coal Selection and Procurement

R-1. **“**Task 2. Coal Selection and Procurement, Subtask 2.1 Coal Selection Plan and Recommendations,” ***Amax R&D Report to the U. S. Department of Energy Contract DE-AC22-92PC92208***, April 29, 1993 (by F. J. Smit and M. C. Jha).

TASK 3: Development of Near-Term Applications

R-2. “Task 3. Development of Near-Term Applications, Subtask 3.1 Engineering Analysis -- Engineering Development and Test Plan,” ***Amax R&D Report to Department of Energy Contract DE-AC22-92PC92208***, March 31, 1993 (by M. C. Jha, F. J. Smit, M. V. Chari and H. Huettenhain).

R-3. “Task 3. Development of Near-Term Applications, Subtask 3.1 Engineering Analysis -- Conceptual Designs and Cost Estimates,” ***Bechtel Report to Amax R&D Department of Energy Contract DE-AC22-92PC92208***, November 5, 1993 (by Process Programs Research and Engineering, Bechtel Corporation).

R-4. “Task 3. Development of Near-Term Applications, Subtask 3.2 Engineering Development,” ***Amax R&D Topical Report to the U. S. Department of Energy Contract DE-AC22-92PC92208***, April 25, 1997 (by F. J. Smit, M. C. Jha, D. I. Phillips and R.-H. Yoon).

R-5. “Hydrophobic Dewatering of Fine Coal,“ **Center for Coal and Minerals Processing *Topical Report to the U. S. Department of Energy Contract DE-AC22-92PC92208***, (for Task 3. Development of Near-Term Applications, Subtask 3.3 Dewatering), July 17, 1997 (by R.-H. Yoon, S. Sohn, J. Luttrell and D. I. Phillips).

TASK 4: Engineering Development of Advanced Froth Flotation for Premium Fuels

R-6. “Task 4. Engineering Development of Advanced Froth Flotation for Premium Fuels, Subtask 4.1 Grinding,” ***Amax R&D Topical Report to the U. S. Department of Energy Contract DE-AC22-92PC92208***, March 29, 1994 (by F. J. Smit, R. F. Hogsett and M. C. Jha).

R-7. “Task 4. Engineering Development of Advanced Froth Flotation for Premium Fuels, Subtask 4.2 Process Optimization Research,” ***Amax R&D Topical Report to the U. S. Department of Energy Contract DE-AC22-92PC92208***, March 16, 1995 (by F. J. Smit, M. C. Jha, B. K. Parekh, J. G. Groppo, G. H. Luttrell, M. T. Vencill and R.-H. Yoon).

R-8 “Task 4. Engineering Development of Advanced Froth Flotation for Premium Fuels, Subtask 4.3 CWF Formulation Studies,” ***Amax R&D Topical Report to the U. S. Department of Energy Contract DE-AC22-92PC92208***, May 23, 1995 (by N. Moro, F. J. Smit and M. C. Jha).

R-9. “Task 4. Engineering Development of Advanced Froth Flotation for Premium Fuels, Subtask 4.4 Bench-Scale Testing and Process Scale-up,” ***Amax R&D Topical Report to the U. S. Department of Energy Contract DE-AC22-92PC92208***, February 6, 1996 (by F. J. Smit, G. L. Shields and M. C. Jha).

R-10. “Task 4. Engineering Development of Advanced Froth Flotation for Premium Fuels, Subtask 4.5 Conceptual Design of Process Development Unit and Advanced Froth Flotation Module -- Conceptual Engineering Package,” ***Bechtel Report to Amax R&D / U. S. Department of Energy Contract DE-AC22-92PC92208***, December 10, 1993 (by Process Programs Research and Engineering, Bechtel Corporation).

TASK 5: Detailed Design of Process Development Unit and Advanced Froth Flotation Module

R-11. “Task 5. Detailed Design of Process Development Unit and Advanced Froth Flotation Module,” ***Bechtel Report to Amax R&D / U. S. Department of Energy Contract DE-AC22-92PC92208***, August 1995 (by Process Programs Research and Engineering, Bechtel Corporation).

TASK 6: Selective Agglomeration Laboratory Research and Engineering Development for Premium Fuels

R-12. “Task 6. Selective Agglomeration Laboratory Research and Engineering Development for Premium Fuels, Subtask 6.1 Agglomeration Agent Selection,” ***Amax R&D Topical Report to the U. S. Department of Energy Contract DE-AC22-92PC92208***, June 24, 1993 (by F. J. Smit, M. C. Jha, J. A. Getsoian and D. V. Keller, Jr.).

