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QUARTERLY TECHNICAL PROGRESS REPORT 13
OCTOBER - DECEMBER, 1995

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

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U. S. Department of Energy
Pittsburgh Energy Technology Center
Pittsburgh, Pennsylvania 15236

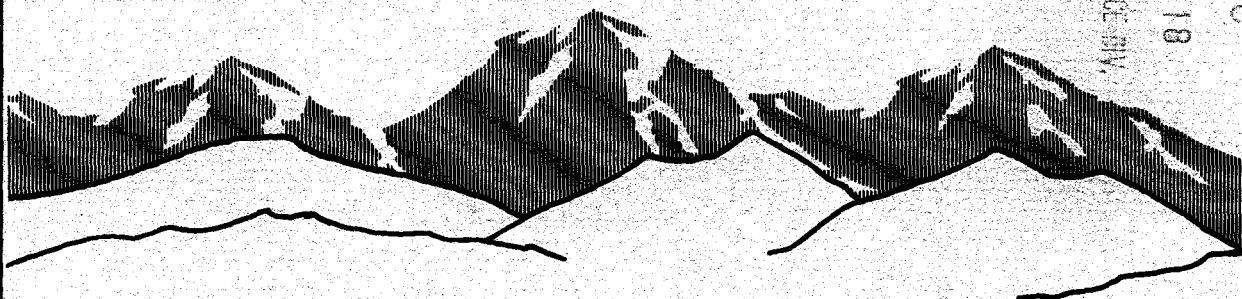
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ABSTRACT

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

During Quarter 13 (October - December 1995), testing of the GranuFlow dewatering process indicated a 3-4% reduction in cake moisture for screen-bowl and solid-bowl centrifuge products. The Orimulsion additions were also found to reduce the potential dustiness of the fine coal, as well as improve solids recovery in the screen-bowl centrifuge. Based on these results, Lady Dunn management now plans to use a screen bowl centrifuge to dewater their Microcel™ column froth product.

Subtask 3.3 testing, investigating a novel Hydrophobic Dewatering process (HD), continued this quarter. Work included contact angle measurements, direct force measurements, the calculation of the potential curve for the displacement of water on the surface of coal, and shakedown testing of the batch dewatering unit.

A draft of the Subtask 4.4 topical report, containing bench-scale flotation and the toxic trace element reduction data was distributed to project team members for comment.

Continuing Subtask 6.4 work, investigating coal-water-fuel slurry formulation, indicated that selective agglomeration products can be formulated into slurries with lower viscosities than advanced flotation products. Subtask 6.5 agglomeration bench-scale testing results indicate that a very fine grind is required to meet the 2 lb ash/MBtu product specification for the Winifrede coal, while the Hiawatha coal requires a grind in the 100- to 150-mesh topsize range. Results with the new Taggart coal show that a grind finer than 100-mesh is required to meet a 1 lb/MBtu product ash specification.

Work was essentially completed on the detailed design of the PDU selective agglomeration module under Task 7. Detailed design work remaining involves the preparation and issuing of the final task report. Utilizing this detailed design, a construction bid package was prepared and submitted to three Colorado based contractors for quotes as part of Task 9.

Material requisitions for the bulk of the capital equipment to be purchased for the construction of the selective agglomeration module were issued with the remaining to be issued in early January, 1996. It has also been determined that eight used equipment items, currently on-site, will be utilized during the construction of the selective agglomeration PDU module.

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, as well as for advanced combustors currently under development.

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

This multi-year cost-share contract started on October 1, 1992. This report discusses the progress made during the 13th quarter of the project from October 1 to December 31, 1995.

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 to efficiently process minus 28-mesh coal fines and convert them 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 Cyprus Amax Coal Company (Midwest and Cannelton Divisions), Arcanum Corporation, Bechtel Corporation, Center for Applied Energy Research (CAER) of the University of Kentucky, and the Center for Coal and Mineral Processing (CCMP) of the Virginia Polytechnic Institute and State University. Entech Global manages the project for Amax R&D and provides research

and development services. Dr. Douglas Keller of Syracuse University and Dr. John Dooher of Adelphi University are both consultants 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 research and design, along with construction and operation of a 2 ton/hr process development unit (PDU). Tonnage quantities of the ultra-clean coals will be produced in the PDU for combustion testing. Near-term applications of advanced cleaning technologies to existing coal preparation plants is also being studied.

ACCOMPLISHMENTS DURING QUARTER

Activity continued during October - December 1995 on Phases I, II, and III of the project. Work was carried out under Tasks 3, 4, 6, 7, 8, and 9 as described below.

Task 3 Development of Near-Term Applications

A 1993 Bechtel engineering analysis evaluating potential column flotation and selective agglomeration applications found a column flotation application at the Lady Dunn Preparation Plant particularly attractive since the plant was being considered for a major capacity expansion. Because of the potential advantages of installing column flotation rather than mechanical flotation cells in the expanded fine coal cleaning circuit, Lady Dunn management was pleased to offer their plant as the study site for a near-term application of column flotation. The Microcel™ flotation column was selected for this study and the Center for Coal and Mineral Processing (CCMP) at Virginia Tech was assigned the responsibility for the on-site test work. Subtask 3.3, investigating a novel dewatering process for advanced flotation products will also be performed by CCMP.

Subtask 3.2 Engineering Development

Performance data were obtained from the 30-inch Microcel™ column which had been installed in Lady Dunn Preparation Plant in order to test the near-term application of advanced column flotation technology for recovering fine clean coal in a washing plant. A high-quality product meeting the plant requirement of 7-9% ash was produced during initial testing on minus 48-mesh and minus 100-mesh streams. Combustible recoveries were in the 65-80% range.

