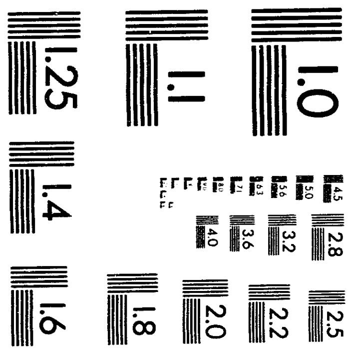
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QUARTERLY TECHNICAL PROGRESS REPORT 5
OCTOBER - DECEMBER, 1993

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

Prepared for
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# EXECUTIVE SUMMARY

This project is a major step in the Department of Energy's program to show that ultra-clean coal-water slurry fuel (CWF) can be produced from selected coals and that this premium fuel will be a cost-effective replacement for oil and natural gas now fueling some of the industrial and utility boilers in the United States.

The replacement of oil and gas with CWF can only be realized if retrofit costs are kept to a minimum and retrofit boiler emissions meet national goals for clean air. These concerns establish the specifications for maximum ash and sulfur levels and combustion properties of the CWF.

This cost-share contract is a 51-month program which started on September 30, 1992. This report discusses the technical progress made during the 5th quarter of the project from October 1 to December 31, 1993.

## SPECIFIC OBJECTIVES OF PROJECT

The project has three major objectives:

- The primary objective is 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 are advanced column flotation and selective agglomeration.

- A secondary objective is to develop the design base for near-term application of these advanced fine coal cleaning technologies in new or existing coal preparation plants for efficiently processing minus 28-mesh coal fines and converting this to marketable products in current market economics.

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

## APPROACH

The project team consists of Cyprus Amax Minerals Company through its subsidiaries Amax Research & Development Center (Amax R&D) and Amax Coal Company (Midwest and Cannelton Divisions), Bechtel Corporation, Center for Applied Energy Research (CAER) of the University of Kentucky, Arcanum Corporation, and Virginia Polytechnic Institute (VPI). Dr. Douglas Keller of Syracuse University is a consultant to the project.

The project effort has been divided into four phases which are further divided into eleven tasks, including coal selection, laboratory and bench-scale process optimization, design, construction, and operation of a 1.8 tonne/hour process development unit (PDU). Tonnage quantities of the ultra-clean coals will be produced in the PDU for combustion testing by the DOE. Near-term applications of advanced cleaning technologies to existing coal preparation plants will also be studied.

## ACCOMPLISHMENTS DURING QUARTER

The annual Task 1 review and updating of the Project Management Plan was accomplished early during the quarter and activities continued on Phase I Tasks 2 through 6 as described below.

### Task 1. Project Planning

During the quarter, the Project Management Plan was revised and submitted to DOE. The following forms showing the detailed plans for FY 1994 were also submitted separately:

| | |
|---|---|
| Form F 1332.3 | Milestone Schedule Plan |
| Form F 1332.4 | Labor Plan |
| Form F 1332.7 | Cost Plan |

The labor plan was revised and Amax R&D's labor hours were reduced for two main reasons. First, Virginia Tech was added to the project team as a subcontractor. Second, some of the labor hours originally planned for analytical and shop activities were reallocated to the "Other Direct Cost" category.

Based on the experience of the first year of the project, some subtask budgets were revised. The overall budget for the project, however, remained unchanged. The project has fallen behind schedule by about three months and is now scheduled for completion by the end of calendar year 1996.

### Task 2. Coal Selection and Procurement

Six coals (namely Taggart, Indiana VII, Sunnyside, Winifrede, Elkhorn No. 3, and Dietz) were selected for testing during Phase I. The first five are washed bituminous coals and the last is a subbituminous coal. Twenty tonne lots of the coals were purchased for use during the project. Each lot has been crushed, sampled, and stored for use as needed during the project.

All of the above test coals were relatively low in pyrite sulfur as well as being low in organic sulfur. Processing these coals may not challenge the pyrite removal abilities of the advanced cleaning technologies, so the DOE requested Amax R&D

to consider alternate test coals that contained more pyrite. The choices of coal would still be restricted to those containing less than 258 g/GJ (0.6 lb/mmBtu) organic sulfur since the latter form of sulfur would not be removed by any physical cleaning process.

Using data provided by the DOE from their database of channel sample analyses, Amax R&D conducted a telephone survey to locate sources of high pyrite sulfur/low organic sulfur ratio coals. Three potential sources were identified and samples were obtained from two of the mines for examination at Amax R&D. The two coals were as follows:

- Ohio No. 7 coal from the Buckeye Industrial Mine.
- Sewickley coal from the Warwick Mine in Pennsylvania.

The Ohio No. 7 coal was indeed found to be a high pyrite coal containing less than 258 g/GJ organic sulfur. It was also found that much of the pyrite would be rejected in a conventional washing plant and that very fine grinding would be required to liberate the ash minerals ahead of advanced flotation or agglomeration. In this respect, the Ohio No. 7 resembles the Winifrede coal previously selected for testing. The Sewickley coal was found to contain more organic sulfur than the channel samples in the DOE database and would not meet the <258 g/GJ specification. It was learned that the third coal (from the Matiki Mine in Maryland) is a medium volatile coal which probably would not respond well to the advanced cleaning technologies.

It was decided that there would be no advantage in substituting one of these three coals for one of the six test coals already being tested during Phase I.

## Task 3. Development of Near-Term Applications

The topical report for Subtask 3.1, Engineering Analysis, was finalized by Bechtel and distributed to DOE and project team members on November 15, 1993. The report presents conceptual designs and cost estimates for the addition of advanced coal cleaning circuits (column flotation or selective agglomeration) to process minus 100-mesh fines into marketable products (filter cake, dry powder, or briquettes). Three Amax coal preparation plants (Ayrshire, Lady Dunn, and Wabash) were evaluated as potential sites. The general conclusion was that column flotation would be less expensive than selective agglomeration. Drying and briquetting add significantly to the processing costs, but their inclusion may be justified based on the specific niche markets.

Work will now move towards actual testing of advanced column flotation technology at bench-scale under Subtask 3.2. The possibility of field testing at a larger scale will also be evaluated.

## Task 4. Engineering Development of Advanced Froth Flotation for Premium Fuels

Task 4 is divided into five subtasks. There was activity on each of the subtasks during the quarter as described below.

### Subtask 4.1. Grinding

A topical report describing the grinding test work was drafted during the quarter for submission to the DOE during January 1994 for comment and approval. The report contains the grinding parameters to be used for each of the five bituminous coals when designing the PDU. Winifrede coal, in particular, will require ultra-fine grinding (to D80 <11 $\mu$m) and installation of the largest grinding capacity (341 kW/tph).

### Subtask 4.2. Process Optimization Research

The laboratory-scale flotation research has been divided among members of the team. CAER is evaluating equipment design variables and the flotation response of Elkhorn No. 3, Sunnyside, and Winifrede coals. VPI is evaluating the performance of the Microcel aeration system and the flotation response of Indiana VII and Winifrede coals. Amax R&D is evaluating Taggart and Dietz coals. Amax R&D is also obtaining comparison data for Winifrede coal and supplying ground coal slurries to the other members of the team.

For the most part, the laboratory flotation response testing of the five bituminous coals was completed at each location and the results were being evaluated at the end of the quarter. The initial observations were that the target ash and HHV recovery specifications can be met for Taggart, Elkhorn No. 3, and Sunnyside coals, but the specifications will be more difficult to meet with Winifrede coal. These observations were consistent with results of earlier laboratory batch flotation testing at Amax R&D to quantify the liberation characteristics of the coals. The column flotation results were not available for the Indiana VII coal, and work on the Dietz coal was deferred until a viable flotation scheme is developed for low-rank coal.

CAER conducted tests on Winifrede and Sunnyside slurries in a 100-mm (4-inch) packed column for comparison with results of tests in conventional columns equipped with internal and external air spargers. Further optimization of the packed column operation is planned, but so far the comparisons have favored the conventional configurations with respect to ash reduction:

| | Conventional | | Packed | |
|---|---|---|---|---|
| | Ash, % | HHV Recovery, % | Ash, % | HHV Recovery, % |
| Winifrede | 3.5 | 85 | 4.25 | >90 |
| Sunnyside | 2.1 | 90 | 3.75 | 99 |

**Subtask 4.3. CWF Formulation Studies**

The objective of Subtask 4.3 is to define the process steps necessary to prepare a premium grade coal-water slurry fuel (CWF) from column flotation product. These process steps include the following:

- Grinding to achieve an optimum particle size distribution.
- Reagent additions to achieve the desired slurry rheology.
- Mixing to reslurry the dewatered froth.

It is expected that the initial test work will be on clean coal produced during Subtask 4.2 (laboratory scale column flotation) and Subtask 4.4 (bench-scale column flotation). The subtask test plan was approved by DOE/PETC project management and issued on October 21, 1993.

Testing began during the quarter on Taggart seam clean coal produced from the Subtask 4.2 100-mm column flotation of minus 62-mesh coal. The initial tests indicated that the flotation product lacked sufficient ultra-fine particles to provide stable slurry with good rheology. It appears that additional grinding will be required after flotation to optimize the particle size distribution.

**Subtask 4.4. Bench-Scale Testing and Process Scale-Up**

The Ken-Flote 0.3-m diameter by 4.6-m (1- by 15-foot) bench-scale column flotation system that had previously been used for the process control emerging technology project at PETC was installed in the Amax R&D pilot plant area for the bench-scale testing. The unit will have capacity in the 50 to 100 kg/hr range (dry coal, equal to 100 to 200 lb/hr). The system was started up successfully with Elkhorn No. 3 slurry, and the fuzzy logic level and flow control systems performed very well. Parametric testing of Elkhorn No. 3 coal will begin in January.

**Subtask 4.5. Conceptual Design of the PDU and Advanced Froth Flotation Module**

The topical report for Subtask 4.5, subtitled "Conceptual Engineering Package", was issued on December 10, 1993. The concept is for the production of 1.8 tph (2.0 stph on a dry basis) of clean coal by column flotation in a PDU to be located at Amax R&D in Golden, Colorado. The unit will be designed for continuous production of ultra-clean coal meeting project specifications and to demonstrate process scale-up when cleaning three of the test coals. The PDU will also be capable of demonstrating near-term applications, including drying and briquetting of fine coal into marketable forms, and will be housed in existing pilot plant buildings at Amax R&D.

## Task 5. Detailed Engineering Design of the PDU and Advanced Flotation Module

Bechtel has started planning activities for this task. Plant 400, filtering and dewatering, will be designed first. The plant will utilize filters already in place at Amax R&D Center as well as filters transferred from the Wilsonville selective agglomeration POC plant.

## Task 6. Selective Agglomeration Laboratory Research and Engineering Development for Premium Fuels

Task 6 is divided into six subtasks. There was activity in three of the subtasks during the quarter, namely Subtask 6.2 (Grinding), Subtask 6.4 (Process Optimization Research), and Subtask 6.6 (Conceptual Design of the Selective Agglomeration Module).

### Subtask 6.2. Grinding

The continuous grinding tests for preparation of feed for selective agglomeration were finished during the quarter with completion of parametric 1.2-m ball mill and 40-liter stirred ball mill tests on Dietz coal. Dietz is a subbituminous coal with a Hardgrove grindability of 41. As a result of the lower grindability, grinding capacities of the two mills were reduced by 40 to 50 percent from the capacities noted for the bituminous coals. Liberation tests indicated that the coal needs to be ground to an 80 percent passing size of 20 μm in order to meet the product ash specification.

### Subtask 6.3. Process Optimization Research

The effects of pH and chelating agent addition on the selective agglomeration of Dietz coal were investigated with heptane bridging liquid. Reduction of the pH was found to be a requirement for efficient agglomeration. EDTA (ethylene diamine tetraacetic acid) chelating was found to be helpful, but acetic acid was found be a more effective agent for pH reduction on a mass basis.

### Subtask 6.6. Conceptual Design of the Selective Agglomeration Module

During the quarter, a preliminary planning meeting was held at Bechtel. Focus of initial activities was on evaluation of relative costs and merits of two potential designs. The first will be similar to the design developed and tested by Arcanum and Bechtel at Homer City using heptane as the agglomerating agent in a series of high and low shear stirred tank reactors. The second approach will use a single-stage novel design reactor proposed by Dr. Keller based on his past experience with the Otisca agglomeration system which used pentane as the agglomerating agent. The second system offers potential for cost savings but may need more development effort.

# INTRODUCTION/BACKGROUND

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

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

The cost-shared contract effort is for 51 months beginning September 30, 1992, and ending December 31, 1996. This report discusses the technical progress made during the fourth quarter of the project, October 1 to December 31, 1993. Four quarterly reports have been issued previously.[1,2,3,4]

## SPECIFIC OBJECTIVES OF PROJECT

The three major objectives of this project are discussed below.

The primary objective is to develop, the design base for prototype commercial advanced fine coal cleaning facilities capable of producing ultra-clean coals suitable for conversion to stable, highly loaded coal-water slurry fuels which contain less than 860 grams ash per gigajoule HHV and preferably less than 430 grams ash per gigajoule HHV and less than 258 grams of sulfur per gigajoule HHV. These amounts are equivalent to the 2 pounds of ash and preferably less than 1 pound of ash per million Btu HHV and less than 0.6 pound of sulfur per million Btu HHV stated in the solicitation. The advanced fine coal cleaning technologies to be employed are advanced column froth flotation and selective agglomeration. Operating conditions during the advanced cleaning processes will allow recovery of at least 80 percent of the Btus in run-of-mine source coals at an annualized cost of less than $2.37 per gigajoule ($2.50 per million Btu), including the mine mouth cost of the raw coal.

A secondary objective of the work is to develop the design base for near-term commercial applications of advanced fine coal cleaning technologies suitable for integration into new or existing coal preparation plants for the purpose of economically and efficiently processing minus 28-mesh coal fines. The design base will also include the auxiliary systems required to yield a shippable, marketable product such as a dry clean coal product.

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

## APPROACH

A team headed by Amax Research & Development Center (Amax R&D) was formed to accomplish the project objectives. Figure 1 shows the project organization chart. Amax R&D is managing the project and also providing laboratory and pilot plant facilities and expertise in the areas of coal characterization and coal slurry fuel preoaration. Amax Coal Company (now part of Cyprus Amax Coal Company) is providing operating and coal marketing experience and some of the coals to be used during the program. Bechtel Corporation is providing engineering and design capabilities and the operating experience it gained while managing similar proof-of-concept projects for the DOE. The Center for Applied Energy Research (CAER) at the University of Kentucky and Virginia Polytechnic Institute and State University (VPI) are providing research and operating experience in the column flotation area, and Arcanum Corporation is providing similar experience in the selective agglomeration area. Dr. Douglas Keller of Syracuse University is serving as a consultant in the area of coal source selection and selective agglomeration.

The overall engineering development effort has been divided into four phases with specific activities as follows:

Phase I encompasses preparation of a detailed Project Work Plan, selection and acquisition of the test coals, and laboratory and bench-scale testing. The laboratory and bench-scale work will determine the cleaning potential of the selected coals and establish design parameters and operating guidelines for a process development unit (PDU) containing advanced column flotation and selective agglomeration modules. A conceptual engineering design is being prepared for a fully integrated and instrumented 1.8-tonne/hour PDU incorporating the features determined from the laboratory and bench-scale studies. A generic approach is being followed during the laboratory studies for selection of the flotation and agglomeration systems for the PDU which will best meet project objectives.

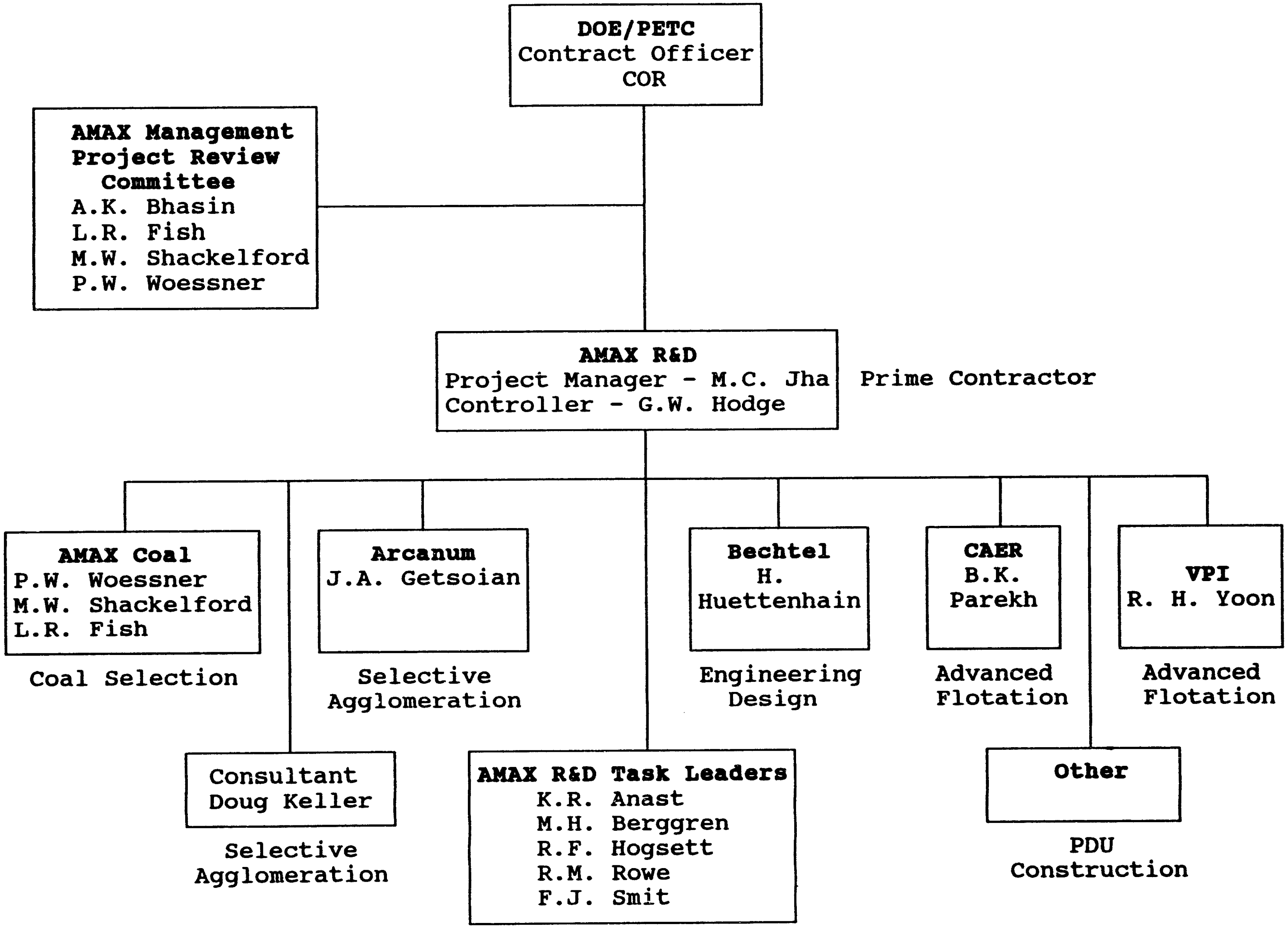



Figure 1. Project management organization chart.

Revised September 29, 1993

The properties of slurry fuels prepared from the ultra-clean coals also will be determined during Phase I, and test lots of ultra-clean coals will be prepared by bench-scale column flotation and bench-scale selective agglomeration for end-use testing by the DOE. The distribution of toxic trace elements will be determined during production of these test lots.

In addition, methods for applying the advanced cleaning technologies in existing coal preparation plants in the near term will be studied during Phase I.