R-13. “Task 6. Selective Agglomeration Laboratory Research and Engineering Development for Premium Fuels, Subtask 6.2 Grinding,” ***Amax R&D Topical Report to the U. S. Department of Energy Contract DE-AC22-92PC92208***, August 11, 1994 (by F. J. Smit and M. C. Jha).

R-14. "Task 6. Selective Agglomeration Laboratory Research and Engineering Development for Premium Fuels, Subtask 6.3 Process Optimization Research," ***Amax R&D Topical Report to the U. S. Department of Energy Contract DE-AC22-92PC92208***, September 28, 1995 (by F. J. Smit, M. C. Jha, D. V. Keller, Jr. and J. A. Getsoian).

R-15. "Task 6. Selective Agglomeration Laboratory Research and Engineering Development for Premium Fuels, Subtask 6.4 CWF Formulation Studies," ***Amax R&D Topical Report to the U. S. Department of Energy Contract DE-AC22-92PC92208***, November 25, 1996 (by F. J. Smit, N. Moro, M. C. Jha and J. P. Dooher).

R-16. "Task 6. Selective Agglomeration Laboratory Research and Engineering Development for Premium Fuels, Subtask 6.5 Selective Agglomeration Bench-Scale Testing and Process Scale-up," ***Amax R&D Topical Report to the U. S. Department of Energy Contract DE-AC22-92PC92208***, June 27, 1997 (by N. Moro and M. C. Jha).

R-17. "Task 6. Selective Agglomeration Laboratory Research and Engineering Development for Premium Fuels, Subtask 6.6 Conceptual Design of Selective Agglomeration Module," ***Bechtel Task Report to Amax R&D / U. S. Department of Energy Contract DE-AC22-92PC92208***, August 31, 1994 (by Process Programs Research and Engineering, Bechtel Corporation).

TASK 7: Detailed Design of Process Development Unit and Selective Agglomeration Module

R-18. "Task 7.0 Detailed Design of Process Development Unit and Selective Agglomeration Module," ***Bechtel Report to Amax R&D / U. S. Department of Energy Contract DE-AC22-92PC92208***, August 1996 (by Technology & Consulting, Bechtel Corporation).

PHASE II PDU and Advanced Column Flotation Module Testing and Evaluation

TASK 8: PDU and Advanced Column Flotation Module

R-19. "Task 8. PDU and Advanced Column Flotation Module, Subtask 8.1 PDU Coal Selection Recommendations," ***Amax R&D Topical Report to the U. S. Department of Energy Contract DE-AC22-92PC92208***, August 17, 1995 (by F. J. Smit and M. C. Jha).

R-20. "Task 8. PDU and Advanced Column Flotation Module, Subtask 8.3 PDU and Advanced Coal Cleaning Module Shakedown and Test Plan," ***Amax R&D Report to the U. S. Department of Energy Contract DE-AC22-92PC92208***, December 14, 1995 (by G. L. Shields, F. J. Smit and M. C. Jha).

R-21. "Task 8. PDU and Advanced Column Flotation Module, Subtask 8.5 Froth Flotation Topical Report," ***Amax R&D Topical Report to the U. S. Department of Energy Contract DE-AC22-92PC92208***, August 28, 1997 (by G. L. Shields, F. J. Smit and M. C. Jha).

PHASE III Selective Agglomeration Module Testing and Evaluation

TASK 9: Selective Agglomeration Module

R-22. "Task 9. Selective Agglomeration Module, Subtask 9.2 Selective Agglomeration Shakedown and Test Plan," ***Amax R&D Report to the U. S. Department of Energy Contract DE-AC22-92PC92208***, December 31, 1996 (by N. Moro, G. L. Shields and M. C. Jha).

R-23. "Task 9. Selective Agglomeration Module, Subtask 9.4 Selective Agglomeration Topical Report," ***Amax R&D Topical Report to the U. S. Department of Energy Contract DE-AC22-92PC92208***, September 29, 1997 (by N. Moro, G. L. Shields and M. C. Jha).

PHASE IV PDU Final Disposition

TASK 10: Disposition of the PDU

R-24. "Task 10. Disposition of the PDU, PDU Decommissioning Plan," ***Amax R&D Report to the U. S. Department of Energy Contract DE-AC22-92PC92208***, August 22, 1997 (by N. Moro and M. C. Jha).

TASK 11: Project Final Report

R-25. "Commercial CWF Production Study," ***Bechtel Corporation Technology & Consulting Topical Report to Amax R&D / U. S. Department of Energy Contract DE-AC22-92PC92208***, June 1997 (by H. Huettenhain and M. K. V. Chari).