Twelve drums of the clean coal froth were collected and shipped to the Pittsburgh Energy Technology Center for centrifuge dewatering tests using their GranuFlow process. The GranuFlow process involves mixing an asphalt emulsion (Orimulsion) with the coal slurry before dewatering in order to reduce the amount of moisture remaining in the cake and to improve the handling properties of the fine coal product.

Performance of screen-bowl and solid-bowl centrifuges were compared and cakes with the following moisture contents were obtained:

	<u>No Additive</u>	<u>6-8 % Orimulsion</u>
Screen Bowl	39.4	35.2-35.7
Solid Bowl	34.8	31.0

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

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

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

Results of this work indicated some ambiguities among the capacity and cake moisture projections which may have been due to the differing amounts of flocculant required for each situation. However, it was clear that filtering coarse spiral concentrate along with the froth slurry, either by layering or by premixing, offered little advantage with respect to capacity or moisture removal. While a horizontal belt filter cycle appeared to offer a somewhat higher capacity on a lb/hr/sq ft basis than a drum belt filter cycle, the moisture contents of the resulting cakes were about the same, that is, in the 34-43% range. Based on these results, Lady Dunn management now plans to use a screen bowl centrifuge to dewater the Microcel™ column flotation froth to be produced after the plant expansion.

Subtask 3.3 Dewatering Studies

This work, being performed by Virginia Tech, is aimed at developing a novel hydrophobic dewatering process (HD) for coal fines. In this process a hydrophobic substance is added to a coal-water slurry to displace water from the coal surface. The hydrophobic substance is then recovered for recycle to the process. Three coals will be tested including the product from near-term testing at the Lady Dunn plant (Subtask 3.2).

Subtask 3.3.2 - Identification of Hydrophobic Substances - To identify suitable substances that can readily displace water from coal, contact angle measurements

continued during this reporting quarter. A Ramé-Hart High-Pressure Apparatus (Model 100-08/30) was obtained for contact angle measurements. This compact high-pressure chamber mounts directly onto the stage of the Ramé-Hart Contact Angle Goniometer, providing a means for determining contact angles at elevated pressures and/or temperatures. With this high-pressure apparatus, the contact angle of butane and other high-pressure substances, on coal, can be measured. Unfortunately, the sample stage holder broke during preliminary measurements and was returned for repairs.

In order to validate the combining rule for long-range hydrophobic forces, the forces between dissimilar solids in water were measured. This work was carried out utilizing an atomic force microscope. Results from this work indicate that the combining rule is valid for hydrophobic forces, and that the hydrophobic force is uniquely determined by contact angle. Further, for similar solids, the hydrophobic force is a function of the contact angles of the forces involved, while for dissimilar solids it is determined by the average contact angle involved.

Calculations of potential curves for the displacement of water on coal surfaces were also carried out. For the dewatering process to be kinetically possible, the potential curve for the interaction of coal-water-hexane must not have a barrier. Based on the results of this work, it appears that the barrier disappears when, for the coal-water-hexane system, the contact angle of the coal is less than 87° . However since it is possible that the thickness of the water layer on coal is restricted, dewatering may occur at coal contact angles as low as 73° . In a similar manner, this critical contact angle of coal (for dewatering to be kinetically possible) can be calculated for any reagent, other than hexane, with a known contact angle.

Subtask 3.3.3 - Process Development - Under Subtask 3.3.3.2, a batch dewatering unit was designed and constructed to test this HD process during the last reporting quarter. Shakedown testing of the HD process unit was conducted this quarter on Lady Dunn plant samples using butane as the hydrophobic substance. During initial testing, it was found that the inability to drain all of the water from the vessel resulted in remixing of water with the dewatered coal. To resolve this problem, a sampling assembly was constructed and installed on the cover of the high-pressure vessel.

Following this modification, preliminary tests resulted in coal moistures of 20-25%. These relatively high moistures were attributed to several factors including the recovery of water with the butane phase during sampling, the freezing of moist air during the evaporation of the butane, and the use of oxidized coal. Based on these results, additional modifications to the system were completed, followed by more testing:

- Modification of the sampling chamber allowing evacuation prior to sampling to reduce the effect of the moist air
- A decrease in the depth of the sampling tube to prevent recovery of liquid from the water phase

- An increase in the amount of butane used to increase the thickness of the butane phase
- The use of a fresh coal concentrate from Elkview (British Columbia, Canada)

Under these conditions, moisture contents of 0.9-1.6% were achieved indicating that the modifications made were successful, and that the unit was ready for parametric testing.

Task 4 Engineering Development of Froth Flotation

Task 4 is divided into five subtasks. Subtasks 4.1 Grinding, 4.2 Process Optimization Research, 4.3 Coal-Water-Fuel Formulation Studies, and 4.5 Conceptual Design of the PDU and Advanced Froth Flotation Module have been completed and were reported during previous quarters. There was activity on the remaining subtask, 4.4, during the thirteenth quarter of this project.

Subtask 4.4 Bench-Scale Testing and Process Scale-Up

A draft of the Subtask 4.4 topical report containing bench-scale flotation and toxic trace element reduction data was distributed to team members for comment.

Task 6 Engineering Development of Selective Agglomeration

Task 6 is divided into six subtasks. Subtasks 6.1 Agglomerating Agent Selection, 6.2 Grinding Studies, 6.3 Process Optimization Research, and 6.6 Conceptual Design of the Selective Agglomeration PDU Module have been completed and were reported during previous quarters. There was activity on the two remaining subtasks during the reporting quarter.