Phases II and III cover the construction and operation of the 1.8-tonne/hour PDU. Phase II will be for advanced column flotation and Phase III will be for selective agglomeration. Process performance will be optimized at the PDU-scale, and 180-tonne lots of ultra-clean coal will be prepared by column flotation and selective agglomeration from each of three test coals. Toxic trace element distributions will also be determined during the production runs. The ultra-clean coals will be delivered to the DOE for end-use testing. In addition and as part of Phases II and III, near-term applications identified in Phase I will be tested in the PDU.

Phase IV covers decommissioning the PDU, restoration of the host site, and preparation of the final project report.

# ACCOMPLISHMENTS DURING QUARTER

As shown in the Work Breakdown Structure (Table 1), the four phases of the project have been further divided into tasks and subtasks. Figure 2 shows the project schedule. Each task and subtask have specific objectives that may be inferred from its title. Work was done on Tasks 1 through 6 of Phase I during the October 1 to December 31, 1993, reporting period.

## TASK 1. PROJECT PLANNING

### Subtask 1.2. Project Plan Revisions

During the quarter, the Project Management Plan was revised and submitted to DOE. Annual revision of the labor and cost plans is required by the contract. The following forms, which were included in the revised Management Plan, were also submitted to DOE separately:

| | |
|---|---|
| Form F 1332.3 | Milestone Schedule Plan |
| Form F 1332.4 | Labor Plan |
| Form F 1332.7 | Cost Plan |

The labor plan was revised and Amax R&D's labor hours were reduced for two main reasons. First, Virginia Tech was added to the project team as a subcontractor, and some of the work to be performed in-house will now be performed by them. They will evaluate the performance of the Microcel column design for production of premium fuels. Secondly, some of the labor hours originally planned for analytical and shop activities have been removed and the equivalent budget has been added to the "Other Direct Cost" category. This was done because of the change in internal policy of Amax R&D.

Based on the experience of the first year of the project, some subtask budgets were revised. The overall budget for the project, however, remains unchanged. The project has fallen behind schedule by about three months and is now scheduled for completion by the end of calendar year 1996.

After the revised Management Plan was submitted to DOE, the merger of AMAX into Cyprus took place and the R&D Center employees received notice of plant closure effective December 29, 1993. However, Cyprus Amax management has planned a transition phase for the project during which key project staff will be retained as contract employees and work will continue while a new project management structure is agreed upon by Cyprus Amax and DOE.

Revised January 12, 1993

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 |
| 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. | SA 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 |

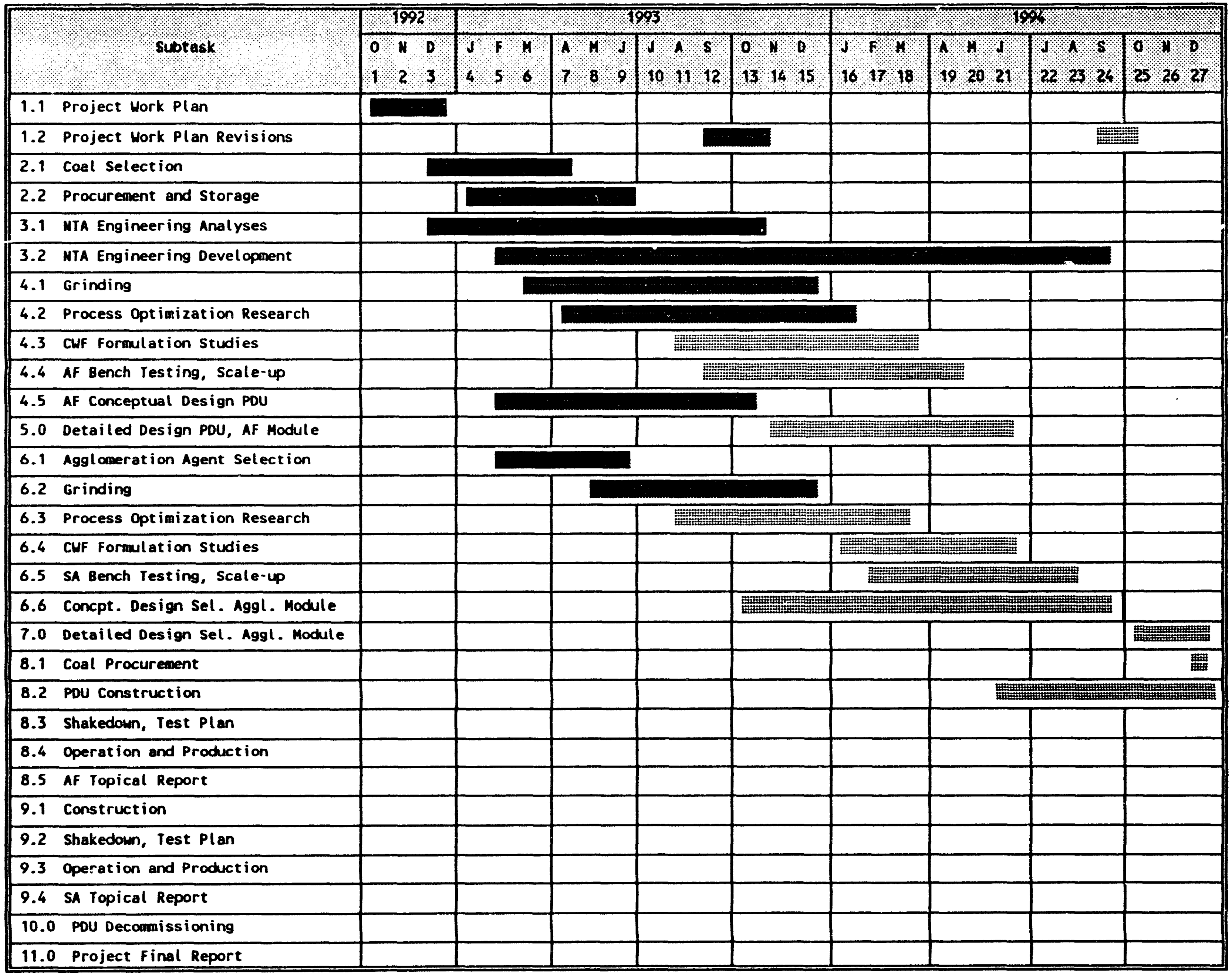



Figure 2. Project schedule.

Revised November 19, 1993

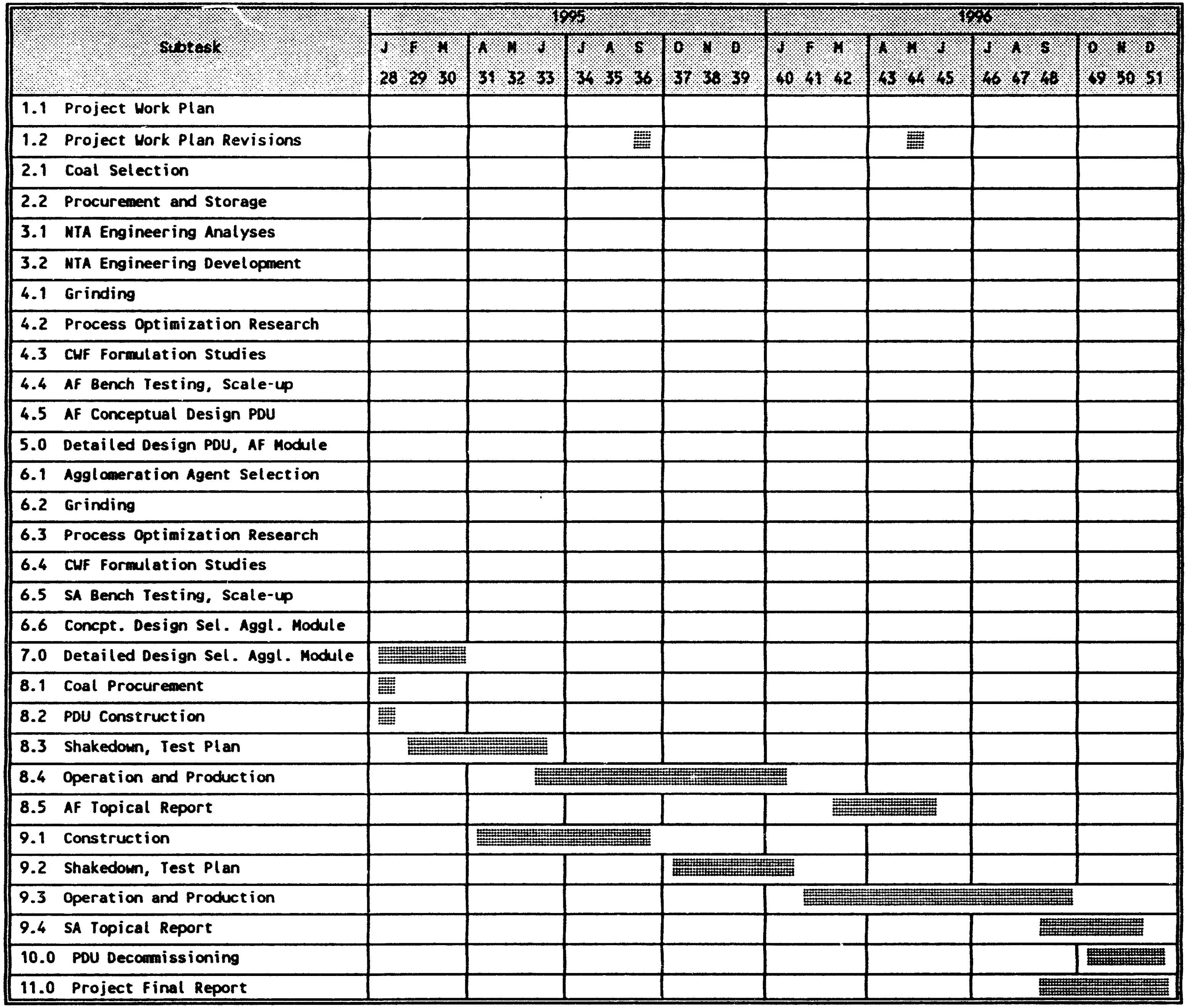



Figure 2. Project schedule (continued). Revised November 19, 1993

## TASK 2. COAL SELECTION AND PROCUREMENT

At the request of the DOE, Amax R&D checked the availability and suitability of alternative coals for testing during the premium fuels project. The DOE specifically asked that coals be considered which contain more pyrite than the six coals selected during Task 2.[5] The DOE also provided a list identifying high pyrite sulfur/low organic sulfur ratio coal seams by county that might fit project requirements. The list was extracted from the DOE database of channel sample analyses.

A telephone survey was made of mines listed in Keystone as operating in the identified counties mining the identified coals. The survey focused on Pennsylvania, Ohio, and Maryland coals. In most cases, the listed mines were found not to be operating in the identified seam or else operating intermittently or at low production rates. In other cases, current production would not meet the target of less than 258 g/GJ (0.6 lb/mmBtu) organic sulfur in the coal.

Two mines were located, though, which had substantial production of high pyrite/low organic sulfur coal and which agreed to furnish Amax R&D with 5-gallon samples of their coals for testing in order to verify sulfur content and cleaning characteristics. The two mine sources were as follows:

1. Ohio No. 7 coal (Upper Freeport), Buckeye Industrial Mine, Columbiana County, Ohio.

2. Sewickley coal, Warwick Mine, Duquesne Light Company, Greene County, Pennsylvania.

The above operators indicated a willingness to supply truck lots of the respective coals in the event their coals were selected for project use. Buckeye mines coal from a number of locations in Columbiana County, and Ohio No. 7 represents a small portion of their production. The rest contains considerably more organic sulfur. All of the Warwick production is from the Sewickley seam. A third potential source (Matiki Mine in Maryland) was also considered, but further checking indicated that Matiki coal is near medium volatile in rank. Higher rank coal is usually very hydrophobic and examples such as Maxwell from Colorado and Upper Freeport from Indiana County, Pennsylvania, have been difficult to work with during flotation and selective agglomeration, so no sample was requested from that mine.

### Sample Testing

The samples were crushed to 6 mesh and splits taken for head analyses and liberation testing. The following analyses were obtained for the two as-received samples:

| | Ohio No. 7 | Sewickley |
|---|---|---|
| Proximate Analysis, % | | |
| Moisture | 2.79 | 4.91 |
| Ash | 7.01 | 8.58 |
| Volatile Matter | 34.58 | 32.18 |
| Fixed Carbon | 55.74 | 54.33 |
| HHV, Btu/lb | 13,606 | 13,169 |
| Sulfur Forms, % | | |
| Total | 2.67 | 1.94 |
| Pyrite | 2.39 | 0.98 |
| Sulfate | 0.002 | 0.002 |
| Organic | 0.28 | 0.96 |
| Organic Sulfur, lb/mmBtu | 0.21 | 0.73 |
| Pyrite Sulfur, lb/mmBtu | 1.76 | 0.74 |

The agglomeration liberation testing was with heptane on coal ground to nominally passing 325 mesh, and the testing produced the following results:

| | Ohio No. 7 | Sewickley |
|---|---|---|
| D80, μm | 18.9 | 18.5 |
| Ash (Dry), % | 3.52 | 5.98 |
| Ash, lb/mmBtu | 2.42 | 4.14 |
| Total Sulfur (Dry), % | 1.73 | |
| Total Sulfur, lb/mmBtu | 1.19 | |
| Pyrite Sulfur (Dry), % | 1.03 | |
| Pyrite Sulfur, lb/mmBtu | 0.71 | |

A sink-float separation was also made on the crushed Ohio No. 7 coal at a specific gravity of 1.6. The bone dry plus 100-mesh float had the following analyses:

| | % | lb/mmBtu |
|---|---|---|
| Ash | 4.00 | 2.74 |
| Total Sulfur | 0.85 | 0.58 |
| Pyrite Sulfur | 0.21 | 0.14 |

Heating value recovery during the sink-float separation was 97.4 percent.

## Discussion

The Sewickley coal did not meet the target organic sulfur specification, so it was not considered further as an alternative test coal.

On the other hand, the organic sulfur content of the Ohio No. 7 coal was acceptable and the pyrite was well liberated by further crushing. In view of the good pyrite liberation during crushing but mediocre pyrite rejection during agglomeration, precleaning would be an obvious step ahead of fine grinding and advanced flotation or selective agglomeration. After precleaning, the Ohio No. 7 coal contained only slightly more pyrite sulfur than the Winifrede or Elkhorn No. 3 coals (0.21 percent versus 0.15 or 0.17 percent). Further testing would be needed to define the actual ash liberation/grinding profile, but the data for minus 325-mesh grinding suggested that the Ohio No. 7 coal resembled Winifrede coal in this regard. That is, very fine and expensive grinding would be necessary if one wished to produce premium fuel meeting a <860 g/GJ ash specification.

At this point, not much would be gained by substituting Ohio No. 7 coal for one of the selected test coals since its processing characteristics are likely to resemble Winifrede coal which is already on the list.

## TASK 3. DEVELOPMENT OF NEAR-TERM APPLICATIONS

The goal of this task is to develop near-term commercial applications for one or both of the advanced physical coal cleaning processes (column flotation and selective agglomeration) to process coal fines into marketable products. The task is divided into two subtasks:

- Subtask 3.1 - Engineering Analyses
- Subtask 3.2 - Engineering Development

The progress made during the quarter on each subtask is summarized below.

### Subtask 3.1. Engineering Analyses

Under this subtask, Amax selected three coal preparation plants (Ayrshire in Indiana, Wabash in Illinois, and Lady Dunn in West Virginia) for evaluation of near-term commercial application. Bechtel performed the engineering analyses which included development of conceptual flowsheets, selection of equipment, and estimation of capital and operating costs. The results were presented as a **draft** topical report to DOE, Amax Coal staff, and other project team members last quarter.

During the reporting quarter, Bechtel addressed the comments received on the **draft** topical report and finalized it. The final report was distributed to DOE and project team members on November 15, 1993.[6] The report presents conceptual designs and cost estimates for the addition of advanced coal cleaning circuits (column flotation or selective agglomeration) to process minus 100-mesh fines into marketable products (filter cake, dry powder, or briquettes). Three Amax coal preparation plants (Ayrshire, Lady Dunn, and Wabash) were evaluated as potential sites. Figure 3 shows the estimated processing cost in $ per ton for various scenarios (plant location, cleaning technology, and product to be sold).

The general conclusion is that column flotation will be less expensive than selective agglomeration. Drying and briquetting add significantly to the processing costs, and their inclusion may be justified only based on the premium price that can be obtained in some specific niche markets.

### Subtask 3.2. Engineering Development

Work will now move towards actual testing of advanced column flotation technology at bench scale under Subtask 3.2. The possibility of field testing at a larger scale will also be evaluated.

A visit was made to the Wabash Mine during the third week of October to discuss how a sample of minus 100-mesh fines can be collected. The line going to the thickener is of large diameter, and there is no possibility of collecting a sample from this line until at the point where it discharges into the thickener. At that point, flocculant has already been added, so flotation testing will not be effective. An alternative location is to collect a sample from one of the cyclone overflow lines. This approach will be followed by the plant personnel as time and other plant operation priorities permit.

## TASK 4. ENGINEERING DEVELOPMENT OF ADVANCED FROTH FLOTATION FOR PREMIUM FUELS

Task 4 activities during the quarter included work in each of the five subtask areas, that is, grinding, process optimization, CWF preparation, bench-scale testing, and conceptual design of the PDU.

As described in the Subtask 2.1 Coal Selection Plan and Recommendations[5] and in previous quarterly reports,[3,4] six coals were identified as good candidate feedstocks for conversion into premium fuel and selected for testing during Task 4. The six coals were as follows:

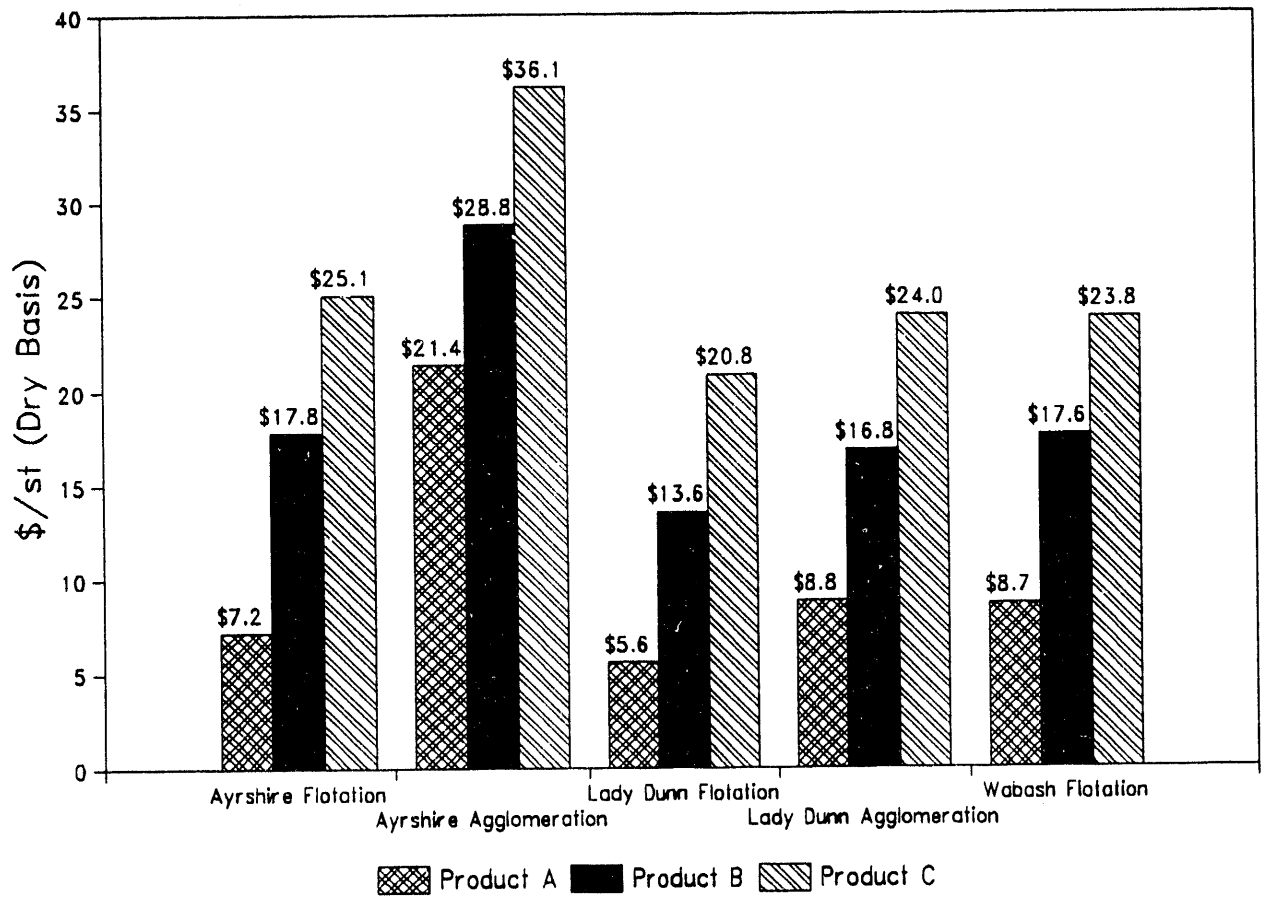



Figure 3. Processing cost summary (dollars per short ton).