R-26 "Task 11 Project Final Report," ***Amax R&D Report to the U. S. Department of Energy Contract DE-AC22-92PC92208***, September 26, 1997 (by M. C. Jha, F. J. Smit, N. Moro, and G. L. Shields).

Also

Sixty ***Monthly Status Reports***, nineteen ***Quarterly Technical Progress Reports*** and four annual **Management Plans** to the DOE on Contract DE-AC22-92PC92208 and ***Test Plans*** for Subtasks 4.1, 4.2, 4.3, 4.4, 6.2, 6.3, 6.4 and 6.5.

PUBLICATIONS AND PRESENTATIONS:

P-1. “Reduction of Trace Element Concentrations by Physically Cleaning Three Finely-Ground Coals,” submitted to ***Coal Preparation*** (by F. J. Smit, M. C. Jha, G. L. Shields, N. Moro and T. J. Feeley, III)

P-2. "Premium Fuel Development," ***Presentation, Coal Liquefaction and Solid Fuels Contractors Review Conference***, Pittsburgh, Pennsylvania, September 3-4, 1997 (by M. C. Jha, F. J. Smit, G. L. Shields and N. Moro).

P-3. “Installation of 4-Meter Diameter Microcel™ Flotation Columns at the Lady Dunn Preparation Plant,” ***Coal Prep 97, Proceedings, 14th International Coal Preparation Exhibition and Conference,*** Lexington, Kentucky, April 29-May 1, 1997, pp 115-123 (by D. I. Phillips, R.-H. Yoon, G. H. Luttrell, L. R. Fish and T. A. Toney).

P-4. “Properties of Premium Coal Water Fuel Prepared by Advanced Physical Fine Coal Cleaning,” ***Proceedings of the 22nd International Technical Conference on Coal Utilization & Fuel Systems,*** Clearwater, Florida, March 16-19, 1997, pp 853-864 (by M. C. Jha, F. J. Smit, N. Moro and T. J. Feeley, III).

P-5. “Reduction of Toxic Trace Elements in Coal by Advanced Cleaning,” ***Proceedings, Thirteenth Annual International Pittsburgh Coal Conference***, Vol 2, Pittsburgh, Pennsylvania, September 3-7, 1996, pp 879-884 (by F. J. Smit, N. Moro, G. L. Shields, M. C. Jha and T. J. Feeley, III).

P-6. "Engineering Development of Advanced Physical Fine Coal Cleaning for Premium Fuel Applications," ***Presentation, First Joint Power and Fuel Systems Contractors Conference***, Pittsburgh, Pennsylvania, July 9-11, 1996 (by M. C. Jha, F. J. Smit, G. L. Shields and N. Moro).

P-7. “Lady Dunn Evaluates Column Flotation,” ***Coal,*** July, 1996, pp 64-67 (by M. C. Jha, F. J. Smit, L. R. Fish, T. A. Toney, D. I. Phillips and T. J. Feeley, III).

P-8. “Evaluation of Column Flotation for Fine Coal Cleaning at the Lady Dunn Preparation Plant,” ***Coal Prep 96, Proceedings, 13th International Coal Preparation Exhibition and Conference,*** Lexington, Kentucky, April 30-May 2, 1996 (by M. C. Jha, F. J. Smit, L. R. Fish, T. A. Toney, D. I. Phillips and T. J. Feeley, III).

P-9. "Engineering Development of Advanced Physical Fine Coal Cleaning for Premium Fuel Applications," ***Proceedings, Eleventh Annual Coal Preparation, Utilization, and Environmental Control Contractors Conference***, Vol 1, Pittsburgh, Pennsylvania, July 12-14, 1995, pp 17-24 (by M. C. Jha, F. J. Smit, G. L. Shields and N. Moro).

P-10. “Selection of Feed Coals For Production of Premium Fuel Using Column Flotation and Selective Agglomeration Processes,” ***High Efficiency Coal Preparation: An International Symposium***, (S. K. Kawatra, Editor), Littleton, Colorado: SME, 1995, pp 391-400 (by M. C. Jha and F. J. Smit).

P-11. "Engineering Development of Advanced Physical Fine Coal Cleaning for Premium Fuel Applications," ***Proceedings, Tenth Annual Coal Preparation, Utilization, and Environmental Control Contractors Conference****,* Vol 1, Pittsburgh, Pennsylvania, July 18-21, 1994, pp 59-66 (by M. C. Jha and F. J. Smit).