Subtask 6.4 CWF Formulation Studies

The primary objective of Subtask 6.4 is to evaluate the formulation of coal-water-fuel (CWF) slurries from selective agglomeration products. The slurry feedstocks used for this work are generated during Subtask 6.5 testing, Selective Agglomeration Bench-scale Testing and Process Scale-up.

While much of this test work will evaluate the effect of various parameters on slurry quality, there are two other objectives for the Subtask 6.4 work. First, this test work will provide a comparison between similar slurries formulated from flotation and agglomeration products, providing some insight into whether one process generates a product inherently more amenable to highly-loaded slurry formulation than the other. Second, the Subtask 6.4 work will attempt to determine slurry quality guidelines for

commercial production. To this end, determinations of required slurry coal loadings, stabilities, and viscosities will be carried out.

The Subtask 6.4 testing began in earnest during the previous quarter of this project. This initial work involved particle size distributions (PSD) characterization of Subtask 6.5 testing final products, i.e., product from the steam stripping circuit used to remove heptane from the recovered agglomerates. This previously reported PSD characterization work was completed for the following coals:

- Taggart 1 (62-mesh topsize) - The Taggart coal utilized for all of the Subtask 4.3 and 4.4 testing, and the initial Subtask 6.5 start-up testing.
- Taggart 2 (62-mesh topsize) - The replacement Taggart coal which will be utilized for the remainder of project testing. While this coal comes from the same seam as the Taggart 1 coal, it is from a different mine.
- Sunnyside (150-mesh topsize) - This coal was used during Subtask 4.3 and 4.4 testing, and the initial Subtask 6.5 testing. This coal has since been replaced with the Hiawatha coal.
- Elkhorn No. 3 (100-mesh topsize) - This coal was used during Subtask 4.3 and 4.4 testing, and is also being used for Subtask 6.5 testing.
- Indiana VII (325-mesh topsize) - This coal was used during Subtask 4.3 and 4.4 testing, and is also being used for Subtask 6.5 testing.

It should be noted that based on Subtask 6.5 testing results, the Taggart 2 coal will require a grind finer than 62-mesh to achieve the 1 lb/MBtu target product ash specification set for this coal. As such, no further slurry formulation work will be carried out with this 62-mesh topsize Taggart 2 coal agglomeration product.

Subtask 6.4 testing continued during this reporting quarter with the formulation of slurries utilizing Elkhorn No. 3 coal product from the 25 lb/hr selective agglomeration bench scale unit. Steam stripping circuit product was used for this work.

Testing was carried out to characterize the formulation of slurries from the "as-received" Elkhorn No. 3 coal agglomeration circuit product. Additional testing was also carried out to provide a comparison between the formulation of slurries from advanced flotation and selective agglomeration products. This was accomplished by repeating the formulation of various slurries carried out during Subtask 4.3 testing. As such, slurries were formulated from the following Elkhorn No. 3 coal feedstocks:

- As-received agglomeration product using 0.5 and 1.0% A-23 dispersant. Coal loadings for these slurries were in the 57-60% (by weight) range.
- A blend of 70% as-received material and 30% material reground for 30 minutes in the attritor mill, using 0.5% A-23 dispersant. This blend is referred to as the 70/30 regrind blend and resulted in coal loadings in the 58-61% (by weight) range.

There is some scatter in the data for slurries formulated from 100% agglomeration product. While it appears that slurries formulated from the 70/30 regrind blend achieved slightly lower viscosities, at the same coal loadings, than the 100% as-received agglomeration product slurries, some uncertainty exists. Also, while no Flocon was utilized in the formulation of these slurries, it is expected that its use as a stabilizer would result in increased slurry viscosity as seen in all previous slurry formulation work.

While the trends observed as a result of PSD manipulation may be similar, it appears that lower viscosities, at similar coal loadings, are achieved with agglomerated product than with flotation product. It is believed that this may be due to several reasons including:

- The presence of aggregates of particles in the steam stripped agglomeration circuit product, effectively coarsening the PSD
- A steam stripping process effect on the surface properties of the coal
- The presence of residual heptane in the agglomeration circuit product

All of the slurries formulated with the Elkhorn No. 3 coal had poor stabilities with a rating of "1" after overnight storage. While this rating of "1" indicates that at least 25% of the slurry volume was occupied by a very hard pack sediment, for all of these slurries, at least 65% was very hard packed.

In addition to this test work, discussions were held with Dr. John Dooher of Adelphi University, a consultant being used for the Subtask 6.4 work. Based upon his recommendations, future testing will incorporate a revised viscosity determination procedure in which more time is provided for the slurry to achieve equilibrium. In addition, evaluation of slurries formulated at a pH of 10, rather than the natural pH of approximately 7, will be carried out.

Subtask 6.5 Bench-Scale Testing and Process Scale-up

During previous testing utilizing the 25 lb/hr bench-scale unit, evaluation of the Taggart 1, Sunnyside, and Elkhorn No. 3 coals were completed. It should be noted, that the Taggart 1 and Sunnyside coals have since been replaced with the Taggart 2 and Hiawatha coals, respectively, and as such will not be tested further. While previous testing on the Indiana VII coal indicated that product ash specifications could be met at the selected 325-mesh tosize grind, additional testing with this coal may be carried out later to optimize the operating conditions. Previous work also included preliminary evaluation of the Taggart 2 coal. Selective agglomeration bench-scale testing this quarter focused on the Winifrede and Hiawatha coals, with some additional work carried out on the Taggart 2 coal.