Product A = Centrifuge Cake/Agglomerates

Product B = Dried Powder

Product C = Briquettes

| Coal | Mine | State | HGI | Ash, % | Sulfur, % |
|---|---|---|---|---|---|
| Taggart | Wentz | VA | 52 | 2.07 | 0.62 |
| Indiana VII | Minnehaha | IN | 55 | 9.25 | 0.49 |
| Sunnyside | Sunnyside | UT | 54 | 5.11 | 0.63 |
| Winifrede | Sandlick | WV | 47 | 8.42 | 0.94 |
| Elkhorn No. 3 | Chapperal | KY | 46 | 6.04 | 0.86 |
| Dietz | Spring Creek | MT | 41 | 4.98 | 0.33 |

The test coals were all washed bituminous coals except the Dietz coal which was a subbituminous coal that had only been crushed before marketing. Washing plant carbon and heating value recoveries were in the 89 to 94 percent range for the five bituminous coals. Thus, near 90 percent carbon and heating value recoveries are necessary during the advanced flotation step in order to meet the project goal of recovering 80 percent of the carbon from the run of mine coals.

## Subtask 4.1. Grinding

The laboratory and continuous bench-scale portions of the grinding studies were completed as described in the last quarterly report. The topical report for Subtask 4.1 was drafted during the fifth quarter and will be issued during January 1994 for DOE's comments and approval.

The principal objective of the subtask was to determine the degree of grinding and liberation required for each test coal in order to meet the target ash and sulfur specifications of premium fuel. The acquisition of grinding circuit parameters for design of the 1.8 tph PDU was an important additional objective of the subtask.

As described in previous quarterly reports,[2-4] the five bituminous test coals were ground at rates between 50 and 500 kg/hr in continuous circuits with various configurations of mills and classifiers. Grinding studies were not done on the Dietz coal because of the uncertainty regarding development of a feasible froth flotation process for subbituminous coal. Liberation testing indicated that the following grind sizes (D80 = 80 percent weight passing) would be needed in order to meet the ash and sulfur specifications:

| Test Coal | D80, μm |
|---|---|
| Taggart | 45 |
| Indiana VII | 20 |
| Sunnyside | 45 |
| Winifrede | 11 |
| Elkhorn No. 3 | 45 |

Depending upon the liberation characteristics of the specific test coal, either single-stage closed-circuit ball milling or two-stage closed-circuit ball milling/stirred ball milling was recommended for the individual coals during the Phase II PDU operation.

## Subtask 4.2. Process Optimization Research

The process optimization studies scheduled in the Subtask 4.2 Test Plan[7] continued during the quarter. Parametric testing using laboratory-scale flotation columns was accomplished on the Sunnyside and Winifrede coals at CAER, on the Winifrede and Indiana VII coals at VPI, and on the Winifrede and Taggart coals at Amax R&D during the quarter. (CAER had completed their parametric testing of Elkhorn No. 3 coal during the third quarter.[3]) CAER also investigated the use of a packed column during the latest quarter and continued comparison testing between external and internal spargers. In all but one instance, the ground coals were provided to the flotation laboratories in slurry form by Amax R&D. CAER prepared their own coarse grind Elkhorn No. 3 slurry.

The laboratory columns are 100 mm (4 inches) in diameter with a standard height of 6.1 meters (20 feet). CAER is testing internal (Foam-Jet) and external (Bureau of Mines) air sparging systems. The Amax R&D laboratory column is set with a porous metal sparger. VPI is using 50 and 75-mm columns fitted with external Microcel air sparging systems. CAER normally emulsified the reagents and added them to the sparger water. The reagents were premixed with the feed slurry at VPI and Amax R&D.

### Parametric Testing of Winifrede Coal

Finely ground (nominally passing 325 mesh) Winifrede coal slurry was tested at each laboratory in order to provide comparisons between performance of equipment at the respective locations. In each case, the slurry was prepared at Amax R&D, and the coal in the slurry was ground to a D80 of 18 $\mu m$. This slurry was chosen for the interlaboratory comparisons because it represents a) a coal known to respond well to ordinary froth flotation, b) a particle size distribution that many observers expect to be typical for premium fuel production, and c) marginal mineral matter liberation that will challenge the ash-rejection capabilities of the respective column flotation procedures.

**Results at CAER**: The parametric testing at CAER on the Winifrede slurry was described in the 4th quarterly report.[4] The following optimized operating conditions were projected for their 100-mm column when equipped with an external sparger:

| | |
|---|---|
| Airflow, Liters/Minute | 4.6 |
| Solids Content, % | 11.7 |
| Retention Time, Minutes | 7.3 |
| Wash Water, Liters/Minute | 0.5 |

These operating conditions were projected to recover 90 percent of the clean coal at an ash content of 3.3 percent (976 g/GJ or 2.22 lb/mmBtu). Comparable results were obtained during the 5th quarter when using the internal sparger.

**Results at VPI**: Preliminary flotation tests were made on the Winifrede slurry at VPI in the 50-mm column fitted with the Microcel aeration system, and additional testing is in progress in the 75-mm system. The best operating point for the 50-mm column was at a HHV recovery of 94 percent and at a clean coal ash content of 3.8 percent. Subsequently, 85 percent of the HHV was recovered at an ash content of 2.9 percent (430 g/GJ or 1.0 lbs/mmBtu) during parametric testing in the larger system. Figure 4 is the grade-recovery curve for the latter work. Further evaluation of the data is in progress.

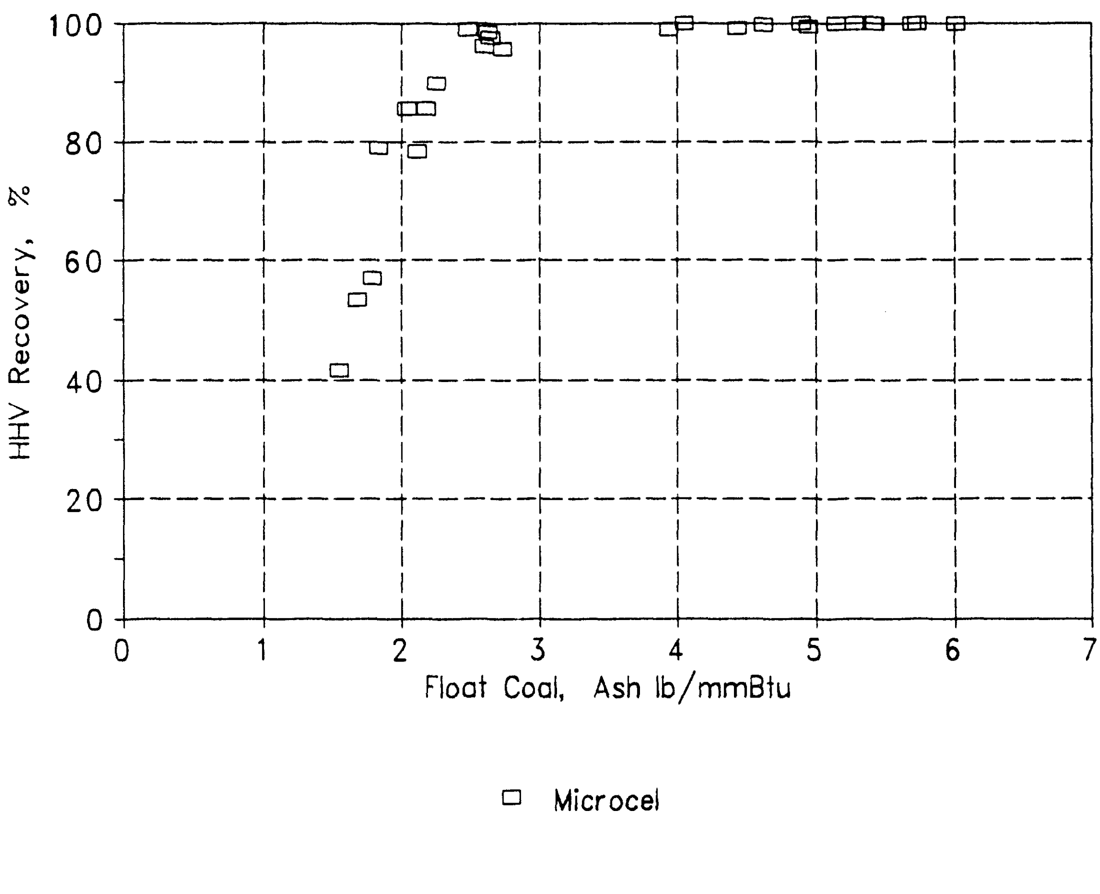



Figure 4. Grade-recovery curve obtained by Microcel flotation of fine Winifrede coal at VPI.

**Results at Amax R&D**: Thirty-nine tests were made on the Winifrede slurry at Amax R&D. The initial work was a series of 27 tests arranged in a 4-factor Box-Behnken matrix of variables. The set was subsequently augmented by additional tests in the regions of particular interest (low ash and/or high recovery of the heating value). The process parameters varied during the initial matrix were dry feed rate, retention time, wash water ratio, and aeration rate. MIBC and diesel fuel additions were held between 18 and 20 ppm each on a slurry basis during the initial matrix tests, but increased dosages were investigated during the follow-up work. The actual ranges covered by the fixed and floating variables during the testing are shown in Table 2. Test results are summarized in Table 3.

Table 2. Actual Ranges of Flotation Parameters During Matrix Testing of Winifrede Coal Slurry at Amax R&D

| | Center Point | Range |
|---|---|---|
| Dry Feed, Grams/Minute | 76.6 | 9 - 119 |
| Feed Slurry, % Solids | 6.6 | 1.0 - 10.3 |
| Aeration, Liters/Minute | 3.9 | 2 - 6 |
| Aerator Water, Liters/Minute | None Used | |
| Retention Time, Minutes | 17.6 | 12.5 - 23.3 |
| Wash Water, Liters/Minute | 0.5 | 0.2 - 0.8 |
| Wash Water Ratio, Liters/kg | 14.7 | 3.9 - 44<br>- (405)[a] |
| Bias Ratio | 0.31 | -0.46 - 0.92<br>(-1.4) - (1.02) |
| Diesel Fuel, kg/t | 0.5 | 0.25 - 0.90<br>- (2.3) |
| MIBC, kg/t | 0.5 | 0.25 - 0.90<br>- (2.3) |
| MIBC, ppm Slurry | 19.6 | 16 - 23 |
| HHV Yield, % | 66 | 14.9 - 99.8<br>(1.7) - (100) |
| Ash in Clean Coal, g/GJ | 1,080 | 820 - 1,630 |
| Production, t/h/sq m | 0.49 | 0.15 - 1.00<br>(0.01) - |

[a] Values in parentheses are extreme values and represent tests that were not included in correlations.

Table 3. Parametric Column Flotation Testing of Winifrede Coal at Amax R&D

| Test | Dry Coal, g/min | Aeration l/min | Retention Time, Minutes | Wash Water Ratio | Ash, % | Ash, lb/mmBtu | S, % | S, lb/mmBtu | HHVc Recovery, % | Hancock Efficiency HElash |
|---|---|---|---|---|---|---|---|---|---|---|
| CLM31 | 63.1 | 4 | 23.3 | 8.7 | 4.67 | 3.28 | 0.88 | 0.62 | 100.0 | 40.3 |
| CLM32 | 85.4 | 2 | 22.5 | 4.6 | 4.81 | 3.38 | | | 100.0 | 35.4 |
| CLM33 | 82.9 | 6 | 21.3 | 6.6 | 5.82 | 4.14 | | | 100.0 | 25.5 |
| CLM34 | 37.5 | 2 | 18.9 | 14.4 | 2.97 | 2.04 | 0.81 | 0.56 | 69.5 | 47.2 |
| CLM35 | 111.7 | 2 | 16.7 | 30.7 | 3.20 | 2.21 | | | 19.5 | 12.2 |
| CLM36 | 46.9 | 6 | 17.9 | 7.8 | 4.47 | 3.13 | 0.89 | 0.62 | 99.8 | 45.3 |
| CLM37 | 112.3 | 6 | 16.1 | 10.3 | 3.79 | 2.63 | | | 57.1 | 31.6 |
| CLM38 | 79.8 | 4 | 12.5 | 4.7 | 4.16 | 2.90 | 0.87 | 0.61 | 99.9 | 50.0 |
| CLM39 | 76.5 | 4 | 12.5 | 31.6 | 2.92 | 2.01 | | | 32.4 | 23.5 |
| CLM40 | 80.9 | 4 | 20.1 | 7.1 | 3.31 | 2.29 | | | 66.1 | 38.4 |
| CLM41 | 69.6 | 4 | 21.5 | 29.2 | 3.01 | 2.07 | | | 33.5 | 22.0 |
| CLM42 | 84.5 | 4 | 16.4 | 10.3 | 3.07 | 2.12 | | | [illegible] | 41.7 |
| CLM43 | 44.4 | 4 | 13.8 | 8.5 | 4.20 | 2.93 | 0.85 | 0.59 | 99.6 | 47.9 |
| CLM44 | 118.7 | 4 | 13.8 | 19.9 | 3.08 | 2.12 | | | 32.2 | 20.0 |
| CLM45 | 31.6 | 4 | 23.1 | 13.3 | 3.32 | 2.30 | 0.90 | 0.62 | 91.9 | 62.1 |
| CLM46 | 92.7 | 4 | 16.5 | 13.1 | 2.97 | 2.04 | 0.85 | 0.59 | 48.8 | 31.5 |
| CLM47 | 82.5 | 2 | 17.8 | 17.2 | 2.92 | 2.01 | | | 26.9 | 17.7 |
| CLM48 | 95.3 | 6 | 16.4 | 3.9 | 5.42 | 3.84 | | | 99.4 | 31.9 |
| CLM49 | 85.2 | 2 | 17.1 | 44.0 | 2.94 | 2.02 | | | 20.4 | 13.6 |
| CLM50 | 86.5 | 6 | 16.6 | 21.3 | 3.11 | 2.14 | | | 41.5 | 26.8 |
| CLM51 | 88.2 | 4 | 16.2 | 11.3 | 3.10 | 2.14 | | | 57.3 | 35.5 |
| CLM52 | 51.7 | 4 | 17.0 | 4.8 | 4.43 | 3.10 | 0.87 | 0.61 | 99.8 | 45.0 |
| CLM53 | 114.1 | 4 | 16.7 | 8.2 | 3.65 | 2.53 | | | 54.4 | 30.9 |
| CLM54 | 47.2 | 4 | 17.0 | 10.6 | 3.80 | 2.64 | 0.85 | 0.59 | 99.8 | 51.6 |
| CLM55 | 114.3 | 4 | 17.2 | 6.9 | 3.79 | 2.63 | | | 63.5 | 34.1 |
| CLM56 | 95.4 | 2 | 13.7 | 40.3 | 2.74 | 1.88 | | | 14.9 | 10.0 |
| CLM57 | 89.2 | 4 | 13.0 | 7.4 | 3.70 | 2.57 | 0.88 | 0.61 | 85.7 | 46.8 |
| CLM58 | 83.2 | 2 | 21.1 | 405.5 | 2.75 | 1.89 | | | 1.7 | 1.2 |
| CLM59 | 87.2 | 4 | 20.6 | 15.4 | 3.60 | 2.50 | | | 42.3 | 24.3 |
| CLM60 | 84.9 | 4 | 16.7 | 12.3 | 3.65 | 2.53 | | | 54.5 | 31.0 |
| CLM61 | 85.1 | 4 | 20.4 | 10.8 | 3.62 | 2.51 | 0.87 | 0.60 | 72.7 | 38.7 |
| CLM62 | 87.0 | 5 | 16.2 | 8.3 | 3.58 | 2.48 | 0.90 | 0.62 | 79.0 | 45.3 |
| CLM63 | 93.3 | 4 | 14.3 | 10.5 | 3.74 | 2.60 | 0.86 | 0.60 | 86.2 | 45.8 |
| CLM64 | 17.4 | 4 | 14.3 | 43.3 | 4.60 | 3.23 | | | 99.0 | 39.7 |
| CLM65 | 59.6 | 4 | 20.6 | 11.1 | 3.18 | 2.19 | | | 57.6 | 35.6 |
| CLM66 | 88.7 | 4 | 13.2 | 11.2 | 3.77 | 2.62 | 0.91 | 0.63 | 85.7 | 5.6 |
| CLM67 | 90.8 | 5 | 17.1 | 9.1 | 5.91 | 4.21 | | | 100.0 | 26.5 |
| CLM68 | 8.3 | 2 | 25.4 | 43.9 | 4.14 | 2.89 | 0.87 | 0.61 | 46.7 | 0.0 |
| CLM69 | 34.3 | 4 | 19.2 | 14.6 | 4.05 | 2.82 | 0.90 | 0.63 | 48.1 | 5.3 |

Figure 5 is a grade-recovery curve for the laboratory column flotation data from the Amax R&D parametric testing. As seen earlier by CAER and VPI, a <860 g/GJ ash specification was not met when recovering 90 percent of the heating value of the Winifrede coal. Furthermore, sacrificing recovery did little to improve ash rejection during the laboratory testing.

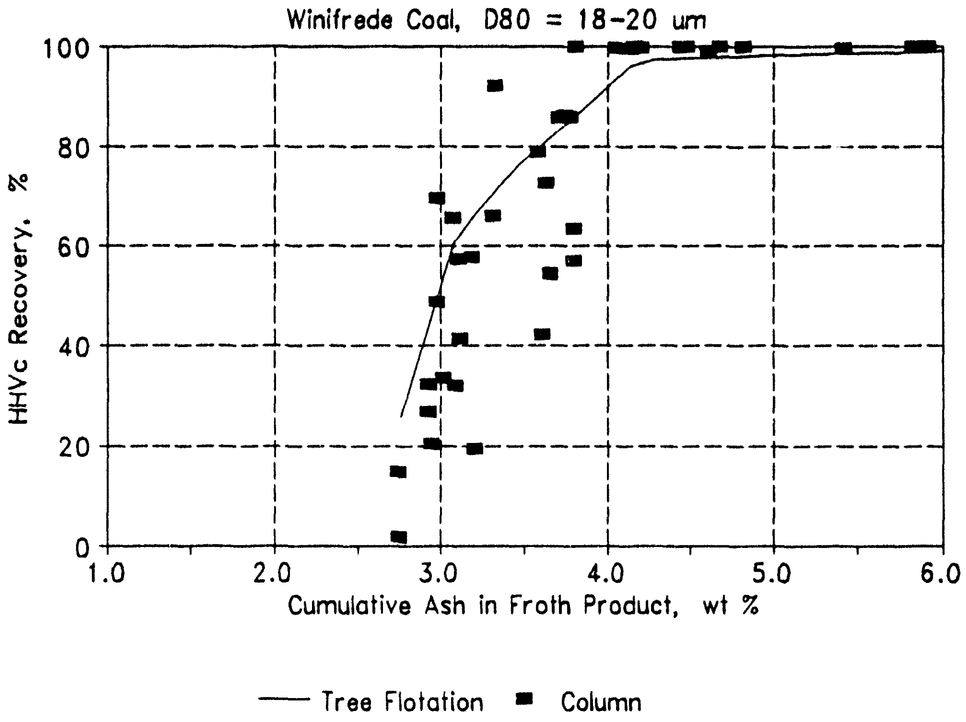



Figure 5. Grade-recovery data for laboratory column flotation of fine Winifrede coal at Amax R&D. Line plots the Denver cell tree flotation results.