P-12. “Engineering Development of Two Advanced Physical Coal Cleaning Processes for Premium Fuel Applications,” ***Proceedings of the 19th International Technical Conference on Coal Utilization & Fuel Systems****,* Clearwater, Florida, March 21-24, 1994 (by M. C. Jha and T. J. Feeley, III).

P-13. “Column Flotation to Produce Ultra-Clean Coals,” ***Presentation, AIME/SME Annual Meeting***, Albuquerque, New Mexico, February 15-18, 1994 (by B. K. Parekh, J. G. Groppo, M. C. Jha, F. J. Smit and T. J. Feeley, III).

P-14. "Engineering Development of Advanced Physical Fine Coal Cleaning for Premium Fuel Applications," ***Proceedings, Ninth Annual Coal Preparation, Utilization, and Environmental Control Contractors Conference***, Pittsburgh, Pennsylvania, July 19-22, 1993, pp 104-111 (by M. C. Jha and F. J. Smit).

APPENDIX

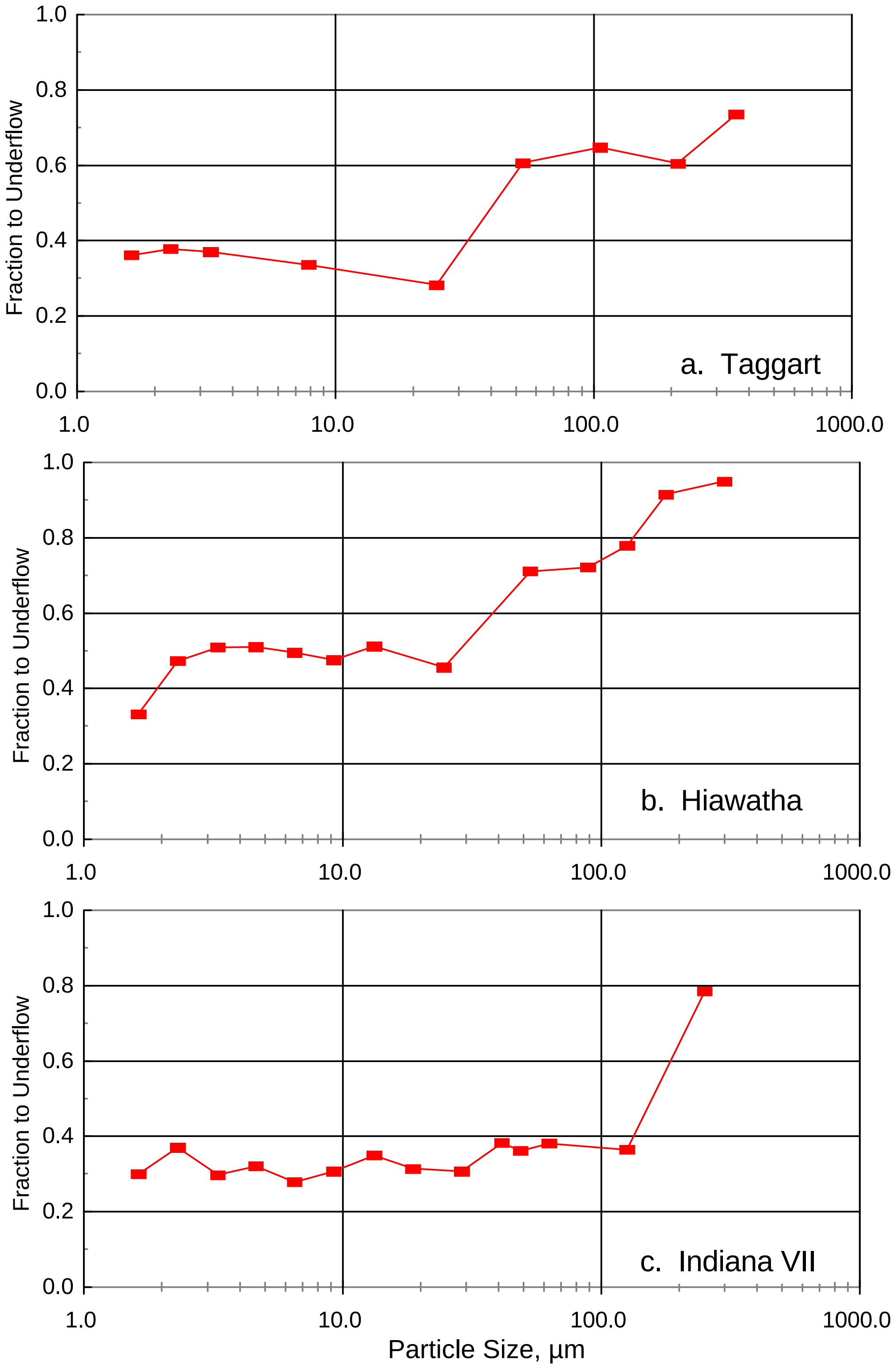


Figure A-1. Cyclone Partition Curves

Table A-1. Properties of Phase I Test Coals

	Taggart Wentz, VA		Sunnyside Sunnyside, UT		Elkhorn No. 3 Chapperal, KY		Indiana VII Minnehaha, IN		Winifrede Sandlick, WV		Dietz Spring Creek, MT	
	As-Received	Bone Dry	As-Received	Bone Dry	As-Received	Bone Dry	As-Received	Bone Dry	As-Received	Bone Dry	As-Received	Bone Dry
Proximate, %:												
Ash	2.01	2.07	4.78	5.11	5.62	6.04	7.50	9.25	7.89	8.42	3.91	4.98
Volatile Matter	35.35	36.46	34.85	37.29	33.45	35.98	28.14	34.70	31.79	33.95	33.08	42.15
Fixed Carbon	59.60	61.47	53.84	57.60	53.90	57.98	45.45	56.05	53.97	57.63	41.49	52.87
Moisture	3.05		6.53		7.03		18.91		6.35		21.52	
Sulfur, %:												
Total	0.60	0.62	0.59	0.63	0.80	0.86	0.40	0.49	0.88	0.94	0.26	0.33
Pyrite	0.05	0.05	0.07	0.07	0.16	0.17	0.12	0.15	0.14	0.15	0.043	0.055
Sulfate	0.001	0.001	0.002	0.002	0.007	0.007	0.002	0.002	0.001	0.001	0.002	0.003
HHV, Btu/lb	14,829	15,296	13,378	14,313	13,138	14,059	10,924	13,472	12,957	13,836	9678	12,332