Winifrede Coal - To achieve the desired particle size distribution (PSD), the Winifrede coal was initially ground in the 4' x 4' ball mill in open circuit. The product from this

grind was then subjected to five passes through the Drais fine grinding mill. This ground product had a mass mean diameter of 7.14 microns. Sixteen continuous agglomeration tests using this ground Winifrede feedstock were carried out during this quarter. This test work utilized both fresh commercial grade heptane and recycled commercial grade heptane.

The primary variables changed during the completion of these sixteen agglomeration tests were:

- Feed slurry solids concentration
- Coal feed rate
- Heptane type
- Heptane concentration
- Asphalt addition
- High shear tip speed
- Low-shear tip speed
- Low-shear impeller configuration
- Low-shear residence time
- Vibrating screen inclination
- Vibrating screen spray water rate

Results from this work indicate that the 2 lb ash/MBtu specification was met for most of the tests completed, indicating that the 7.14 micron mass mean diameter grind provides sufficient mineral-matter liberation. This data also indicates that very high Btu recoveries (>97%) were achieved for all but one of the tests. Corresponding tailings ash values for most of these tests were in the 78 to 89% range. No difference in the operation of the test unit was observed when fresh commercial grade heptane was used as compared to recycled commercial grade heptane.

During virtually all of these tests, only a marginal inversion was achieved in the high shear unit operation. This is attributed to the extreme fineness of the grind and the inability of the high-shear reactor to provide higher energy inputs. As a general trend, higher high-shear impeller tip speeds and lower volumetric feed rates, i.e., longer residence times, provided improved inversion. It should be noted, however, that very clear inversions were never achieved.

It was found, during this testing, that even though a good inversion could not be achieved during high shear, agglomerate growth during low shear was sufficient to afford good recovery during screening. In fact, when sufficient heptane was utilized, growth during low shear was easily achieved and sometimes proved difficult to control.

Generally, when sufficient heptane was used, tests completed with a low-shear impeller tip speed of 4.8 m/s resulted in continuous agglomerate growth, eventually plugging the low-shear discharge. However, when an 8 m/s low-shear impeller tip speed was used,

agglomerate size usually cycled from the <0.5 mm range to the 2-3 mm range. This trend is similar to the bulk of testing completed previously. Also as observed previously, it was found that when well formed agglomerates were produced, lower product ash contents were achieved. This is due primarily to better drainage of mineral matter bearing process water.

Since the Winifrede coal will not be used during PDU 2 t/hr testing, no additional testing of this coal in the 25 lb/hr bench-scale agglomeration test unit is planned.

Hiawatha Coal - Fourteen continuous agglomeration test were completed with the Hiawatha coal during this quarter. This coal was closed circuit ground in the 4' x 4' ball mill with a 150-mesh screen. This grind size was chosen based on an estimate of liberation requirements to achieve the desired 2.0 lb/MBtu product ash content.

The test work investigated a number of agglomeration operating variables, but focused primarily on the evaluation of the low-shear unit operation. It particularly focused on the discharge from the low-shear vessel, which has been of primary concern due to plugging problems experienced throughout Subtask 6.5 testing.

In an effort to eliminate plugging of the low-shear vessel, two additional sets of discharge ports were installed in the vessel. One set are 2-inches in diameter, and located to allow an overflow discharge. The second set are 1-inch in diameter, and located at the same elevation as the impellers, utilizing an upflowing discharge to maintain the correct operating level in the vessel.

Evaluation of these different discharge arrangements has resulted in the following observations:

- While the use of the 2-inch overflow discharge works well when the vessel is operated half-full, excessive splashing appears to interrupt the desired mixing pattern. As such, no agglomerate growth was observed.
- The 2-inch overflow discharge, when utilized with the vessel full, provides good agglomerate discharge. However, it was found that the lower half of the vessel packs full of agglomerates. As such, solids discharge reduces over time resulting in solids buildup and eventual plugging of the vessel.
- Use of the 1-inch discharge port located at the impeller height worked well when the vessel was operated half-full. At these conditions, steady-state operation appears to have been achieved. However, monosized agglomerates were not formed. Instead, agglomerates typically ranged from 0.5 to 3 mm in size and were oval in shape rather than round.
- Use of the 1-inch discharge port with a full vessel resulted in plugging of the lower half of the vessel, as was the case for the 2-inch discharge port.

Based on these observations, it appears that the transfer of agglomerates from the lower to upper section of the low-shear vessel is a bottleneck. Attempts will be made to

improve this flow during future testing by increasing the opening in, and/or completely removing, the horizontal baffle which separates the two mixing zones.

It should be noted that the location of the discharge ports at the impeller height has resulted in improved agglomerate discharge, confirming the low shear PDU design.

Product ash values for this test work ranged from approximately 2.1 to 3.2%, indicating that sufficient liberation is achieved at the 150-mesh topsize grind to meet the desired 2 lb/MBtu product ash specification. As such, during future testing, a 100-mesh topsize grind will be evaluated. Tailings ash values for all of these tests were consistently in the 82-87% range, indicating that Btu recoveries were in the 95-99% range.

Taggart 2 Coal - During previous testing, the Taggart 2 coal was closed-circuit ground to a 62-mesh topsize for evaluation in the continuous agglomeration bench-scale unit. Results from work using this feedstock indicated that product ash contents of 1.3-1.5 lb ash/MBtu were achieved. Therefore, the Taggart 2 coal was closed-circuit ground to a 100-mesh topsize this reporting quarter in an attempt to achieve the target product ash content of 1 lb ash/MBtu.

Two agglomeration tests were completed utilizing this feedstock. These two tests resulted in product ash contents of 1.42 and 1.44 lb ash/MBtu, indicating no additional liberation for this finer grind. Additional liberation studies will be carried out with the Taggart 2 coal during future testing to determine the grind required to insure that a product ash content of 1 lb ash/MBtu can be met in the 25 lb/hr continuous selective agglomeration bench-scale unit.