The deviations of individual test results from the average grade-recovery curve are being studied by regression analysis[8] to identify the operating variables which have significant impact on coal recovery and ash rejection. All of the variables listed in Table 2 were considered during the statistical treatment of the data. The statistical analysis completed so far on the Amax R&D flotation data indicates that the feed solids concentration and wash water ratio have the greatest impact upon ash reduction when reagent levels, retention times, and aeration are set to achieve high recovery of the heating value in the coal. There was little indication from this analysis that it will be possible to achieve less than 3.4 percent ash (1,030 g/GJ) in the clean coal while maintaining a 90 percent HHV yield from this slurry.

**Further Work on Winifrede Coal**: At a PSD of D80 = 18 μm, the Winifrede coal in the test slurry was not ground quite fine enough for complete ash liberation. This was confirmed by a selective agglomeration test performed on the slurry. The resulting clean coal contained 3.72 percent ash or 1,120 g/GJ (2.6 lb/mmBtu). The VPI work provides encouragement that sufficient composite middling could be rejected by column flotation at this grind so that the froth would come close to the target ash and HHV recovery specifications, but the obvious direction to move in order to insure rejection of more ash from the Winifrede coal would be to grind the coal finer than D80 = 18.

The Subtask 4.1 grinding study summarized earlier in this report indicated that Winifrede coal should be ground to D80 <11 μm in order to achieve the desired liberation. For this reason, two comparison laboratory flotation experiments were conducted in a Denver cell. Both were rougher flotation tests followed by two stages of cleaner flotation to simulate the washing action occurring in a column. One test was on a cut of the slurry used for the column flotation testing and the other test was on a cut of the slurry that had been ground an additional amount in a laboratory batch stirred ball mill. Table 4 is a comparison of the two test results. The additional grinding cut the D80 in half, to 9.1 μm, but made the flotation separation more difficult rather than easier. An agglomeration test on the same reground slurry produced clean coal containing 2.46 percent ash or 727 g/GJ (1.7lb/mmBtu). That matched the ash liberation seen earlier on extra finely ground Winifrede coal.[4]

Table 4. Denver Cell Flotation of
Original and Reground Winifrede Slurry

| | Slurry | |
|---|---|---|
| | Original | Reground |
| PSD D80, μm | 17.8 | 9.10 |
| Second Cleaner Float | | |
| Dry Weight, % | 81.2 | 78.8 |
| HHV Yield, % | 86.1 | 83.3 |
| Ash, % | 3.28 | 3.81 |
| Ash, lb/mmBtu | 2.27 | 2.65 |
| Total Sulfur, % | 0.85 | 0.85 |
| Total Sulfur, lb/mmBtu | 0.59 | 0.59 |
| Pyrite Sulfur, % | 0.065 | 0.081 |
| Pyrite Sulfur, lb/mmBtu | 0.045 | 0.056 |

## Parametric Testing of Taggart Coal

The laboratory column flotation testing of Taggart coal was conducted at

Amax R&D. A statistically designed set of 15 tests was run on Taggart coal that had been ground in the 1.2-m ball mill in closed circuit with a 62-mesh screen. Three variables were investigated (dry feed rate, retention time, and wash water rate). A fixed aeration rate of 4 liters/minute was used for these tests since that airflow performed well in previous testing. The ranges of the set and resulting operating parameters are summarized in Table 5.

Table 5. Actual Ranges of Flotation Parameters During Matrix Testing of Taggart Coal Slurry at Amax R&D

| | Center Point | Range |
|---|---|---|
| Dry Feed, Grams/Minute | 98.36 | 63 - 129 |
| Feed Slurry, % Solids | 7.2 | 3.3 - 9.9 |
| Aeration, Liters/Minute | 4.0 | 4 (Fixed) |
| Aerator Water, Liters/Minute | None Used | |
| Retention Time, Minutes | 13.7 | 11.0 - 17.3 |
| Wash Water, Liters/Minute | 0.5 | 0.4 - 1.0 |
| Wash Water Ratio, Liters/kg | 7.2 | 5.1 - 9.3 |
| Bias Ratio | 0.62 | 0.32 - 0.62 |
| Diesel Fuel, kg/t | 0.45 | 0.33 - 0.79 |
| MIBC, kg/t | 0.45 | 0.33 - 0.79 |
| MIBC, ppm Slurry | 19.9 | 18 - 22 |
| HHV Yield, % | 99.6 | 99.4 - 99.7 |
| Ash in Clean Coal, g/GJ | 422 | 380 - 450 |
| Production, t/h/sq m | 1.06 | 0.69 - 1.39 |

The D80 of the Taggart feed slurry was 101 $\mu$m. This PSD was considerably coarser than the PSD of the test slurries prepared from Winifrede, Indiana VII, and Sunnyside coals. As expected, the Taggart coal responded very well to column flotation. Heating value recovery consistently exceeded 99 percent, as shown in Table 6. The resulting clean coal contained between 380 and 450 g ash/GJ (1.34 and 1.58 percent ash or 0.88 and 1.04 lb/mmBtu). The amounts of sulfur in the clean coals easily met the target specification of less than 278 g/GJ. A more extensive statistical analysis will be performed on the data, but at this point, it appears that the full capability of the column flotation system was not tested during this series of experiments. Higher feed rates should be investigated to define the upper capacity of unit, and increasing wash water rates and/or finer grind should also be investigated in order to produce clean coal containing <430 g ash/GJ on a consistent basis. The present plan, though, is to defer such testing until bench-scale flotation during Subtask 4.4 .

## Table 6. Parametric Column Flotation Testing of Taggart Coal at Amax R&D

| Test | Dry Coal, g/min | Aeration l/min | Retention Time, Minutes | Wash Water Ratio | Ash, % | Ash, lb/mmBtu | S, % | S, lb/mmBtu | HHVc Recovery, % | Hancock Efficiency Index |
|---|---|---|---|---|---|---|---|---|---|---|
| CLM70 | 64.6 | 4 | 14.2 | 5.62 | 1.54 | 1.01 | 0.56 | 0.37 | 99.7 | 20.1 |
| CLM71 | 121.3 | 4 | 14.7 | 5.99 | 1.52 | 1.00 | 0.56 | 0.37 | 99.6 | 22.8 |
| CLM72 | 67.6 | 4 | 14.6 | 8.94 | 1.50 | 0.99 | 0.59 | 0.39 | 99.7 | 17.3 |
| CLM73 | 121.6 | 4 | 12.5 | 8.18 | 1.50 | 0.99 | 0.57 | 0.37 | 99.5 | 23.1 |
| CLM74 | 102.8 | 4 | 14.0 | 7.06 | 1.52 | 1.00 | 0.56 | 0.37 | 99.6 | 21.7 |
| CLM75 | 63.0 | 4 | 12.1 | 7.68 | 1.55 | 1.02 | 0.59 | 0.39 | 99.6 | 20.6 |
| CLM76 | 129.3 | 4 | 11.0 | 7.49 | 1.48 | 0.97 | 0.59 | 0.39 | 99.5 | 21.2 |
| CLM77 | 66.0 | 4 | 17.3 | 7.33 | 1.37 | 0.90 | 0.58 | 0.38 | 99.7 | 24.5 |
| CLM78 | 129.1 | 4 | 12.1 | 7.43 | 1.49 | 0.98 | 0.57 | 0.37 | 99.5 | 22.1 |
| CLM79 | 100.9 | 4 | 14.1 | 7.20 | 1.48 | 0.97 | 0.60 | 0.39 | 99.6 | 23.4 |
| CLM80 | 101.2 | 4 | 11.0 | 5.39 | 1.44 | 0.95 | 0.57 | 0.37 | 99.4 | 22.6 |
| CLM81 | 104.6 | 4 | 11.2 | 8.69 | 1.57 | 1.03 | 0.58 | 0.38 | 99.5 | 22.0 |
| CLM82 | 107.2 | 4 | 17.3 | 5.07 | 1.50 | 0.99 | 0.60 | 0.39 | 99.7 | 20.8 |
| CLM83 | 97.8 | 4 | 14.9 | 9.28 | 1.34 | 0.88 | 0.58 | 0.38 | 99.6 | 25.9 |
| CLM84 | 97.7 | 4 | 14.5 | 7.43 | 1.51 | 0.99 | 0.60 | 0.39 | 99.6 | 22.1 |

## Parametric Testing of Indiana VII Coal

Parametric laboratory column flotation testing of nominally minus 325-mesh Indiana VII coal slurry was assigned to VPI. The testing has been completed and the results were being evaluated by VPI at the end of the quarter.

## Parametric Testing of Sunnyside Coal

Preliminary testing of Sunnyside coal was described last quarter. The preliminary results were used to statistically design a set of 20 experiments which were conducted at CAER on Sunnyside coal that had been ground to nominally passing 325 mesh (D80 = 21 μm). These were followed by additional tests to determine the effects of increasing wash water and to compare performance of the internal and external sparging systems.

Table 7 lists the experimental conditions and percent ash in clean coal and HHV recovery data for the series of tests conducted using the statistically designed experiments. The HHV recovery ranges from 10.5 to 99.6 percent and ash content ranges from 1.43 to 2.93 percent depending on the operating condition utilized.

Table 7. Column Flotation Parameters and Percent Ash and HHV Recovery Obtained Using Statistically Designed Experiments for Sunnyside Coal

| Column Parameters | | | Results | |
|---|---|---|---|---|
| Airflow, Liters/Minute | Retention Time, Minutes | Feed Slurry Solids Conc., Weight % | HHV Recovery | Percent Ash |
| 2.0 | 4.10 | 10.0 | 16.60 | 1.79 |
| 5.0 | 7.20 | 5.0 | 89.20 | 1.84 |
| 2.0 | 4.10 | 5.0 | 61.60 | 2.66 |
| 3.5 | 5.65 | 7.5 | 22.70 | 1.25 |
| 2.0 | 7.20 | 10.0 | 37.40 | 1.82 |
| 5.0 | 7.20 | 10.0 | 65.80 | 1.73 |
| 3.5 | 5.65 | 7.5 | 25.10 | 1.38 |
| 2.0 | 7.20 | 5.0 | 56.80 | 1.93 |
| 3.5 | 5.65 | 7.5 | 25.40 | 1.60 |
| 3.5 | 5.65 | 7.5 | 23.30 | 1.60 |
| 5.0 | 4.10 | 10.0 | 32.70 | 2.29 |
| 5.0 | 4.10 | 5.0 | 60.90 | 2.93 |
| 3.5 | 5.65 | 7.50 | 19.10 | 1.60 |
| 3.5 | 5.65 | 3.29 | 61.20 | 2.04 |
| 6.0 | 5.65 | 7.50 | 46.80 | 2.15 |
| 3.5 | 5.65 | 7.50 | 29.30 | 1.64 |
| 3.5 | 5.65 | 11.71 | 17.40 | 1.43 |
| 3.5 | 8.25 | 7.50 | 99.60 | 2.27 |
| 3.5 | 3.04 | 7.50 | 17.90 | 1.86 |
| 0.9 | 5.65 | 7.50 | 10.50 | 1.84 |

A response surface curve (contour plot) and block diagram for solids concentration and retention time for HHV recovery at a fixed airflow of 3.5 liters is shown in Figure 6. This figure shows that the maximum HHV recovery is obtained at high retention time (8.2 minutes) and low solids (3.3 percent) concentration. Similarly, a response surface block diagram and contours for percent ash in clean coal is shown in Figure 7. It shows that the lowest ash is obtained at high solids (approximately 10.3 percent) and about 5.8 minutes retention time. Based on the statistical analysis of all data, the optimum column conditions for flotation of the Sunnyside coal were determined to be as follows:

| | |
|---|---|
| Airflow, Liters/Minute | 3.98 |
| Retention Time, Minutes | 7.10 |
| Solids Concentration, Weight % | 3.29 |

Using these conditions, theoretically a clean coal product containing 1.75 percent ash (520 g/GJ or 1.2 lb/mmBtu) could be obtained with 90 percent HHV recovery.

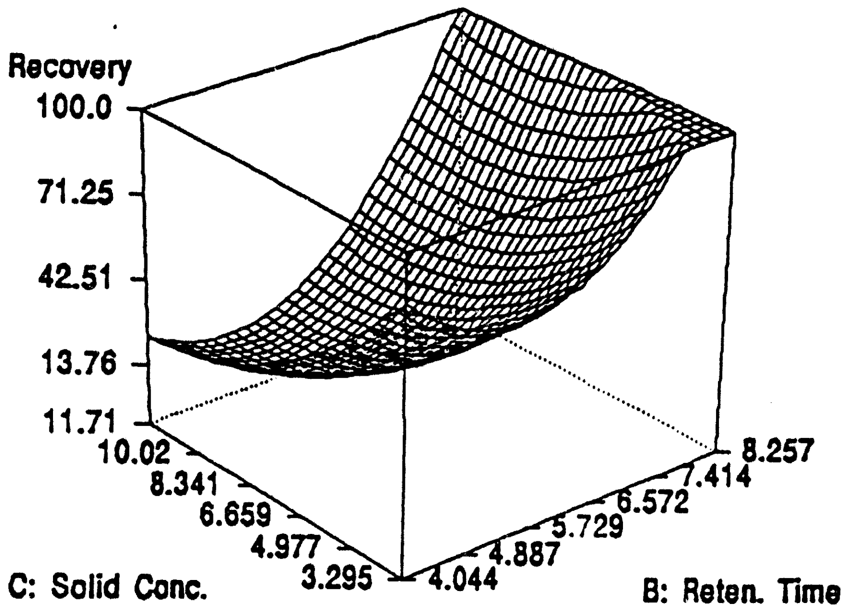
A series of column flotation tests were conducted using the optimum conditions and varying only the wash water addition rate. Figure 8 shows the HHV recovery and percent ash in clean coal obtained using the external and internal spargers. It shows that the external sparger provided more than 90 percent and the internal sparger provided close to 80 percent HHV recovery. However, the ash content of the product using the internal sparger was lower (approximately 1.7 percent, 500 g/GJ) compared to the external sparger which produced clean coal containing about 2.1 percent ash (620 g/GJ).

Figure 9 shows all the column and laboratory tree flotation data for the Sunnyside coal. Note that most of the column data are very close to the tree flotation curve.

## Parametric Testing of Packed Column

A 100-mm diameter by 6-m packed laboratory column was purchased from GNL&V Ontario and installed in the CAER laboratory. The packed column is filled with baffles to distribute the flow of slurry and air bubbles across the diameter of the column. Air is admitted directly into the bottom of the column without any special sparging or dispersion device. GNL&V markets the column for the inventor, Dr. David Yang formerly of Michigan Technical University.

Parametric testing of the packed column began during the quarter to compare its performance with the performance of columns with the internal and external air spargers. The parametric tests have been completed on minus 325-mesh Winifrede and Sunnyside coals.



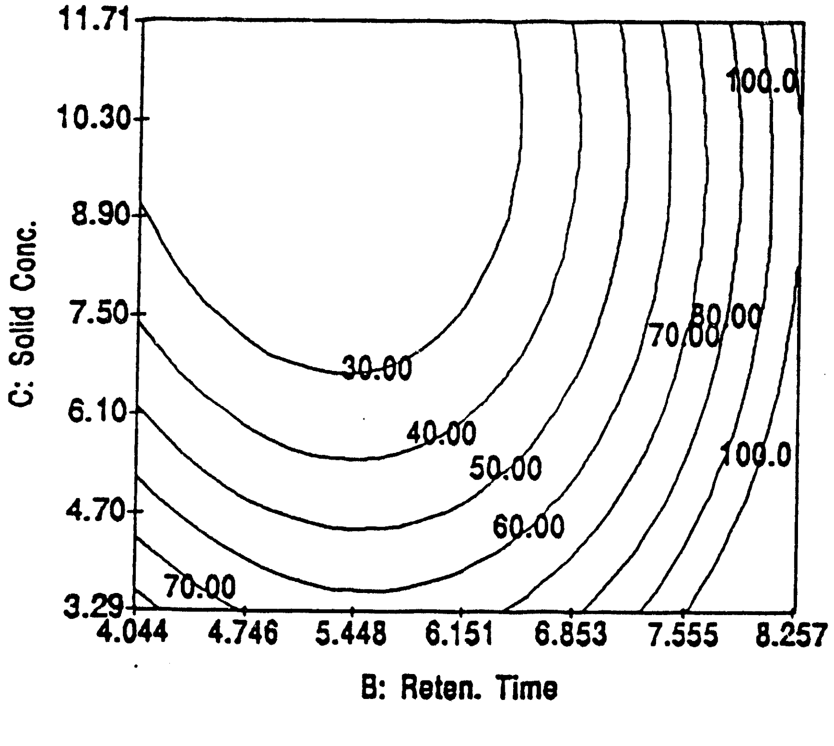




Figure 6. Response surface block diagram and contours for solids concentration and retention time for HHV recovery at a fixed airflow of 3.5 liters/minute.