HHV, GJ/t	34.48	35.56	31.10	33.28	30.55	32.69	25.40	31.32	30.13	32.17	22.51	28.68
Air-Dried Moisture, %	1.27		2.03		2.42		10.29		1.73			
Equilibrium Moisture, %	1.01		2.52		3.04		9.30		2.93		22.64	
Hardgrove Grindability Index	52		54		46		55		47		41	
Density, Dry Coal, kg/m^3		1,267		1,303		1,330		1,387		1,362		1,386
Sulfur, lb/MBtu	0.41		0.44		0.61		0.36		0.68		0.27	
Sulfur, g/GJ	174		189		263		156		292		116	

Table A-2. Washing Plant Recovery and Properties of ROM Phase I Test Coals

	Taggart		Sunnyside		Elkhorn No. 3		Indiana VII		Winifrede		Dietz*	
	Wentz, VA		Sunnyside, UT		Chapperal, KY		Minnehaha, IN		Sandlick, WV		Spring Creek, MT	
	As-Received	Bone Dry	As-Received	Bone Dry	As-Received	Bone Dry	As-Received	Bone Dry	As-Received	Bone Dry	As-Received	Bone Dry
						ROM Coal Properties						
Proximate, %:												
Ash	28.72	31.73	14.07	15.19	42.91	45.34	34.02	38.10	25.91	27.67	3.91	4.98
Volatile Matter	22.70	25.08	30.87	33.33	21.81	23.05	23.01	25.77	25.93	27.69	33.08	42.15
Fixed Carbon	3.10	43.19	47.68	51.48	29.92	31.61	32.26	36.13	41.80	44.64	41.49	52.87
Moisture	9.48		7.38		5.36		10.72		6.37		21.52	
Sulfur, %:												
Total	0.38	0.42	0.53	0.57	0.59	0.62	0.71	0.80	0.79	0.84	0.26	0.33
Pyrite	0.12	0.13	0.21	0.23	0.24	0.25	0.47	0.53	0.25	0.26	0.04	0.055
Sulfate	0.003	0.003	0.002	0.002	0.003	0.003	0.018	0.020	0.001	0.001	0.002	0.003
HHV, Btu/lb	9,402	10,387	11,650	12,578	7,277	7,689	7,483	8,382	10,061	10,746	9,678	12,332
Sulfur, lb/MBtu	0.40		0.45		0.81		0.95		0.78		0.27	
Air-Dried Moisture, %	1.06		5.28		3.21		8.42		4.17			
Density,												
Dry Coal, kg/m^3		1,560		1,370		1,731		1,696		1,567		1,386
						Washing Plant Performance						
Clean Coal Yield, wt %	59.8	64.0	78.5	79.2	52.5	51.6	62.0	56.3	69.5	69.5	100.0	100.0
HHV Recovery, %	94.3	94.3	90.1	90.1	94.6	94.6	90.5	90.5	89.5	89.5	100.0	100.0

* The mine does not wash the Dietz coal before sale.