Task 7 PDU Selective Agglomeration Module Detailed Design

Module Detailed Design

Work was essentially completed on the detailed design of the PDU selective agglomeration module during this reporting quarter. Sufficient progress was made on this design to allow for the issuing of the construction bid package. Selective agglomeration module detailed design work remaining involves the preparation and issuing of the final task report.

Design Overview

The following sections of this report discuss design features of the plant unit operations, with emphasis on the selective agglomeration module.

Coal Grinding and Dewatering - It is not anticipated that any major changes in either the coal grinding or dewatering circuits will be required for the switch over from the flotation to agglomeration process module. As such, the agglomeration process will

use slurry ground by the installed equipment. The ground product will be pumped to the current Microcel™ flotation column feed tank. The product stream from this tank will be diluted to the desired solids concentration and sent to one of two ground slurry storage tanks. From these storage tanks, the slurry will be metered to the agglomeration process. Once processed, the final steam stripped clean coal product and the process tailings will be dewatered utilizing the existing equipment.

High Shear - High-shear agglomeration will be carried out in a circuit with two high-shear reactors. These reactors will be sized to provide 0.5 and 1 minute residence times respectively, and each will be powered by a variable speed drive that can achieve up to 18 m/s impeller tip speeds. In this manner, high shear residence times from 0.5 to 1.5 minutes can be achieved by operating either unit individually, or both together in series. Heptane will be metered to the agglomeration process as required, currently anticipated not to exceed 40% of the coal feed rate. The ability to add asphalt to the high-shear circuit will also be provided for Indiana VII coal testing.

Low Shear - Low-shear agglomeration will be carried out in a single vessel divided into two sections via a horizontal baffle. Each section will provide 2.5 minutes of residence time. The discharge piping will be arranged such that one or both sections can be utilized. The low shear vessel will be powered by a single variable speed agitator driving one impeller for each section of the vessel. The impellers will be of the radial-flow type and the drive unit of sufficient HP to achieve tip speeds in the 3-5 m/s range.

Agglomerate Recovery - The vibrating screen to be used to recover agglomerates from the low shear product will be a 48-mesh dewatering screen approximately 2-feet wide by 6-feet long. Sufficient spray nozzles will be provided to insure replacement of mineral matter bearing process water with fresh water. The vibrating screen underflow (tailings) will then be processed through a froth skimmer, which will provide three minutes residence time for any carbonaceous material to float. If necessary, nitrogen will be used to help float this material. A continuously rotating paddle will scrape the floating material to a launder from where it will be combined with the screen product.

Steam Stripper - The combined screen and froth skimmer product will be diluted with hot water to approximately 25% solids. This feed will then be treated in the first stage steam stripper which will provide 5 minutes residence time at 94°C and ambient pressure. The heat source for this stirred vessel will be the vapor product (steam and heptane) from the second stage stripper. This first stage stripper has two primary functions. First it will remove the bulk of the heptane present (about 99%), and second, it will generate a handleable and pumpable product. This first-stage stripper product will then be pumped to the second-stage stripper which will provide 10 minutes residence time at 115-120°C and 15-20 psig. The product from this second stage stripper will then be cooled and sent to the dewatering circuit.

Condenser and Gravity Separator - Vapors from the first stage stripper will contain steam and recovered heptane and be condensed in a two-stage process. Initial condensation and some cooling will be carried out by an air cooler, followed by a shell

and tube heat exchanger to provide the necessary sub-cooling. The condensed liquids will then be sent to the gravity separator from which both the heptane and the process water will be recycled to the process.

Equipment Specifications

Work was also completed on the development of equipment specifications for the bulk of the major capital equipment items slated for purchase. As such, purchasing began late in the quarter. Plant equipment for which specification finalization remains includes primarily instrumentation.

Miscellaneous Tasks

Work was initiated to bring the existing GC analyzer on line during this reporting quarter. This will allow trace heptane determinations during the latter part of Subtask 6.5, and throughout Subtasks 9.2 and 9.3, to be completed in-house. Huffman Laboratories was retained as a consultant for this work. The GC analyzer has been started up and some preliminary testing initiated, with results indicating that the GC is working well. Work remaining under this task involves the programming of the integrator and final procedure development to allow routine operation of the analyzer throughout the remainder of the project.

Task 8 PDU and Advanced Column Flotation Module

Work completed as part of Task 8 this reporting quarter involved coal selection and procurement under Subtask 8.1 and PDU start-up, shakedown, and test plan development under Subtask 8.3

Subtask 8.1 Coal Selection and Procurement

Four cars of Indiana VII compliance coal were received from the Minnehaha Mine and stored at a coal yard in Denver.

Subtask 8.3 PDU Advanced Coal Cleaning Module Shakedown and Test Plan

As scheduled, shakedown testing of the PDU flotation module was completed during the quarter. Entech personnel completed many physical changes in the PDU flotation module which resulted in improved capacity and reliability.

The test plan was also completed and submitted to the DOE Pittsburgh Energy Technology Center for review and approval. The plan describes the proposed testing schedule, along with the parameters to be evaluated for each of the three test coals.

PDU Flotation Module Shakedown - Shakedown of the PDU flotation module was completed during the quarter. Because the operation of individual equipment items were checked during the previous quarter, efforts were directed to overall startup and operation of the PDU flotation module. Taggart coal was used for all startup efforts. Physical and mechanical improvements resulted in the elimination of process bottlenecks which allowed Entech personnel to achieve the desired PDU feed rate of 4,200 lb/hr.