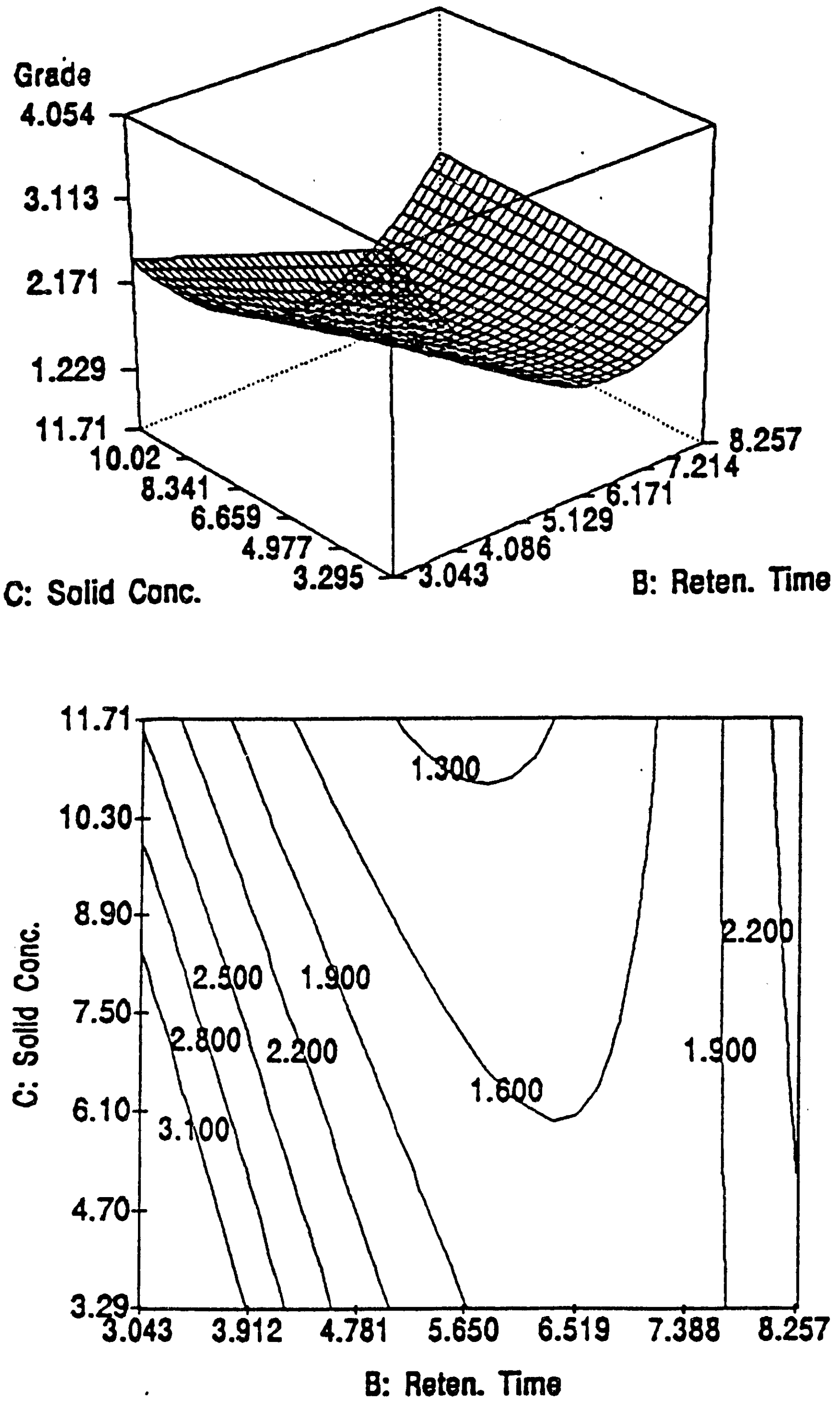



Figure 7. Response surface block diagram and contours for solids concentration and retention time for percent ash in clean coal at a fixed airflow of 3.5 liters/minute.

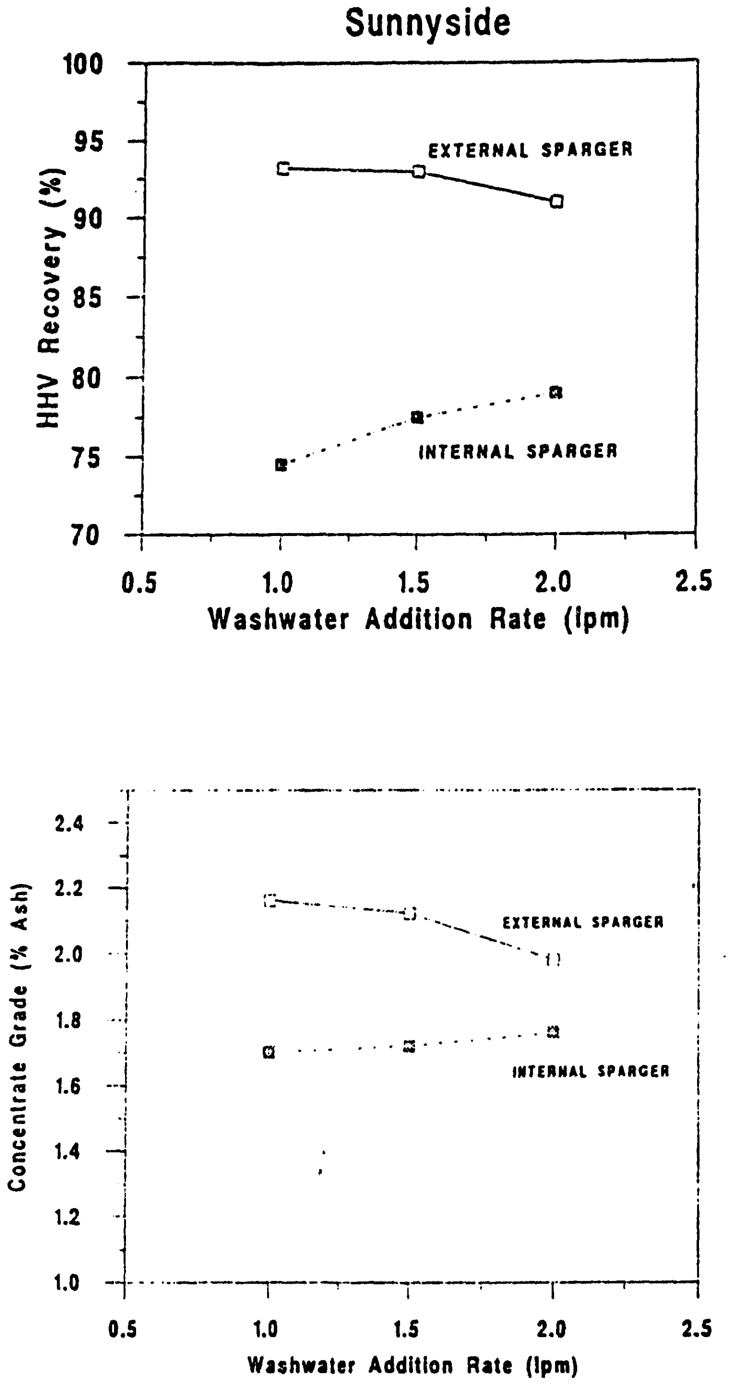



Figure 8. HHV recovery and percent ash in clean coal as a function of wash water addition for Sunnyside coal (4 liters/minute airflow, 7.1 minutes retention time, and 3.3% solids).

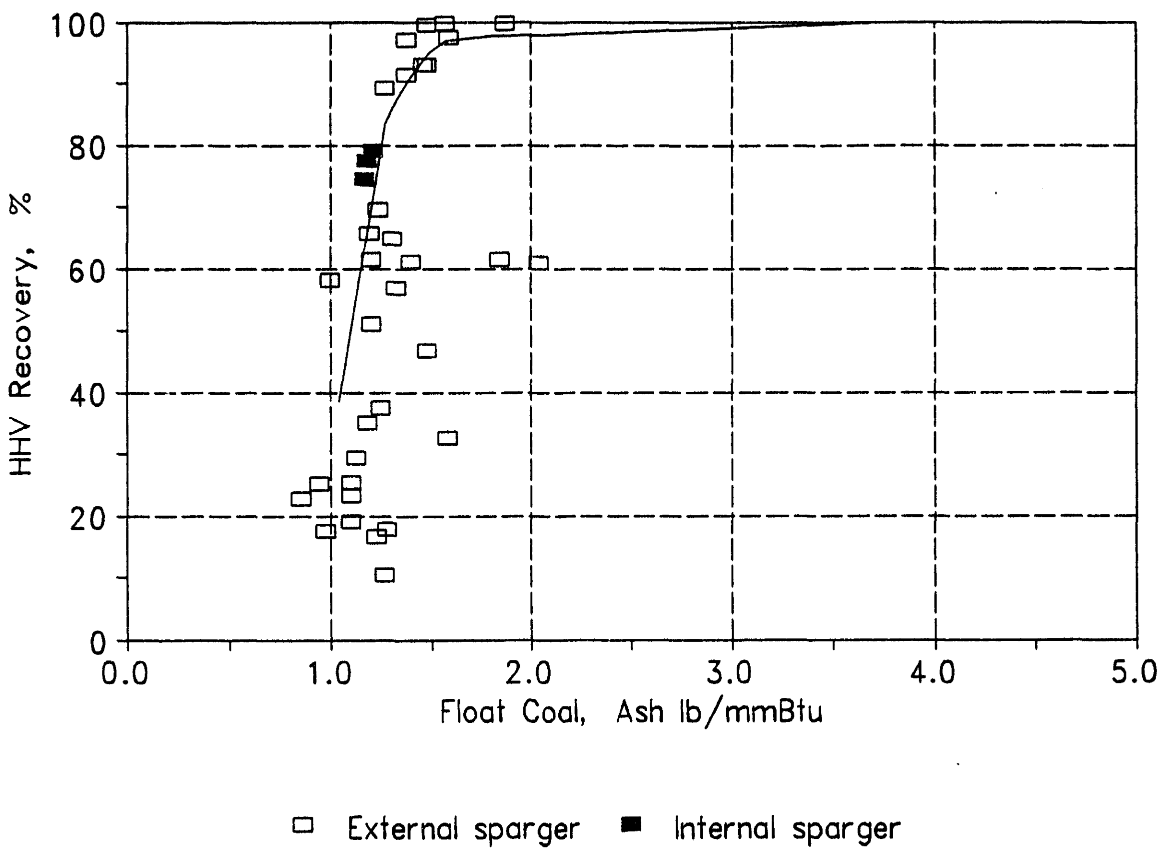



Figure 9. Column flotation and release analysis curve for Sunnyside coal.

**Procedure**: A packed column of 10-cm (4-inch) I.D. and 6-m (20-foot) height was utilized for the flotation studies. For each test, the column was filled first with water containing frother. Flotation reagents, fuel oil (0.25 Kg/t), and MIBC (0.25 Kg/t) were added on-line as an emulsion to the feed slurry. Froth depth in the column was kept constant at 1 meter for all the tests. The pressure of air utilized for bubble generation was kept at 20 psi. Samples of froth and tailings were collected after allowing at least double the retention time of the feed slurry in the column.

**Winifrede Coal**: All of the packed column flotation tests were conducted with ultra-fine (D80 = 18 μm) ground coal slurry provided by Amax R&D. Table 8 lists the experimental conditions, percent ash in clean coal, and HHV recovery data for the series of tests conducted using statistically designed experiments. The experimental conditions for the test matrix were established by a separate series of preliminary testing of Winifrede coal in the column. Note that the percent ash in the clean coal ranged from 1.93 to 7.08 at HHV recoveries ranging from 2.9 to 99.6 percent. A low ash product was obtained at low HHV recovery.

Table 8. Packed Column Flotation for Winifrede Coal (Wash Water ~ 1 Liter/Minute)

| Airflow,[a] Liters/Minute | Retention Time, Minutes | Solids Concentration, Weight % | Feed Rate, Kg/hr | Ash, % | HHV Recovery, % |
|---|---|---|---|---|---|
| 10 | 6 | 5 | 22.3 | 4.14 | 8.3 |
| 30 | 6 | 5 | 22.3 | 5.71 | 85.2 |
| 10 | 10 | 5 | 7.4 | 3.96 | 30.2 |
| 30 | 10 | 5 | 7.4 | 6.54 | 99.3 |
| 10 | 6 | 10 | 44.6 | 2.06 | 2.9 |
| 30 | 6 | 10 | 44.6 | 6.88 | 72.3 |
| 10 | 10 | 10 | 14.7 | 1.93 | 3.5 |
| 30 | 10 | 10 | 14.7 | 7.08 | 91.9 |
| 20 | 8 | 7.5 | 17.1 | 5.46 | 73.7 |
| 20 | 8 | 7.5 | 17.1 | 5.54 | 73.5 |
| 20 | 8 | 7.5 | 17.1 | 5.95 | 85.5 |
| 20 | 8 | 7.5 | 17.1 | 5.90 | 80.9 |
| 3 | 8 | 7.5 | 17.1 | 1.73 | 7.6 |
| 3 | 8 | 7.5 | 17.1 | 7.71 | 99.3 |
| 20 | 4.6 | 7.5 | 29.1 | 6.08 | 68.8 |
| 20 | 11.3 | 7.5 | 9.2 | 6.68 | 99.3 |
| 20 | 8 | 3.4 | 6.5 | 5.00 | 56.9 |
| 20 | 8 | 11.7 | 23.3 | 6.98 | 99.6 |
| 20 | 8 | 7.5 | 17.1 | 6.17 | 93.0 |
| 20 | 8 | 7.5 | 17.1 | 6.23 | 92.3 |

[a] Air pressure maintained at 20 psi.

A response surface curve (contour) and block diagram for solids concentration and airflow at a fixed retention time of 8 minutes for HHV recovery are shown in Figure 10. Note that a high (>90 percent) HHV recovery occurs at high (25 liters/minute) airflow using about 7.5 weight percent solids in the feed slurry. The response surface block diagram and contours for the ash content of the clean coal (Figure 11) show that the lowest ash in the clean coal is obtained at high (~ 10 percent) feed solids and low (~ 4 liters/minute) airflow rates.

Based on the statistical analysis of all data, the optimum conditions for flotation of Winifrede coal using the packed column were determined to be as follows:

| | |
|---|---|
| Airflow, Liters/Minute | 36.8 |
| Retention Time, Minutes | 3.29 |
| Solids Concentration, Weight % | 11.3 |

Under these conditions, it is projected that a clean coal product containing 4.9 percent ash at 89.8 percent HHV recovery would be obtained from the Winifrede slurry.

Figure 12 shows the effect of increased wash water rates on the HHV recovery and the ash content of clean coal obtained using the optimized column operating conditions. Note that increasing wash water had minimum effect on HHV recovery which remained at >90 percent; however, the ash content of the clean coal decreased from 5.1 to 4.25 percent as wash water rate was increased from 1 to 3 liters/minute.

Sunnyside Coal: Minus 325-mesh ground coal slurry with D80 = 21 $\mu$m, prepared from Sunnyside coal, was supplied by Amax R&D. Table 9 lists the packed column experimental conditions, percent ash in clean coal, and HHV recovery data for the series of tests conducted using statistically designed experiments. As for the Winifrede slurry, these test conditions were established by preliminary tests in packed column. Note that the ash content of clean coal varies from 1.10 to 4.93 percent and HHV recovery ranges from 9.4 to 99.9 percent depending on the experimental conditions.

An HHV recovery response surface block diagram and contour plot for varying solids concentration and airflow at a constant 5 minutes retention time are shown in Figure 13. High (>90 percent) HHV recovery is obtained using about 3 weight percent solid slurry. Similarly, Figure 14 shows the response surface block diagram and contours for the ash content of the clean coal. It shows that an airflow of about 9 liters/minute will provide a low (~ 1.5 percent) ash product irrespective of percent solids in the slurry.

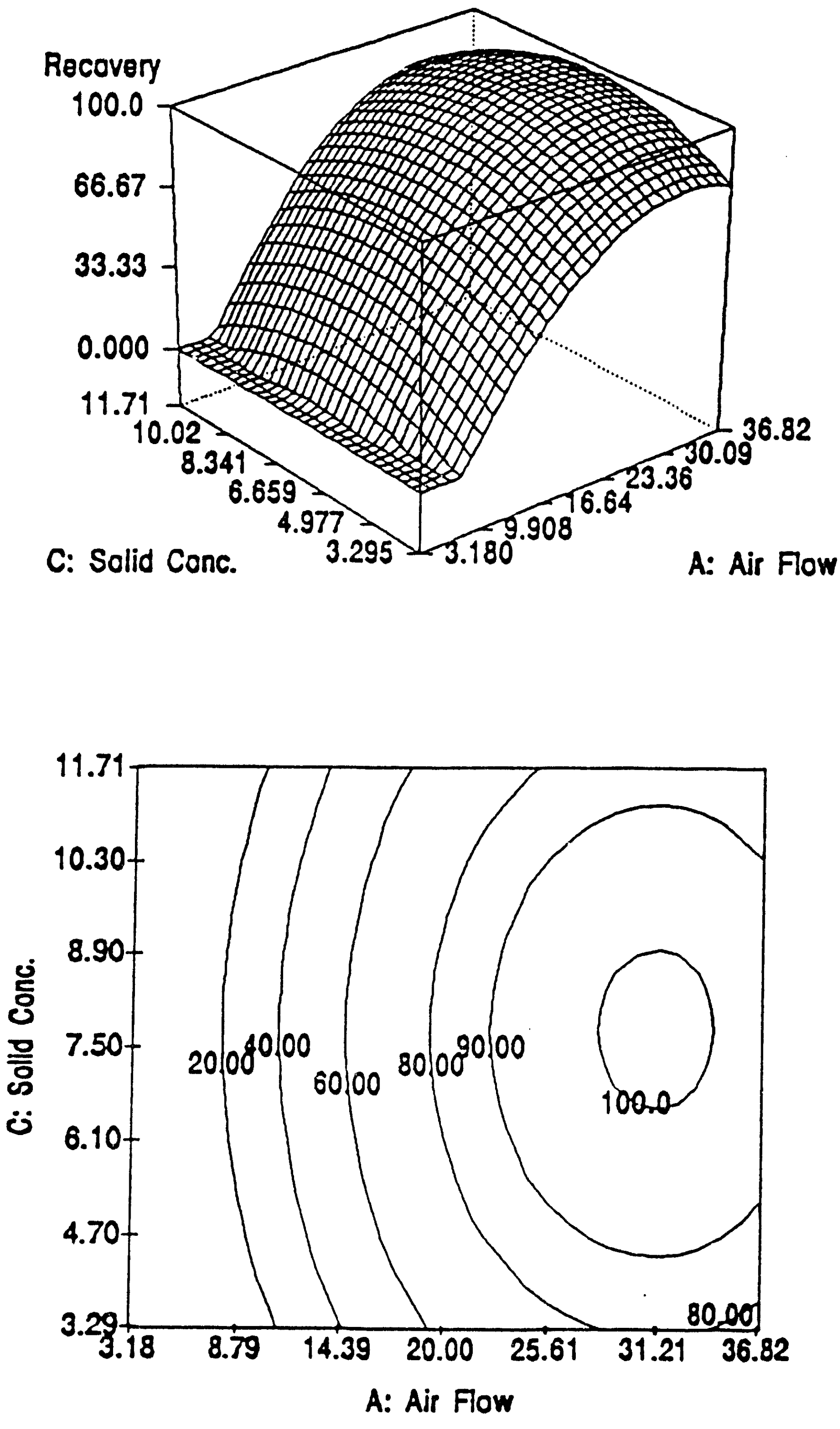



Figure 10. Response surface block diagram and contours for solids concentration and airflow for HHV recovery for Winifrede coal at 8 minutes retention time.