Table A-3. Additional Properties of PDU Coals

	Taggart		Indiana VII		Hiawatha	
	As-Received	Bone Dry	As-Received	Bone Dry	As-Received	Bone Dry
Proximate, %:						
Ash	3.30	3.50	7.94	9.55	7.75	8.20
Volatile Matter	32.13	34.12	27.36	32.92	40.02	42.35
Fixed Carbon	58.73	62.38	47.81	57.53	46.72	49.45
Moisture	5.84		16.89		5.51	
Sulfur, %:						
Total	0.61	0.65	0.42	0.51	0.49	0.79
Pyrite	0.05	0.05	0.12	0.15	0.07	0.12
Sulfate	< 0.01	< 0.01	0.01	0.01	< 0.01	< 0.01
Organic	0.56	0.60	0.29	0.35	0.42	0.67
Ultimate Analysis, %:						
Moisture	5.84		16.89		5.51	
Carbon	80.30	85.28	62.40	75.39	72.93	77.18
Hydrogen	4.66	4.95	3.94	4.74	4.92	5.21
Nitrogen	1.38	1.47	1.40	1.68	1.37	1.45
Sulfur	0.61	0.65	0.42	0.51	0.49	0.52
Ash	3.30	3.50	7.94	9.55	7.75	8.20
Oxygen	3.91	4.15	6.75	8.13	7.03	7.44
Heating Value, Btu/lb	13,874	14,735	10,828	13,028	12,725	13,647
Equilibrium Moisture, %	2.6		14.5		4.3	
Density, kg/m^3		1,260		1,360		1,275
Hardgrove Grindability Index	49		54		44	
Coal Rank	hvA		hvC		hvA	
Preparation Plant Weight Recovery, %		57.2		61.9		100.0
Preparation Plant Btu Recovery, %		84.9		90.5		100.0

Table A-4. Trace Elements in Bench-Scale Coals and Products (dry basis)

			Clean Coal		Fine Refuse	
	ROM Coal	Test Coal	Flotation	Agglomeration	Flotation	Agglomeration
Taggart:						
Ash, %	31.73	2.14	1.52	1.58	54.16	72.40
S(tot), %	0.53	0.64	0.63	0.65	1.21	0.94
S(py), %	0.07	0.11	0.12	0.14	0.31	0.74
Sb, ppm	0.20	0.38	0.42	0.3	0.59	0.3
As, ppm	5.5	1.9	1.7	1.7	23	42
Be, ppm	2.1	1.7	1.8	1.3	2.8	2.4
Cd, ppm	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Cr, ppm	22	6.4	4.2	6	130	930
Co, ppm	8.6	7.9	8.0	6.8	26	31
Pb, ppm	3	2	2	2	< 20	40
Mn, ppm	120	7	4	4.0	280	470
Hg, ppm	0.02	0.01	0.01	< 0.01	0.06	0.09
Ni, ppm	6.6	13	10	16	24	930
Se, ppm	4.4	1.9	3.1	1.7	9.4	3.7
Cl, ppm	44	63	68	58	46	31
Winifrede:						
Ash, %	27.67	8.64	2.96	2.76	9.27	82.20
S(tot), %	0.92	0.97	0.89	0.94	0.96	1.19
S(py), %	0.33	0.21	0.19	0.13	0.28	0.97
Sb, ppm	0.26	0.34	0.55	0.5	0.34	0.2
As, ppm	5.0	2.6	1.5	1.7	2.8	18
Be, ppm	3.2	3.3	2.9	2.5	3.2	5.3
Cd, ppm	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Cr, ppm	26	56	28	56	56	680
Co, ppm	8.9	9.1	7.6	7.9	9.0	15.0
Pb, ppm	9	5	5	4	5	31
Mn, ppm	27	16	4	5.3	14	81
Hg, ppm	0.04	0.03	0.03	0.02	0.03	0.12
Ni, ppm	11	25	15	19	23	150
Se, ppm	5.9	5.9	5.2	2.3	5.3	5.4
Cl, ppm	1050	880	940	651	880	112
Elkhorn No. 3:						
Ash, %	42.91	5.74	2.63	2.57	30.82	60.00
S(tot), %	0.84	0.96	0.92	0.93	1.15	1.16
S(py), %	0.40	0.26	0.24	0.12	0.67	0.67
Sb, ppm	0.19	0.33	0.48	0.3	0.22	0.2
As, ppm	7.3	4.6	2.5	2.4	12	39
Be, ppm	2.3	1.9	1.9	1.6	2.4	2.1
Cd, ppm	0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Cr, ppm	29	16	9.6	12	60	220
Co, ppm	11	6.3	5.6	4.9	13	18
Pb, ppm	4	3	2	7	6	42
Mn, ppm	200	25	10	6.8	150	190
Hg, ppm	0.03	0.03	0.02	0.01	0.08	0.11
Ni, ppm	11	18	14	15	65	140
Se, ppm	4.7	4.4	2.1	2.1	7.1	6.9
Cl, ppm	860	1170	1240	1080	780	214