Process Improvements - Operation of the PDU Flotation Module during the quarter indicated that some plant changes were required to improve overall process reliability and reduce any potential downtime. The most noticeable improvements were made in the PDU's processing capacity and clarified water quality. Bottlenecks at the Primary Ball Mill entrance and Microcel™ launder were eliminated allowing the PDU feed rate to be increased to 4,200 lb/hr. To eliminate the high solids concentration in the drum filter filtrate, the WesTech filtrate stream was re-routed to a sump which can be fed to any of the PDU's pressure filters. The result has been improved clarified water quality.

Instrument Calibration - To better operate and control the PDU Flotation Module, all process control and display instrumentation was double-checked during the quarter for measurement accuracy. Entech personnel are confident that instruments performing various process measurements are accurate.

PDU Flotation Module Test Plan - The PDU flotation module test plan was completed and submitted on December 14, 1995 to the Pittsburgh Energy Technology Center for review and approval. A training session, described in the test plan, was given to all Entech technicians on December 7, 1995. The session covered startup, operation, and shutdown of the PDU flotation module.

Though parametric testing of the three test coals was initially scheduled for December, 1995, unexpected problems encountered during startup and shakedown of the PDU flotation module will push the start of test work to January, 1996. This schedule adjustment will not impact the overall project schedule since PDU process improvements will permit more testing to be completed each day.

Task 9 Selective Agglomeration Module

Phase III of this project involves the construction and operation of a 2 t/hr selective agglomeration (SA) PDU module. This SA module will be integrated with the existing PDU facility constructed during Subtask 8.2 and currently being operated under Subtasks 8.3 and 8.4. During operation of the SA module, the existing coal handling and grinding circuits will be used to generate ground slurry feed for the selective agglomeration process. Similarly, the existing product and tailings dewatering circuits will also be used. As such, the SA module will essentially replace the Microcel™ flotation column, with the remainder of the plant remaining intact.

Just like the advanced flotation PDU, selective agglomeration process performance will be optimized at the 2 t/hr scale, and 200 ton lots will be cleaned for each of the three test coals. Toxic trace element distributions will also be determined during the production runs. The ultra-clean coals will be delivered to DOE for end-use testing.

Subtask 9.1 Construction

The initial task under Phase III of the project involves the construction of the SA module. This work was initiated during Task 7 (Phase II) with the completion of the detailed design of the SA module. Utilizing this detailed design, a construction bid package was prepared and submitted to the following Colorado based contractors on December 15, 1995:

- The Industrial Company, Steamboat Springs, CO
- Read Industrial Corporation, Wheatridge, CO
- Western Industrial Contractors, Denver, CO
- Mech El, Inc., Aurora, CO

As of the end of this reporting quarter, the following schedule is planned for completion of Subtask 9.1:

- | | |
|-------------------------------|--|
| • January 3, 1996, Wednesday: | Site Inspection by Interested Bidders |
| • January 15, 1996, Monday: | Proposal Due at Amax R&D |
| • January 22, 1996, Monday: | Selection of the Subcontractor |
| • February 1, 1996, Thursday: | Letter of Intent to Award the Contract |
| • February 12, 1996, Monday | Signing of the Subcontract |
| • February 15, 1996, Thursday | Mobilization |
| • June 30, 1996 | Construction Completion |

Material Requisitions - Material requisitions (MR) for the bulk of the capital equipment to be purchased for SA module construction were issued during December and early January. Material requisitions for seven additional capital items have yet to be issued. It has also been determined that eight used equipment items, currently on-site, will be used during the construction of the SA PDU module. Any reconditioning or modifications required for this used equipment will be initiated early next quarter.

Water Supply - In anticipation of Task 9 utility water requirements, and to reduce the occurrence of drops in the pilot-plant water pressure during on-going plant operations, installation of a 6-inch water line was completed. Plans for this water supply include a 2-inch line to tie into the existing plant water system, a 2-inch line for Microcel™ wash water, and several other 2-inch supplies to be used during PDU selective agglomeration module operation.

INTRODUCTION AND BACKGROUND

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

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

This multi-year cost-shared contract effort began on October 1, 1992, and is scheduled for completion by June 30, 1997. This report discusses the technical progress made during the thirteenth quarter of the project, October 1 to December 31, 1995. Twelve quarterly reports have been issued previously [1-12].

SPECIFIC OBJECTIVES OF THE PROJECT

The three main objectives of this project are discussed below.

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

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

purpose of economically and efficiently processing minus 28-mesh coal fines. The design base 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 targeted. They are antimony, arsenic, beryllium, cadmium, chromium, cobalt, lead, manganese, mercury, nickel, and selenium. The results will show the potential for removing these toxic trace elements from coal by advanced physical cleaning.

APPROACH

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

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

Phase I

Phase I encompassed preparation of a detailed Project Work Plan, selection and acquisition of the test coals, and laboratory and bench-scale testing. The laboratory

and bench-scale work determined the cleaning potential of the selected coals and established design parameters and operating guidelines for a 2 t/hr PDU containing both advanced column flotation and selective agglomeration modules. A conceptual engineering design was prepared for a fully integrated and instrumented 2 t/hr PDU incorporating the features determined from the laboratory and bench-scale studies.