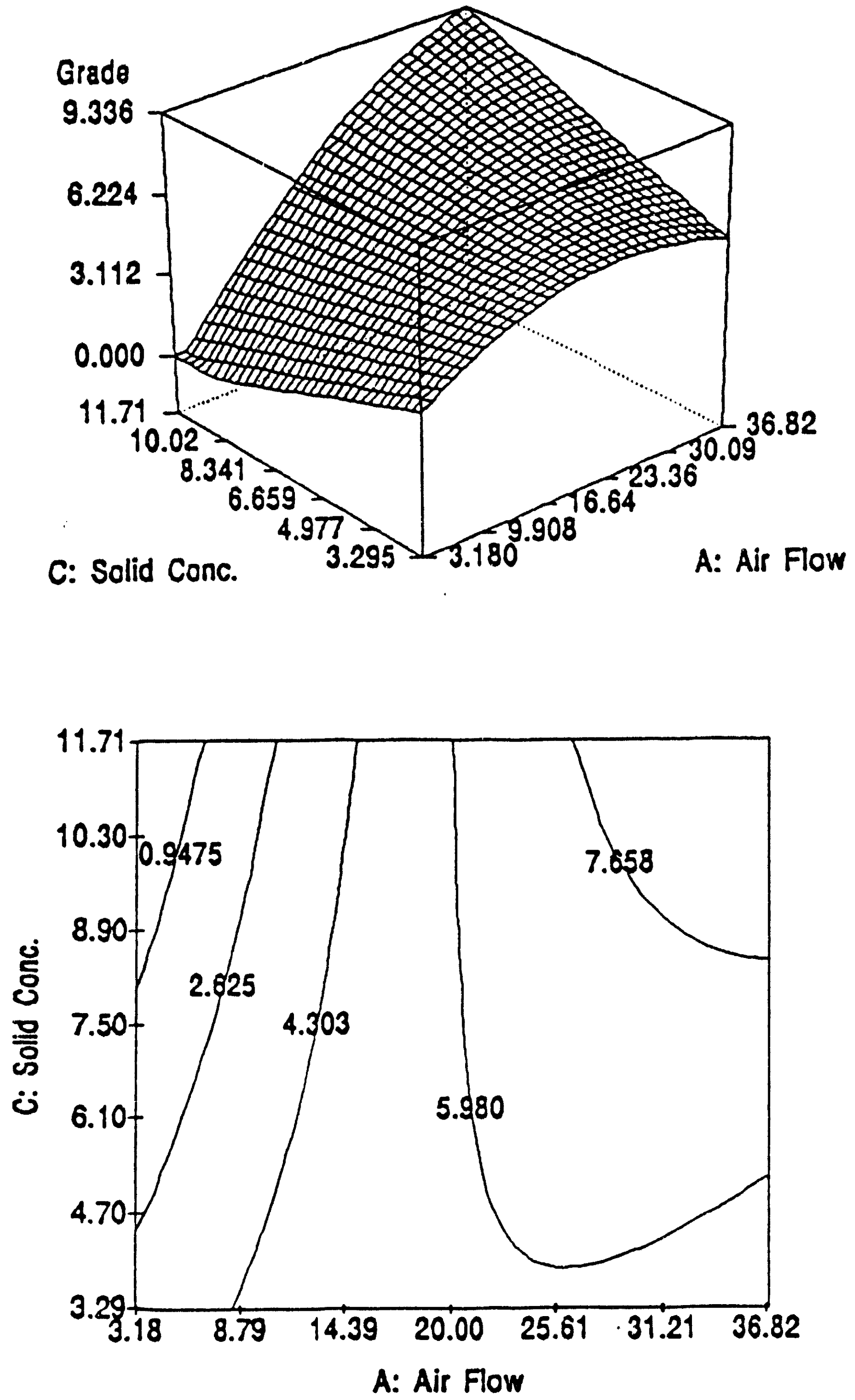



Figure 11. Response surface block diagram and contour for solids concentration and airflow for grade (ash content) for Winifrede coal at 8 minutes retention time.

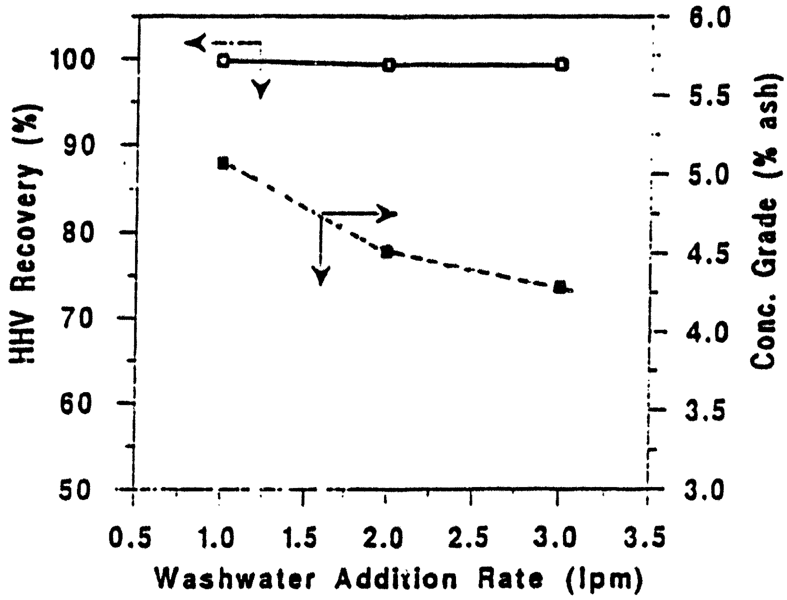



Figure 12. Effect of wash water rate on HHV recovery and percent ash in clean coal for Winifrede coal using the packed column (36.8 liters/minute airflow, 11.3 minutes retention time, 3.3% feed solids, 3.95 Kg/hr feed rate).

Table 9. Packed Column Flotation for Sunnyside Coal (Wash Water ~ 1 Liter/Minute)

| Airflow,[a] Liters/Minute | Retention Time, Minutes | Solids Concentration, Weight % | Feed Rate, Kg/hr | Ash, % | HHV Recovery, % |
|---|---|---|---|---|---|
| 10 | 6 | 5 | 22.3 | 1.46 | 36.5 |
| 30 | 6 | 5 | 22.3 | 3.62 | 98.3 |
| 10 | 10 | 5 | 7.4 | 1.23 | 50.2 |
| 30 | 10 | 5 | 7.4 | 4.53 | 99.9 |
| 10 | 6 | 10 | 44.6 | 1.10 | 9.4 |
| 30 | 6 | 10 | 44.6 | 4.05 | 71.1 |
| 10 | 10 | 10 | 14.7 | 1.25 | 30.3 |
| 30 | 10 | 10 | 17.1 | 4.93 | 99.6 |
| 20 | 8 | 7.5 | 17.1 | 3.53 | 76.3 |
| 20 | 8 | 7.5 | 17.1 | 3.53 | 79.0 |
| 20 | 8 | 7.5 | 17.1 | 3.55 | 79.2 |
| 20 | 8 | 7.5 | 17.1 | 3.55 | 79.5 |
| 3.2 | 8 | 7.5 | 17.1 | 1.23 | 35.2 |
| 36.8 | 8 | 7.5 | 17.1 | 4.06 | 98.7 |
| 20 | 4.6 | 7.5 | 29.1 | 3.38 | 50.1 |
| 20 | 11.4 | 7.5 | 9.2 | 3.57 | 98.3 |
| 20 | 8 | 3.3 | 6.5 | 3.29 | 98.0 |
| 20 | 8 | 11.7 | 23.3 | 3.99 | 60.0 |
| 20 | 8 | 7.5 | 17.1 | 3.49 | 88.2 |
| 20 | 8 | 7.5 | 17.1 | 3.38 | 86.2 |

[a] Air pressure maintained at 20 psi.

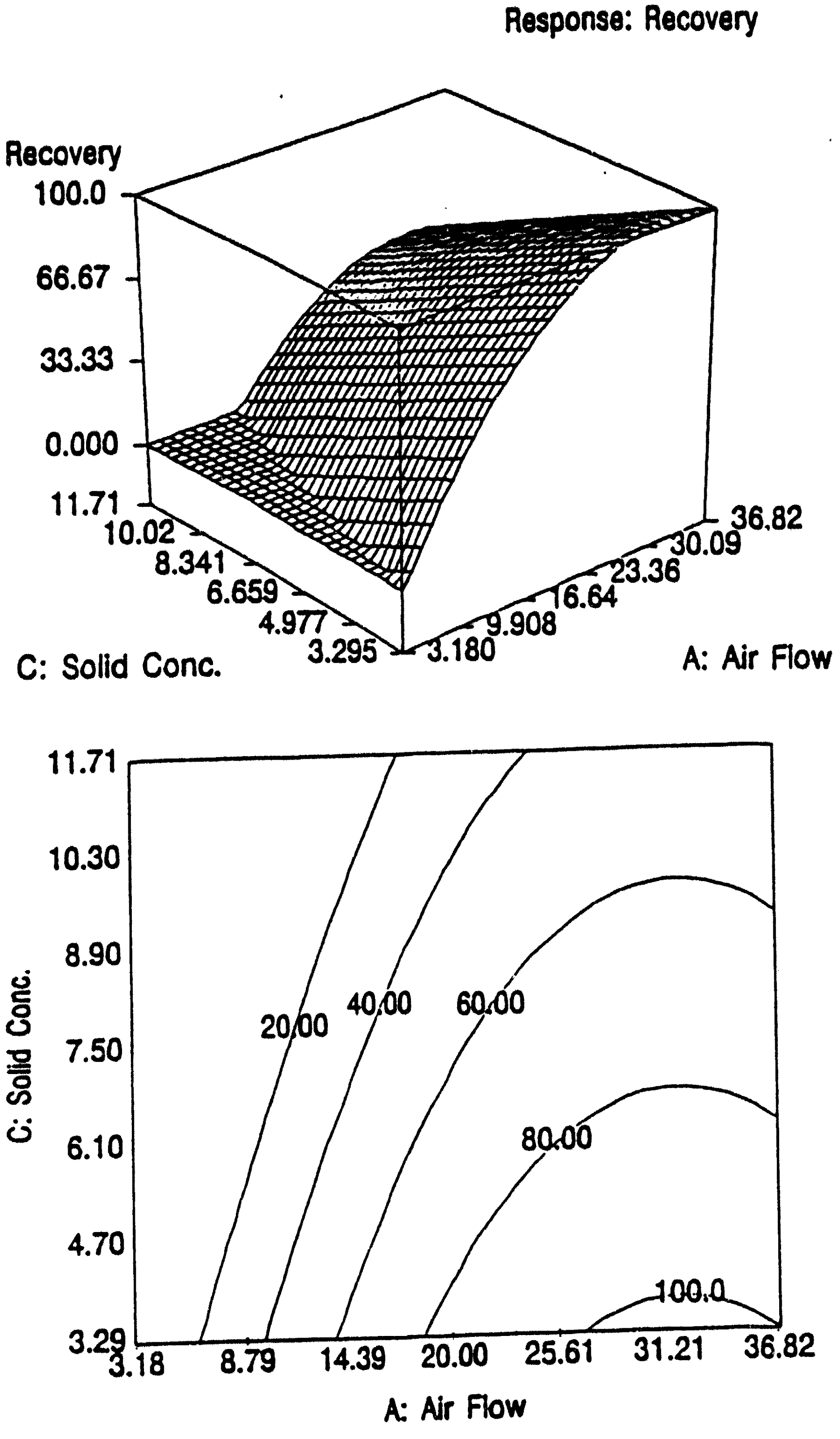



Figure 13. Response surface block diagram and contour for solids concentration and airflow for HHV recovery for Sunnyside coal at 5 minutes retention time.

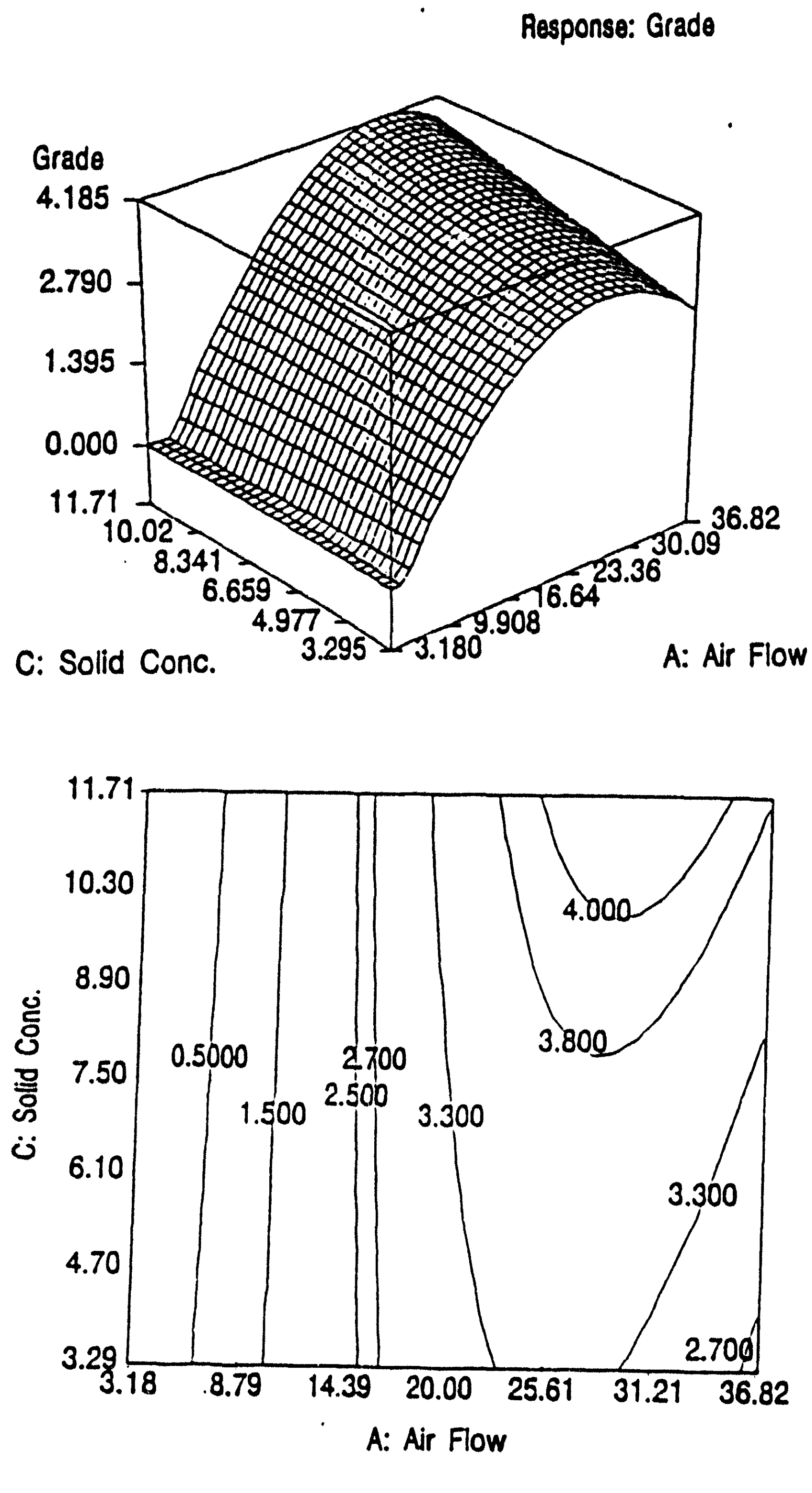



Figure 14. Response surface block diagram and contour for solids concentration and airflow for grade (ash content) of clean coal for Sunnyside coal at 5 minutes retention time.

Based on statistical analysis of all data, the following optimum conditions for the packed column were identified for Sunnyside coal:

| | |
|---|---|
| Airflow, Liters/Minute | 36.8 |
| Retention Time, Minutes | 4.9 |
| Solids Concentration, Weight % | 3.3 |

Using these optimum conditions, it is projected that a clean coal containing 2.58 percent ash (770 g/GJ or 1.8 lb/mmBtu) at near 100 percent HHV recovery could be obtained.

The effect of increasing wash water rates using the optimum operating conditions of the packed column on HHV recovery and percent ash in clean coal is shown in Figure 15. Note that increasing wash water flow rates from 1 to 3 liters/minute had a minimum effect on HHV recovery and ash content of the clean coal. The two responses remained constant at ~ 99 and 3.75 percent (2,130 g/GJ or 2.6 lb/mmBtu), respectively.

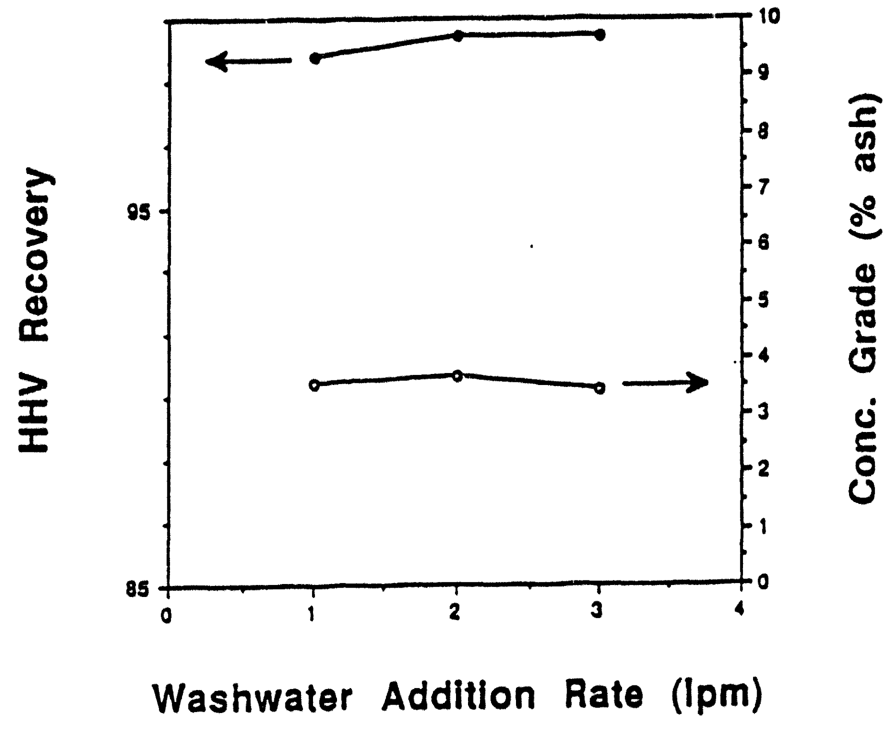



Figure 15. Effect of wash water rate of HHV recovery and percent ash in clean coal for Sunnyside coal using the packed column (36.8 liters/minute airflow, 4.9 minutes retention time, 3.3% feed solids, and 7.67 Kg/hr feed rate.

**Status of Packed Column Testing**

The experimental data show that when the packed column is operated under comparable conditions as operation of the "Ken-Flote" column, it was not effective in providing a low ash clean coal from the Winifrede or Sunnyside coals. Surprisingly, the ash content of both the clean coals was significantly higher for the packed column compared to the conventional "Ken-Flote" column. The following is a comparison:

| | Conventional | | Packed | |
|---|---|---|---|---|
| | Ash, % | HHV Recovery, % | Ash, % | HHV Recovery, % |
| Winifrede | 3.5 | 85 | 4.25 | >90 |
| Sunnyside | 2.1 | 90 | 3.75 | 99 |

It should be pointed out that the developer of the packed column, Dr. David Yang, prefers to operate the column with a deeper froth column than used in the present study. There is a possibility that a deeper froth may provide a lower ash clean coal product than indicated by the work completed so far.

Packed column tests with Elkhorn No. 3 coal are planned. Time permitting, additional tests will be conducted using the packed column to identify the effect of frother type and froth height as well as combining the "Ken-Flote" bubble generating system with the packed column.

## Subtask 4.3. CWF Formulation Studies

The objective of Subtask 4.3 is to define the process steps necessary to prepare a premium grade coal-water slurry fuel (CWF) from column flotation product. Process steps include:

- Grinding requirements to achieve an optimum particle size distribution.
- Reagent additions to achieve the desired slurry performance characteristics.
- Mechanical agitation techniques to reslurry dewatered froth from the column flotation cell.

As described in the test plan[9] issued on October 21, 1993, test work will focus on the flotation products produced during Subtasks 4.2 and 4.4 using the coals selected during Task 2. If necessary, some testing may be performed on the feed coals in order to determine the effects of flotation reagents and mineral matter changes on coal-water slurry properties.

## Taggart Seam Coal-Slurry Fuels

Initial tests were performed using the Taggart seam coal froth product, produced in Subtask 4.2. Approximately 50 gallons of flotation froth product was used for preparing coal-water slurry. The coal had been ground to nominally passing 62 mesh and floated in a laboratory column with MIBC frother and No. 2 diesel fuel collector. Both the collector and frother were added at a rate of 0.5 to 1.0 pound per ton of coal. The froth was collected and stored in a drum for use as feedstock when preparing slurries. Slurry from the drum was filtered and air dried as needed prior to formulating slurry fuel.

Two slurries were prepared for initial evaluation of the flotation product as a slurry fuel feedstock. An "as-produced" slurry was prepared from the flotation product by repulping the filter cake with dispersant and additional water. A "reground" slurry was also prepared from filter cake which was ground in a ball mill for 30 minutes.

The as-produced slurry was prepared from cake with the addition of 2 percent A23 dispersant at about 64 weight percent coal. The slurry was blended with the aid of a laboratory mixer. After the slurry was well blended, it was rolled in a plastic bottle to aid the removal of air bubbles. Then slurry properties were examined.