continued . . .

Table A-4 Continued. Trace Elements in Bench-Scale Coals and Products (dry basis)

			Clean Coal		Fine Refuse	
	ROM Coal	Test Coal	Flotation	Agglomeration	Flotation	Agglomeration
Indiana VII:						
Ash, %	38.10	9.64	2.86	2.74	29.15	81.60
S(tot), %	0.77	0.67	0.57	0.63	1.06	1.83
S(py), %	0.51	0.31	0.19	0.14	0.76	1.50
Sb, ppm	1.2	1.8	2.3	1.9	0.9	0.2
As, ppm	4.1	6.5	1.7	1.7	6.5	14
Be, ppm	2.3	2.6	2.7	2.2	2.7	1.9
Cd, ppm	0.1	< 0.1	< 0.1	<0.1	0.3	< 0.1
Cr, ppm	22	26	14	15	65	190
Co, ppm	11	9.2	9.2	9.0	11	9.0
Pb, ppm	14	7	5	7	12	24
Mn, ppm	150	33	14	17	95	200
Hg, ppm	0.02	0.03	0.02	< 0.01	0.04	0.07
Ni, ppm	30	38	36	33	48	90
Se, ppm	0.78	0.90	0.78	0.5	0.76	1.3
Cl, ppm	38	65	76	101	54	35
Sunnyside:						
Ash, %	14.07	5.14	2.69	2.54	55.60	71.20
S(tot), %	0.59	0.66	0.65	0.65	0.59	0.73
S(py), %	0.12	0.10	0.13	0.14	0.33	0.53
Sb, ppm	0.06	0.09	0.10	0.1	0.24	0.3
As, ppm	0.83	0.32	0.15	0.6	3.7	7.6
Be, ppm	0.8	0.8	0.8	0.5	0.9	0.6
Cd, ppm	< 0.1	< 0.1	< 0.1	< 0.1	0.3	< 0.1
Cr, ppm	9.2	9.7	5.9	10	79	480
Co, ppm	2.2	1.4	1.3	0.6	4.8	4.6
Pb, ppm	4	2	2	3	< 10	13
Mn, ppm	42	31	12	15	360	700
Hg, ppm	0.02	0.02	< 0.01	< 0.01	0.01	0.07
Ni, ppm	1.7	7.7	3.3	13	84	590
Se, ppm	0.90	0.67	0.71	0.7	2.9	1.4
Cl, ppm	1180	1100	1120	982	250	85

Table A-5. Trace Elements in PDU Coals and Products (dry basis)