Additional activities to be completed during Phase I include:

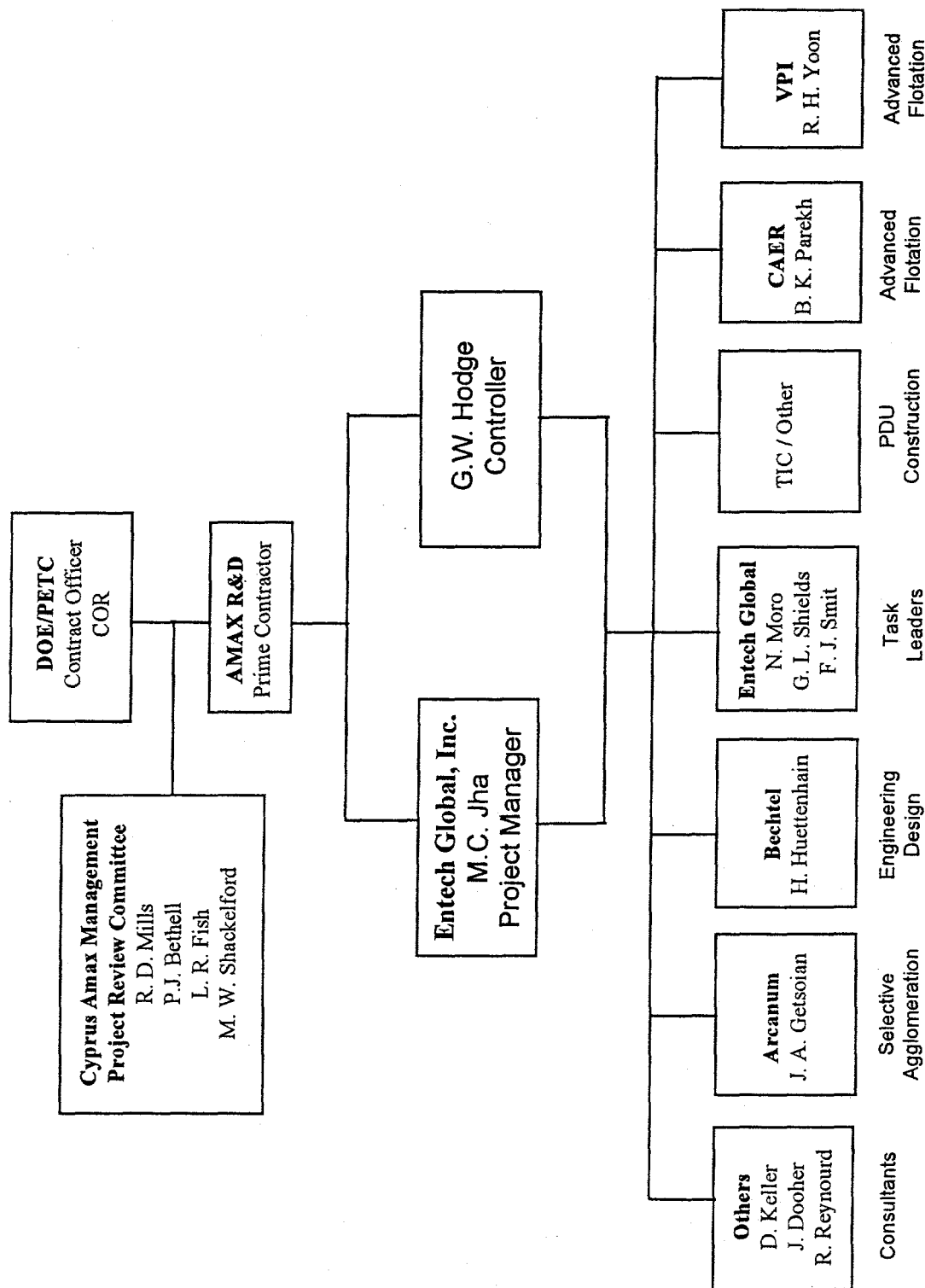
- Production of ultra-clean coal test lots by bench-scale column flotation and selective agglomeration for end-use testing by DOE or a designated contractor
- Determination of toxic trace element distribution during production of these test lots
- Evaluation of the rheological properties of slurry fuels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

Phases II and III

Phases II and III cover the construction and operation of the 2 t/hr PDU. Phase II is for advanced column flotation while Phase III is for selective agglomeration. Process performance will be optimized at the PDU-scale, and 200 ton lots of ultra-clean coal will be generated by each process for each of the three test coals. Toxic trace element distributions will also be determined during the production runs. The ultra-clean coals will be delivered to DOE or a designated contractor for end-use testing.