The reground slurry was prepared in a rubber-lined ball mill with a 7-1/2 inch inside diameter by 9-inch length. The mill has a ball charge which fills approximately 45 percent of the volume of the mill. The ball charge consists of the following media:

| | |
|---|---|
| 1-1/2-Inch Balls | 50% |
| 1-Inch Balls | 30% |
| 3/4-Inch Balls | 15% |
| 1/2-Inch Balls | 5% |

Approximately 1,444 grams of coal plus water and reagent was added to the mill for grinding. This represents a 1:1 material:void ratio, assuming the void space in the ball charge is 41 percent. The mill was operated for 30 minutes at a speed of 60 rpm which is 62 percent of critical speed. The resulting slurry was 62.6 percent coal and contained 2 percent A23 dispersant.

Table 10 compares the characteristics of the two slurry fuels which were prepared from Taggart seam coal. Neither slurry was stable and both were slightly rheopectic. This is undesirable as the slurry will settle easily and responds to increasing shear rates by an apparent increase in viscosity. This can be improved by optimizing the particle size distribution. The fine fraction of the particle size distribution must be increased in order to closer approximate the distribution Funk identified as optimum. The formula and resulting estimated size distribution for different values of "n" are shown in Table 11.

Table 10. Taggart Column Flotation Concentrate Slurry Properties

| | Cleaned Taggart | Cleaned Taggart |
|---|---|---|
| Mill Charge | | |
| Test | Taggart/Repulped | Taggart/Regrind |
| Grind Time, Minutes | 0 | 30 |
| Mill Product | | |
| Weight % Solids | 60.1 | 59.9 |
| Weight % A23 | 2.0 | 2.0 |
| Weight % Coal | 58.9 | 58.8 |
| Cumulative % Passing Size, $\mu m$ | | |
| 300 | 100.0 | 100.0 |
| 150 | 92.7 | 97.6 |
| 75 | 65.8 | 78.2 |
| 38 | 48.2 | 59.1 |
| 30 | 41.7 | 53.3 |
| 20 | 31.5 | 41.8 |
| 15 | 25.5 | 35.8 |
| 10 | 18.6 | 28.6 |
| 8 | 15.9 | 24.8 |
| 6 | 12.0 | 20.1 |
| 4 | 7.8 | 14.4 |
| 3 | 5.4 | 10.6 |
| 2 | 2.7 | 6.4 |
| 1 | 0.2 | 1.1 |
| MMD, $\mu m$ | 64.2 | 46.1 |
| Fann Viscosity, cp | | |
| 100/Second, Up | 600 | 800 |
| 100/Second, Down | 225 | 260 |
| 1,000/Second, Up | 425 | 635 |
| 1,000/Second, Down | 310 | 340 |
| Viscosity Notes | Very Unstable, Hardpack Sediment | Unstable, Hardpack |

Table 11. Optimized Size Distribution for 300 x 0.5 Micron Size Range

"Funk" Formula for Optimum Particle Packing:

$$CPFT = \frac{\left(D_u^n - D_s^n\right)}{\left(D_l^n - D_s^n\right)} \times 100$$

where: CPFT = Cumulative Percent Finer Than $D_u$
$D_u$ = Diameter of Particle ($\mu m$)
$D_l$ = Diameter of Largest Particle ($\mu m$)
$D_s$ = Diameter of Smallest Particle ($\mu m$)
n = Numerical Exponent (Between 0.1 and 1.0)

= ln v/ln k = 0.17

where: v = Volume Fraction of Liquid in Slurry = 0.34 (Typical for <300 $\mu m$)
k = $D_s/D_l$ = 0.001667

Table of Values of CPFT as a Function of Exponent "n"

Dl = 300 $\mu m$
Ds = 0.5 $\mu m$

| Screen Size | Diameter, $\mu m$ | Exponent "n" 0.10 | 0.15 | 0.20 | 0.40 |
|---|---|---|---|---|---|
| 50M | 300 | 100.0 | 100.0 | 100.0 | 100.0 |
| 100M | 150 | 85.8 | 84.0 | 82.1 | 73.8 |
| 200M | 74 | 72.4 | 69.3 | 66.2 | 53.5 |
| 400M | 37 | 60.0 | 56.3 | 52.6 | 38.5 |
| | 30 | 56.5 | 52.7 | 48.9 | 34.8 |
| | 20 | 49.8 | 45.9 | 42.1 | 28.3 |
| | 15 | 45.2 | 41.3 | 37.6 | 24.3 |
| | 10 | 39.0 | 35.2 | 31.6 | 19.4 |
| | 8 | 35.7 | 32.0 | 28.6 | 17.0 |
| | 6 | 31.5 | 28.0 | 24.8 | 14.3 |
| | 4 | 25.8 | 22.7 | 19.9 | 10.9 |
| | 3 | 21.9 | 19.1 | 16.6 | 8.8 |
| | 2 | 16.6 | 14.4 | 12.3 | 6.2 |
| | 1 | 8.0 | 6.8 | 5.7 | 2.7 |
| MMD, $\mu m$ | | 60.3 | 65.8 | 71.4 | 93.6 |

A comparison of the particle size distribution for both slurries is plotted in Figure 16 against the range of optimum distributions for "n" between 0.1 to 0.4. The plots show a definite deficiency in the amount of fine particles. Future development work will focus on increasing the fine fraction of coal particles in the slurry fuels by grinding a portion of the cake in a stirred ball mill.

## Subtask 4.4. Bench-Scale Testing and Process Scale-up

Subtask 4.4 is for verification of the key process optimization findings and scale-up relationships from Subtasks 4.1, 4.2, and 4.3. A bench-scale column flotation system capable of producing about 45 kg per hour of clean coal will be used for the verification, and clean coal will be prepared from each of the coals during the testing.

A Ken-Flote 0.3-m diameter by 4.6-m (1- x 15-foot) bench-scale column flotation system that had previously been used by Process Technology, Inc. for the process control emerging technology project at PETC (Contract No. DE-AC22-92 PC 92207) was installed in the Amax R&D pilot plant area for the bench-scale testing. The nominal capacity of the unit is in the range of 50 to 100 kg of dry coal per hour (100 to 200 lb/hr). As installed, the column includes the level and flow control system that was part of the operation at PETC. The ash-analyzer portion of the control system will not be used at this time, and the analyzer equipment was placed into storage.

Process Technology, Inc. assisted with setting up the column, calibrating the instrumentation, and verifying the process control programming. Shakedown runs with coal slurry were completed successfully at month-end. The fuzzy logic control system for regulating slurry levels and flows performed well.

The column was equipped with a porous metal air sparger when received, and it was used during the shakedown testing. A Foam-Jet sparger was ordered to replace the porous metal sparger. In the meantime, the Foam-Jet sparger was borrowed from CAER to use when beginning actual test work. The interior parts of the progressive cavity slurry pumps feeding and discharging the column appeared to be worn, so replacements were also ordered so that the repairs may be made before complete failure of either pump occurs.

Elkhorn No. 3 coal was used during the shakedown and is the first coal scheduled for the Subtask 4.4 parametric testing to begin in January. The slurry will be prepared by closed circuit grinding in the 1.2m ball mill. A complete test plan is being prepared for submittal to the DOE.

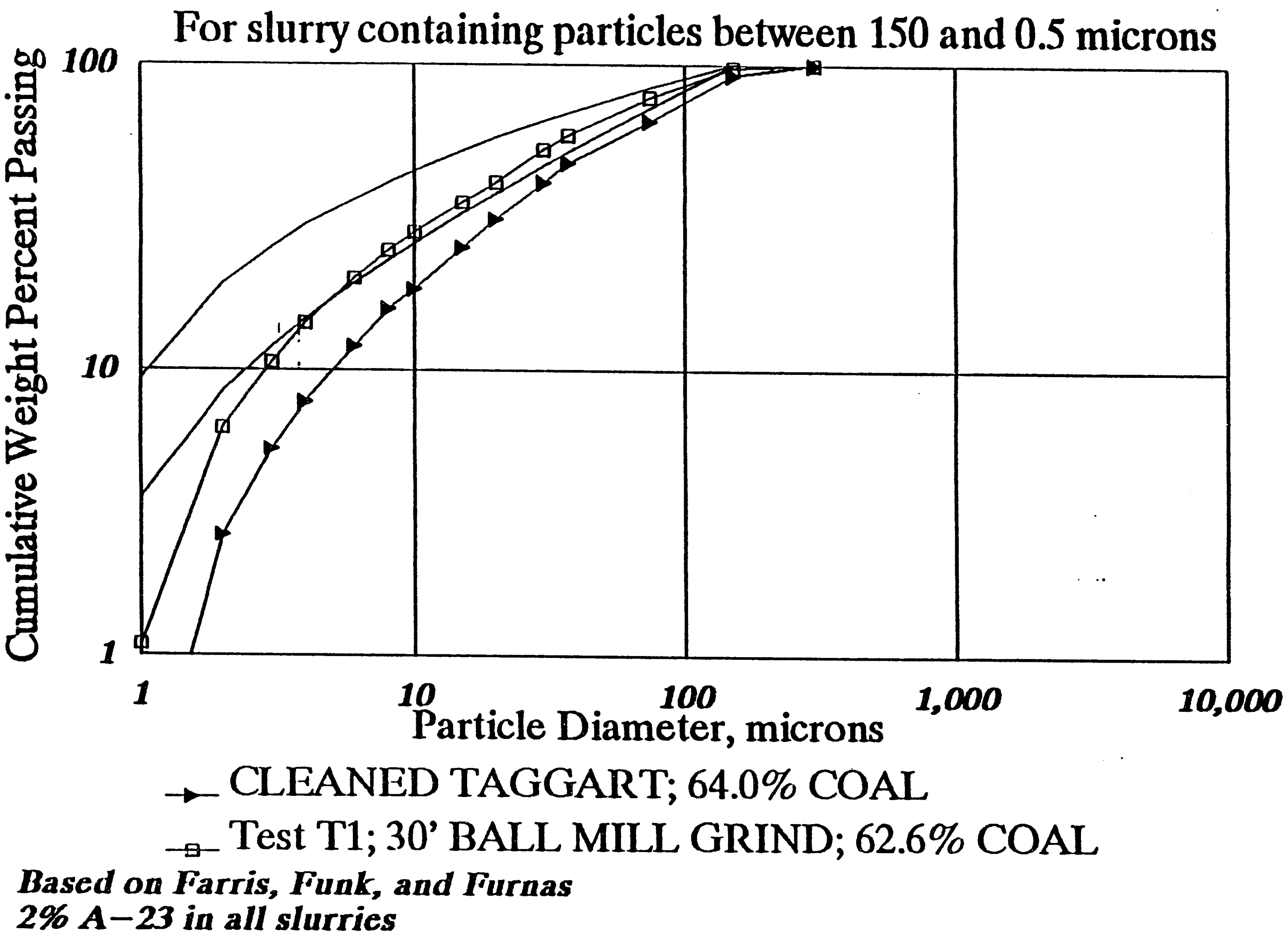



Figure 16. PSD comparisons of as-produced and reground Taggart flotation coals with optimized CWF distributions.

## Subtask 4.5. Conceptual Design of the PDU and Advanced Froth Flotation Module

The topical report for Subtask 4.5 written by Bechtel and entitled "Conceptual Engineering Package" was issued on December 10, 1993.[10] The concept is for the production of 1.8 tph (2.0 stph on a dry basis) of clean coal by column flotation in a PDU to be located at Amax R&D in Golden, Colorado. The unit will be designed for continuous production of ultra-clean coal meeting project specifications and to demonstrate process scale-up when cleaning three of the test coals. The PDU will also be capable of demonstrating near-term applications, including the drying and briquetting of coal to marketable forms. As such, the subtask conceptual design shows five work areas:

- Plant 100 Selective Grinding
- Plant 200 Column Flotation
- Plant 400 Product Dewatering
- Plant 500 Product Treatment
- Plant 600 Utilities

Plant 300 is reserved for selective Agglomeration Module. The PDU with the advanced flotation module will be housed in a existing pilot plant buildings at Amax R&D. The selective agglomeration module will be added later.

The conceptual design package presented in the topical report includes the following:

- Plant Design Basis

- Conceptual Engineering Design, which includes:

  - Process and Plant Description
  - Process Flow Diagrams and Material Balances
  - Estimates of Utility Requirements
  - Instrumentation and Control Philosophy
  - Preliminary P&IDs
  - Preliminary Layout and Equipment Arrangement Drawings
  - Preliminary Electrical Single Line Diagram

- Preliminary Quotations from Vendors for Major Equipment

- Conceptual Cost Estimate

- Long Lead Major Equipment

## TASK 5. DETAILED ENGINEERING DESIGN OF THE PDU AND ADVANCED FLOTATION MODULE

Bechtel has started planning activities for this task. The PDU will consist of the following six plants:

| | |
|---|---|
| Plant 100 | Selective Grinding |
| Plant 200 | Column Flotation |
| Plant 300 | Selective Agglomeration (To Be Designed Later) |
| Plant 400 | Product Dewatering |
| Plant 500 | Product Treatment |
| Plant 600 | Utilities |

It was decided to start the detailed design work with Plant 400, which will consist of filtering and dewatering of the clean coal as well as the tailings. This decision was based on the following considerations:

1. The plant will utilize two Shriver plate and frame filter presses already in place at Amax R&D Center as well as two Netzsch plate and frame filter presses transferred from the Wilsonville selective agglomeration POC plant. While the filtering characteristics will vary somewhat with the coal type and its particle size distribution, the same machines can be used for filtering and dewatering with surge capacity (thickener or storage tank) in between the cleaning and filtering steps.

   Since the equipment is already in possession of Amax R&D, all necessary equipment specifications and data can be made available to Bechtel. The building location is already defined. Thus, Bechtel can start detailed design work for refurbishment and installation of these filters and associated sumps, pumps, piping, and instruments.

2. The research and testing work has been completed on liberation characteristics and grinding power requirements for the six candidate coals. There is a vast difference in the grinding capacity requirement to produce 2 ton/hour clean coal from Sunnyside or Elkhorn No. 3 coal than from Winifrede or Indiana No. 7 coal. A decision has to be made as to which coals have to be ground at what capacity before Bechtel can perform the detailed design of Plant 100. This decision had to be postponed until the first quarter of 1994, in view of the uncertainties created by the Cyprus Amax merger and lab closure.

3. Laboratory research for process optimization of column flotation is nearing completion. It appears that both the Ken-Flote and Microcel type column designs are capable of meeting the product quality and recovery specifications from Elkhorn No. 3, Taggart, Sunnyside, and Indiana No. 7 coals, but both will

have difficulty in meeting targets with Winifrede coals. The performance of the packed column was inferior. In view of the above, bench-scale testing has been started with the Ken-Flote type column at Amax R&D Center (see Subtask 4.4), and it is planned to test the Microcel type column on bench-scale as well. These results will not be available until the end of the first quarter of 1994 and, therefore, the detailed design of Plant 200 has to be delayed to the second quarter of 1994.

## TASK 6. SELECTIVE AGGLOMERATION LABORATORY RESEARCH AND ENGINEERING DEVELOPMENT FOR PREMIUM FUELS

Task 6 activities during the quarter included completion of the scheduled bench-scale grinding tests and initial process optimization. Bechtel also began some conceptual engineering studies for design of the PDU.

### Subtask 6.2. Grinding

The testing portions of the grinding studies for preparation of the feed for selective agglomeration were completed during the quarter. For the most part, the Subtask 6.2 studies drew upon the same laboratory and pilot-plant testing as the Subtask 4.1 grinding studies for preparation of flotation feed slurry. Preparation of the Subtask 6.2 topical report is scheduled to begin shortly after the Subtask 4.1 topical report is completed.

The basic conclusions of the Subtask 6.2 work will be that the grinding requirements ahead of selective agglomeration are similar to the grinding requirements ahead of advanced froth flotation.

The only extensive testing done specifically for Subtask 6.2 was continuous ball mill and stirred ball mill grinding tests on subbituminous Dietz coal. These were all open-circuit grinding tests, and Table 12 records the performance of each mill. The results are also plotted in Figures 17 and 18 for comparison with similar grinding tests on the bituminous test coals. The capacities of the mills were between 40 and 50 percent less when grinding Dietz coal than when grinding the other coals. The observed difference in grinding performance is consistent with the lower grindability index (HGI = 41) of the Dietz coal. Water requirements are also a major difference between grinding subbituminous coal such as Dietz and bituminous coals. A significant amount of water soaks into the structure of subbituminous coal, so extra water must be added to the slurry in order to provide sufficient fluidity for flow through the mills and pipelines.

Agglomeration liberation tests were performed on the stirred ball mill product slurries from the continuous grinding tests. (The acidification procedure was followed as described last quarter.[4]) The ash contents of the agglomerates are plotted versus the D80 grind sizes in Figure 19. According to this plot, Dietz coal needs to be ground to D80 = 20 $\mu$m in order to meet a <860 g/GJ ash specification. This is the same grind size recommended for Indiana VII coal.

## Table 12. Parametric Grinding Tests on Dietz Coal (HGI = 41) in 1.2-m Ball Mill and 40-Liter Stirred Ball Mill

| | Feed | | | | Product | | |
|---|---|---|---|---|---|---|---|
| Test | HGI | Dry Coal, kg/hr | % Solids | D80 µm | D98 µm | D80 µm | D50 µm |
| **Ball Mill Grinding** | | | | | | | |
| B15-1 | 41 | 196 | 35.7 | 5,267 | 447 | 149 | 52 |
| B15-2 | 41 | 388 | 32.9 | 5,267 | 541 | 213 | 88 |
| **Stirred Ball Mill Grinding** | | | | | | | |
| S15-1 | 41 | 468 | 33.2 | 149 | 103 | 41 | 16.9 |
| S15-2 | 41 | 224 | 33.1 | 149 | 63 | 27 | 11.2 |
| S15-3 | 41 | 141 | 33.0 | 149 | 49 | 20 | 8.9 |

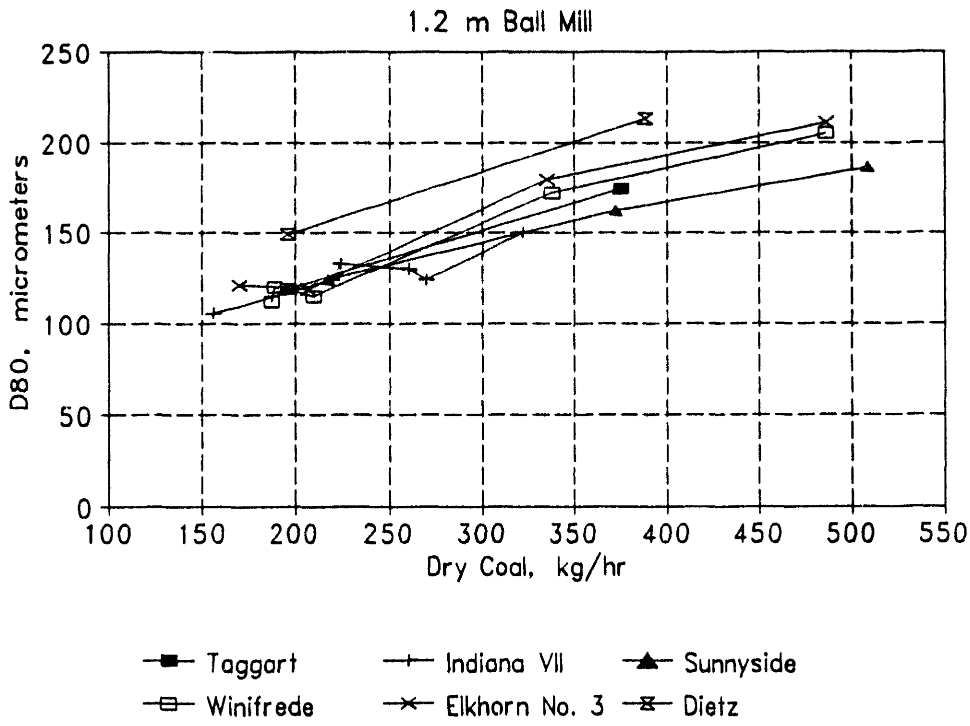



Figure 17. Ball mill grinding rate versus 80 percent passing size (D80) for six test coals.