	ROM Coal	Crushed Test Coal	Clean Coal		Fine Refuse	
			Flotation	Agglomeration	Flotation	Agglomeration
Taggart:						
Ash, %	34.7	3.48	1.83	1.59	39.65	62.47
S(tot), %	0.46	0.70	0.72	0.67	0.68	0.88
S(py), %	0.02	0.07	0.08	0.06	0.09	0.47
Sb, ppm	0.17	0.8	0.6	0.8	0.8	0.7
As, ppm	2.47	3.69	2.14	2.26	18.6	30
Be, ppm	2.0	2.0	2.2	2.0	1.6	2.5
Cd, ppm	0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Cr, ppm	30	6	9	7	288	174
Co, ppm	12	8.7	9.0	8.8	23	22
Pb, ppm	8	4	3.5	3	64	34
Mn, ppm	110	7	9	4.0	370	229
Hg, ppm	0.03	0.02	0.02	0.01	0.12	0.08
Ni, ppm	11	11	11	11	33	50
Se, ppm	1.39	1.52	1.02	1.38	1.88	4.15
Cl, ppm	177	192	75	140	154	115
Indiana VII:						
Ash, %	38.10	9.45	3.23	2.76	34.08	90.00
S(tot), %	0.77	0.54	0.59	0.40	1.33	0.76
S(py), %	0.51	0.10	0.13	0.04	1.11	0.63
Sb, ppm	1.2	1.08	2.8	2.4	0.9	0.22
As, ppm	4.1	1.27	1.9	0.6	5.8	5.40
Be, ppm	2.3	2.3	2.8	2.3	2.2	1.8
Cd, ppm	0.1	< 0.1	< 0.1	< 0.1	< 0.1	1.6
Cr, ppm	22	14	13	13	66	83
Co, ppm	11	9.4	8.1	9.5	9	12
Pb, ppm	14	6	9	7	53	48
Mn, ppm	150	28	17	8	123	392
Hg, ppm	0.02	< 0.01	0.02	< 0.01	0.04	0.05
Ni, ppm	30	50	38	53	27	37
Se, ppm	0.78	0.45	0.41	0.51	0.66	0.30
Cl, ppm	38	41	138	42	123	23
Hiawatha:						
Ash, %	7.52	7.52	2.70	2.81	35.63	78.62
S(tot), %	0.56	0.56	0.63	0.52	0.80	0.83
S(py), %	0.11	0.11	0.11	0.07	0.39	0.71
Sb, ppm	0.09	0.09	0.09	0.10	0.07	0.10
As, ppm	0.71	0.71	0.39	0.50	4.14	4.59
Be, ppm	0.3	0.3	0.3	0.3	0.4	0.4
Cd, ppm	< 0.1	< 0.1	0.1	< 0.1	< 0.1	< 0.1
Cr, ppm	4.8	4.8	4.4	9.0	18	158
Co, ppm	0.8	0.8	0.6	0.9	2.7	4
Pb, ppm	< 2	< 2	< 2	< 2	< 20	< 20
Mn, ppm	8	8	4	3	34	144
Hg, ppm	0.01	0.01	0.02	0.01	0.03	0.04
Ni, ppm	1	1	1	3	< 1	43
Se, ppm	1.12	1.12	0.81	1.07	1.54	1.54
Cl, ppm	266	266	234	216	126	19

ACRONYMS AND ABBREVIATIONS

$	U.S. dollar
Btu	British thermal unit
CAAA	1990 Clean Air Act Amendments
cfm	cubic feet per minute
CWF	coal-water (slurry) fuel
cP	centipoise
D80	80 percent passing size
ft	foot or feet
GJ	gigajoule (10^9 Joule)
g, gm	gram
gal	gallon or gallons
gpm	gallons per minute
h, hr	hour
HHV	higher heating value (water condensed to liquid)
hp	horsepower
k	kilo (10^3)
kg	kilogram
kJ	kilojoule
kW	kilowatt electricity
lb, lbs	pound, pounds
m	meter
M	million, mega (10^6)
MAF	moisture- and ash-free
MMD	mass mean diameter
min	minute
mm	millimeter
MBtu	million Btu
O&M	operating and maintenance
PDU	process development unit
ppm	parts per million
PSD	particle size distribution
psi, psia, psig	pounds per square inch, pounds per square inch absolute, pounds per square inch gauge
ROM	run-of-mine
s, sec	second
st, t	short ton
stph, tph	short tons per hour
stpy, tpy, Mtpy	short tons per year, million short tons per year
T	metric ton (1000 kg)
wt	weight
y, yr	year
µm	micrometer, micron, 10^{-6} meter
µg	microgram, 10^{-6} gram

CONVERSION FACTORS TO SI UNITS

1 Btu = 1055 Joules
1 cfm = 0.028 cubic meter per minute
1 cP = 1 millipascal · second
1 foot = 0.305 meter
1 gallon = 3.785 liters, 0.003785 cubic meter
1 gpm = 0.003785 cubic meter per minute
1 hp = 0.746 kilowatt
1 pound (lb) = 0.454 kilogram
1 MBtu = 1.055 gigajoules
1 ppm = 1 microgram per gram (µg/g)
1 psi = 6,898 Pascal
1 short ton = 908 kg, 0.908 metric tons
1 tph = 0.908 metric tons per hour
1 tpy = 0.908 metric tons per year

48 mesh = 300 µm = 0.300 mm
60/62 mesh = 250 µm = 0.250 mm
70 mesh = 212 µm = 0.212 mm
100 mesh = 150 µm = 0.150 mm
140/150 mesh = 106 µm = 0.106 mm
200 mesh = 74 µm = 0.074 mm
270 mesh = 53 µm = 0.053 mm
325 mesh = 45 µm = 0.045 mm