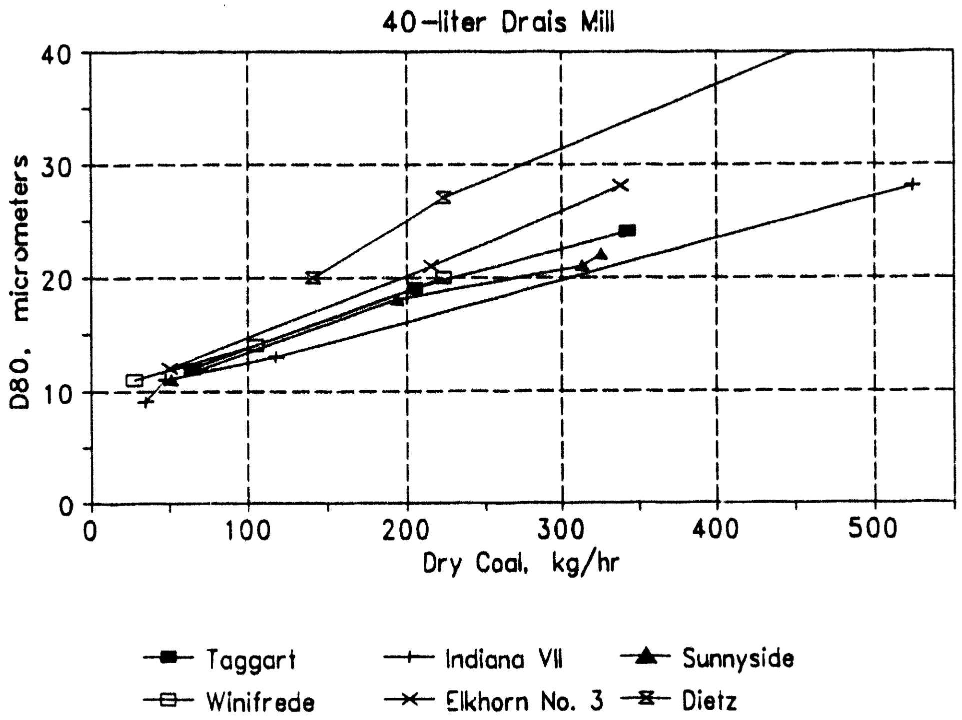



Figure 18. Stirred ball mill grinding rate versus 80 percent passing size (D80) for six test coals.

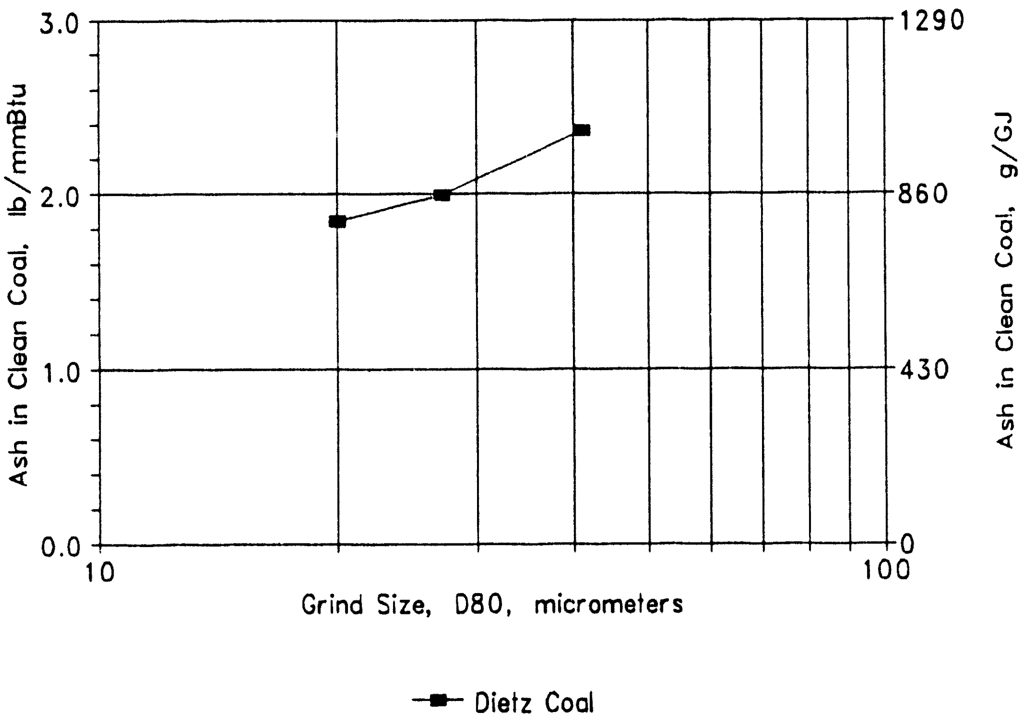



Figure 19. Ash liberation versus 80 percent passing size (D80) for Dietz coal ground in the 40-liter stirred ball mill.

## Subtask 6.3. Process Optimization Research

The emphasis of the process optimization research during the quarter was on the selective agglomeration of subbituminous Dietz coal. This work is being done at Arcanum on slurry ground in their laboratory.

Heptane bridging liquid containing varying amounts of asphalt has been used for this work. As described last quarter, acidification of the slurry was necessary for the inversion reaction to occur. Arcanum has since examined the use of chelating agents as well as acids, specifically EDTA (ethylene diamine tetraacetic acid) and sodium metaphosphate. Table 13 lists the tests performed with these reagents. The pH reduction power of acetic acid and EDTA is shown in Figure 20. The following conclusions were drawn from this work:

- In the presence of an asphalt binder, pH reduction to 5.0 - 5.5 activates the Dietz coal sufficiently to achieve reasonably high shear agglomeration times. Ethylhexanol was not effective for activating agglomeration at these pH reductions. These pH levels are moderate enough that corrosion would be less severe in a production plant than when operating at the highly acidic levels indicated previously.

- pH reduction alone seems to be sufficient to achieve the improved activation noted during testing. Inversion times are seen to correlate strongly with pH. EDTA seems to be effective at smaller pH changes. This may be due to the chelating power of EDTA. While acetic acid and EDTA give roughly the same pH reduction per equivalent of reagent used per gram of coal, acetic acid is nearly three times as efficient on a weight basis due to its much lower molecular weight per equivalent.

- Sodium metaphosphate was not effective for activating agglomeration either alone or in conjunction with EDTA additions.

- Finer grinding seemed to reduce the efficiency of the separation.

Selected test conditions were being repeated with pentane bridging liquid to provide the first points in a heptane/pentane comparison matrix. Further optimization of the EDTA/acetic acid systems will be conducted during continuous testing in a laboratory scale apparatus.

## Subtask 6.6. Conceptual Design of the Selective Agglomeration Module

During the quarter, a preliminary planning meeting was held at Bechtel. Focus of initial activities was on evaluation of relative costs and merits of two potential designs. The first will be similar to the design developed and tested by Arcanum and Bechtel at Homer City using heptane as the agglomerating agent in a series of high and low shear stirred tank reactors. The second approach will use a

## Table 13. Low-Rank Coal Results

### Agglomeration of Ball Milled Dietz Coal with Heptane Bridging Liquid and Asphalt Binder

| Test No. | Bridging Liquid Dose, % | Asphalt, % | Additive | Dose, % | pH | Inversion Time, Seconds | D80, μm | Feed Ash, % | Product Ash, % | Tails Ash, % |
|---|---|---|---|---|---|---|---|---|---|---|
| BA-281-0 | 53.00 | 1.67 | EDTA | 5.00 | 5.60 | | | 4.50 | | |
| BA-286-0 | 33.00 | 1.70 | EDTA | 5.50 | 5.60 | 150 | 36 | 4.50 | 2.73 | |
| BA-298-0 | 39.00 | 2.00 | EDTA | 2.50 | 6.10 | | | 4.48 | 2.82 | |
| BA-300-0 | 43.00 | 2.50 | EDTA | 2.50 | 6.10 | 200 | | 4.48 | 2.90 | |
| BA-300-0 | 42.00 | 2.00 | EDTA | 5.00 | 5.60 | 135 | | 4.48 | 2.74 | |
| BA-305-0 | 42.00 | 2.00 | EDTA | 0.78 | 6.80 | | 22 | 4.56 | 2.94 | 81.88 |
| BA-305-0 | 42.00 | 2.00 | EDTA | 0.78 | 6.80 | 330 | 22 | 4.56 | 2.98 | 86.89 |
| BA-314-0 | 40.00 | 2.00 | EDTA | 2.00 | 6.20 | 200 | 36 | 4.50 | 2.77 | 87.60 |
| BA-314-0 | 40.00 | 2.67 | Na(Meta)PO4 | 5.00 | 6.90 | 600 | 36 | 4.50 | 3.15 | 88.92 |
| BA-316-0 | 44.00 | 3.00 | EDTA | 1.58 | 6.65 | 540 | 31 | 4.56 | 2.85 | 80.46 |
| BA-319-0 | 34.00 | 1.90 | Na(Meta)PO4 | 1.75 | 6.90 | 250 | 50 | 6.25 | 3.52 | 87.63 |
| BA-319-0 | 45.00 | 3.00 | EDTA | 1.58 | 6.63 | 195 | 31 | 4.56 | 2.79 | 82.57 |
| BA-321-0 | 40.00 | 2.00 | EDTA | 1.00 | 6.70 | 205 | 50 | 6.25 | 3.34 | 89.55 |
| BA-321-0 | 40.00 | 2.00 | EDTA | 2.00 | 6.45 | 160 | 50 | 6.25 | 3.02 | 91.45 |
| BA-323-0 | 40.00 | 1.75 | Acetic Acid | 1.64 | 5.60 | 195 | 32 | 4.72 | 2.90 | 92.92 |
| BA-323-0 | 40.00 | 2.00 | Acetic Acid | 2.50 | 5.00 | 150 | 32 | 4.72 | 2.90 | 90.17 |
| BA-326-0 | 40.00 | 0.00 | Acetic Acid | 2.50 | 5.00 | | 32 | 4.72 | | |
| BA-333-0 | 40.00 | 1.50 | Acetic Acid | 2.50 | 5.00 | | 31 | 4.72 | 3.00 | 88.80 |
| BA-333-0 | 40.00 | 1.50 | Acetic Acid | 2.50 | 5.00 | 210 | 31 | 4.72 | 2.90 | 90.86 |

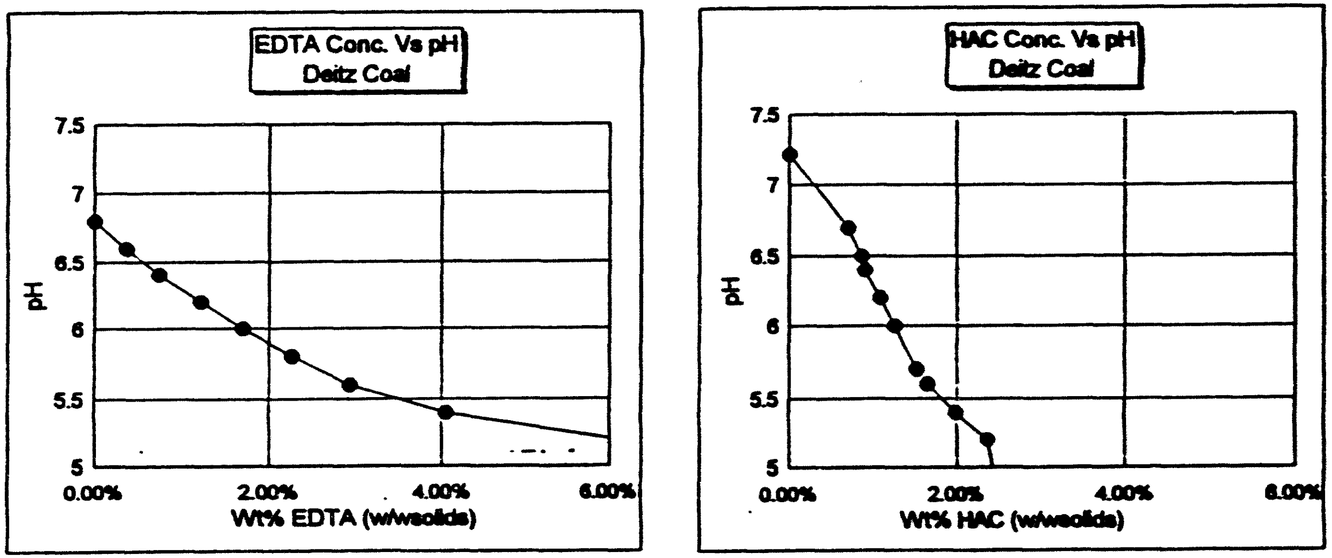



Figure 20. pH reduction power of acetic acid and EDTA.

single-stage novel design reactor proposed by Dr. Keller based on his past experience with the Otisca agglomeration system which used pentane as the agglomerating agent. The second system offers potential for cost savings but may need more development effort.

# PLANS FOR NEXT QUARTER

Task 4, advanced flotation activities, will receive concentrated attention during the January - March quarter. The topical report for the Subtask 4.1 grinding study and the test plan for Subtask 4.4 will be issued. Subtask 4.2, laboratory flotation optimization research, will be completed at CAER, VPI, and Amax R&D, and the findings will be organized into a draft topical report presenting the combined conclusions of the three research groups. The bench-scale flotation for Subtask 4.4 has begun in the 0.3-m column and will continue on into the following quarter. The initial effort will be on Elkhorn No. 3 coal, followed by Winifrede and Indiana VII coals. Production of 40 to 80 kg/hr quantities of clean coal with the 0.3-m column will allow Subtask 4.3, CWF formulation studies, to begin in earnest during the quarter.

Bechtel will continue the scheduled work on parts of Task 5, final design of the PDU, which will not be impacted by any unanticipated results of the Subtask 4.4 test work. The design for the water recirculation system, in particular, will receive attention during the quarter.

The topical report describing the Subtask 6.2 study for grinding coal prior to selective agglomeration will also be issued during the quarter. In the meantime, optimization of the selective agglomeration process for subbituminous coal will continue at Arcanum, and a 50-gram per minute continuous laboratory system will be assembled and placed into service at Amax R&D for testing bituminous coals. Bechtel will continue their conceptual design study for the Subtask 6.6 selective agglomeration module. The latter study will include a comparison of circuits designed for use with the pentane agglomerant with circuits designed for use with the heptane agglomerant.

## REFERENCES


1. Smit, F. J. and Jha, M. C., Quarterly Technical Progress Report 1, October - December, 1992, "Engineering Development of Advanced Physical Fine Coal Cleaning for Premium Fuel Applications", Amax R&D Report to the U. S. Department of Energy, January 18, 1993.

2. Smit, F. J. and Jha, M. C., Quarterly Technical Progress Report 2, January - March, 1993, "Engineering Development of Advanced Physical Fine Coal Cleaning for Premium Fuel Applications", Amax R&D Report to the U. S. Department of Energy, April 26, 1993.

3. Smit, F. J. and Jha, M. C., Quarterly Technical Progress Report 3, April - June, 1993, "Engineering Development of Advanced Physical Fine Coal Cleaning for Premium Fuel Applications", Amax R&D Report to the U. S. Department of Energy, July 28, 1993.

4. Smit, F. J., Hogsett, R. F., and Jha, M. C., Quarterly Technical Progress Report 4, July - September 1993, "Engineering Development of Advanced Physical Fine Coal Cleaning for Premium Fuel Applications," Amax R&D Report to the U. S. Department of Energy, November 4, 1993.

5. Smit, F. J. and Jha, M. C., "Engineering Development of Advanced Physical Fine Coal Cleaning for Premium Fuel Applications, Subtask 2.1 Coal Selection Plan and Recommendations", Amax R&D Report to the U. S. Department of Energy, April 29, 1993.

6. Bechtel Corporation, "Engineering Development of Advanced Physical Fine Coal Cleaning for Premium Fuel Applications, Task 3. Development of Near-Term Applications, Subtask 3.1 Engineering Analysis Conceptual Designs and Cost Estimates", Report to the U. S. Department of Energy, November 5, 1993.

7. Smit, F. J. and Jha, M. C., "Engineering Development of Advanced Physical Fine Coal Cleaning for Premium Fuel Applications, Subtask 4.2. Process Optimization Research for Advanced Froth Flotation Test Plan", Amax R&D Report to the U. S. Department of Energy, September 29, 1993.

8. Norusis, M. J., SPSS/PC+ for the IBM PC/XT/AT, Chicago, SPSS Inc., 1986.

9. Berggren, M. H., Smit, F. J., and Jha, M. C., "Engineering Development of Advanced Physical Fine Coal Cleaning for Premium Fuel Applications, Subtask 4.3 CWF Formulation Studies Test Plan", Amax R&D Report to the Department of Energy, October 21, 1993.

10. Bechtel Corporation, "Engineering Development of Advanced Physical Fine Coal Cleaning for Premium Fuel Applications, Subtask 4.5 Conceptual Design of Process Development Unit and Advanced Froth Flotation Module Conceptual Engineering Package", Report to the U. S. Department of Energy, December 10, 1993.

# APPENDIX A

# ACRONYMS/ABBREVIATIONS

## ACRONYMS/ABBREVIATIONS

| | |
|---|---|
| Amax R&D | Amax Research & Development Center |
| CAER | Center for Applied Energy Research, University of Kentucky |
| CWF | Coal Water (Slurry) Fuel |
| D50 | Particle size opening where 50 percent by weight of a distribution will pass. |
| D80 | Particle size opening where 80 percent by weight of a distribution will pass (often used for grinding calculations). |
| D98 | Nominal top size of a distribution. Particle size opening where 98 percent by weight of a distribution will pass. |
| DOE | United States Department of Energy |
| FY | Federal Fiscal Year |
| HHV | Higher Heating Value |
| HHVc | Calculated Higher Heating Value |
| MIBC | 2-Methyl Pentanol-4 (Methyl Isobutyl Carbinol) |
| PETC | Pittsburgh Energy Technology Center |
| PDU | Process Demonstration Unit |
| POC | Proof-of-Concept |
| PSD | Particle Size Distribution |
| Rec | Recovery |
| S(t) | Total Sulfur |
| S(py) | Pyritic Sulfur |
| S(pyc) | Calculated Pyritic Sulfur |
| Btu | British Thermal Unit (Unit of Energy) |
| g | Gram |
| GJ | Gigajoule (One Billion Joules) |
| HGI | Hardgrove Grindability Index |
| hp | Horsepower |
| hr | Hour |
| J | Joule (Unit of Energy) |
| kg | Kilogram |
| kW | Kilowatt (Unit of Power) |
| lb | Pound |
| lpm | Liters per Minute |
| m | Meter or, on occasion, Mesh |
| M | Mega, Million |
| mg | Milligram (One Thousandth Gram) |
| mm | Millimeter (One Thousandth Meter) |
| mmBtu | Million Btu |
| pH | Acidity/Alkalinity of an Aqueous Solution |
| st | Short Ton (2,000 Pounds) |
| t | Tonne, Metric Ton (1,000 kg) |
| VPI | Virginia Polytechnic Institute and State University |
| $\mu$m | Micrometer, Micron (One Millionth Meter) |
| $\mu$g | Microgram (One Millionth Gram) |
| 48 mesh | 300 $\mu$m |
| 100 mesh | 150 $\mu$m |
| 200 mesh | 200 $\mu$m |
| 325 mesh | 45 $\mu$m |

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