Phase IV

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



Revised December 1, 1995

Figure 1. Project Management Organization Chart

Table 1. Outline of Work Breakdown Structure

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

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

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

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

Phase III. Selective Agglomeration Module Testing and Evaluation

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

Phase IV. PDU Final Disposition

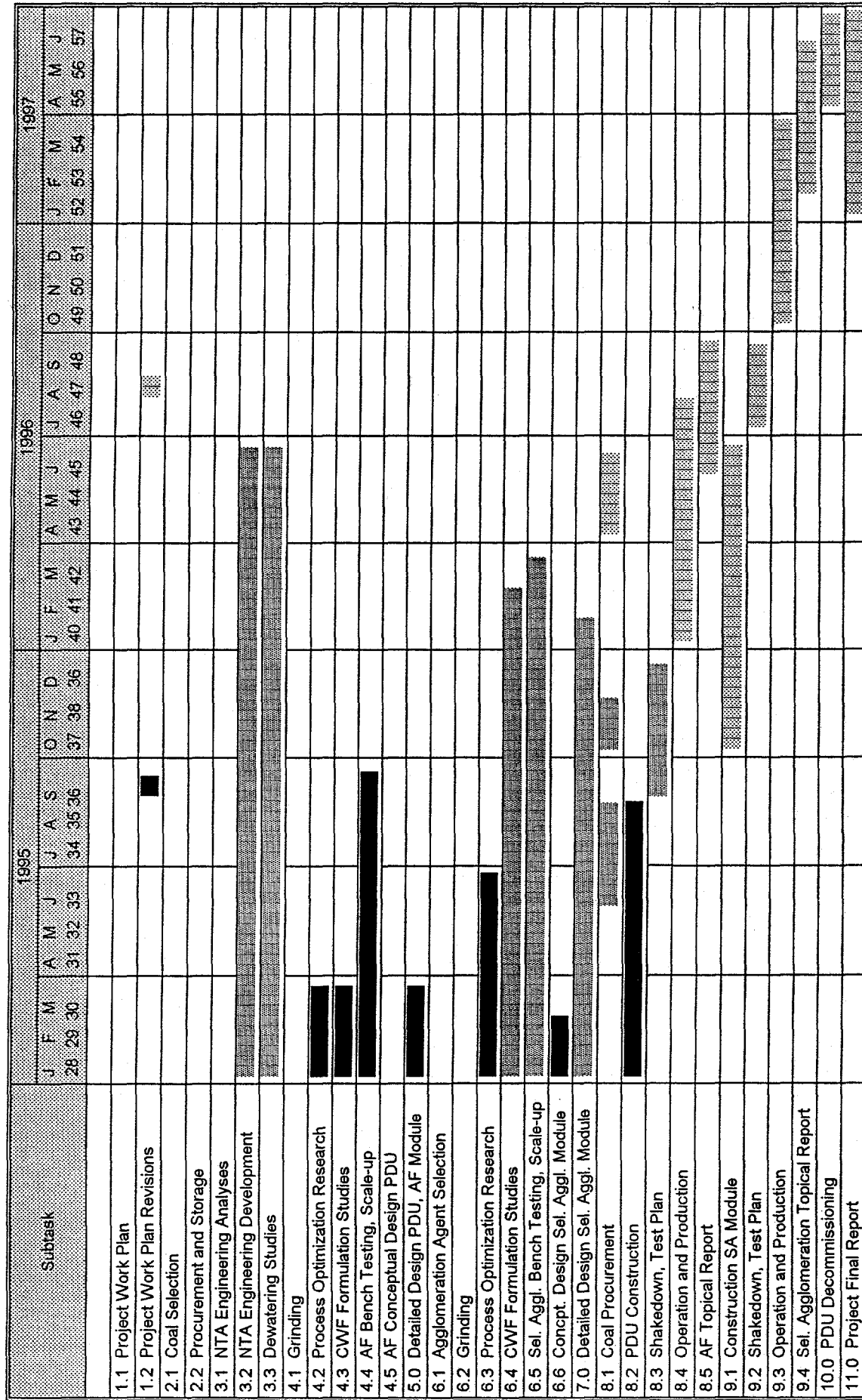
- Task 10. Disposition of the PDU
 - Task 11. Project Final Report
-

Revised April 25, 1995

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

Figure 2. Project Schedule

Revised October 16, 1995



Revised October 16, 1995

Figure 2. Project Schedule (Cont'd)

ACCOMPLISHMENTS DURING QUARTER

Work was carried out on Tasks 3, 4, 6, 7, 8, and 9 during the thirteenth quarter (October 1 to December 31, 1995) reporting period. Good progress was made on these tasks as discussed below.

TASK 3 DEVELOPMENT OF NEAR-TERM APPLICATIONS

During 1993, Bechtel performed an engineering analysis evaluating potential applications for column flotation and selective agglomeration at three coal preparation plants operated by what is now Cyprus Amax Coal Company [13]. Economic projections favored column flotation over selective agglomeration and an application at the Lady Dunn Preparation Plant (Cannelton Coal Company) was found to be particularly attractive since the plant was being considered for a major capacity expansion. Because of the potential advantages of installing column flotation rather than mechanical flotation cells in the expanded fine coal cleaning circuit, Lady Dunn management was pleased to offer their plant as the study site for a near-term application of column flotation. The Microcel™ flotation column was selected for this study and the Center for Coal and Mineral Processing (CCMP) at Virginia Tech was assigned the responsibility for the on-site column testing under Subtask 3.2 Engineering Development. During the previous reporting quarter, a new subtask, 3.3 "Dewatering Studies", was added to the project. This work is also being performed by CCMP.

Subtask 3.2 Engineering Development

As discussed in previous quarterly reports [10,11,12], the Lady Dunn Preparation Plant in West Virginia is the host site for testing Microcel™ column flotation recovery of clean coal in the fines screened and cycloned from the raw coal. The clay content is quite high in this stream at the Lady Dunn Plant, and as a result, clean coal recovery with the existing mechanical cells is poor. For this reason, the Lady Dunn application is a good test of the near-term applicability of column flotation in many preparation plants. The Center for Coal and Mineral Processing (CCMP) at Virginia Tech is supervising the test work for the local plant management. The main emphasis of the work so far has been to obtain scale-up information for a plant expansion which includes column flotation.

An existing 30-inch diameter Microcel™ test unit was refurbished and installed in the plant for this test work. It was first piped to receive minus 100-mesh desliming cyclone overflow. A high-quality product, 7-9% ash, meeting Cannelton requirements was produced at a good recovery of combustible matter during preliminary testing.

A goal of Cannelton management, though, was to clean a coarser stream by column flotation. Specifically, they wished to float the fines generated when preparing feed for the spiral separators which would be installed in 1996 as part of the plant expansion.

For this reason the flotation column was repiped to receive minus 0.75-mm fines screened directly from the raw coal. Parametric testing has begun on the coarser slurry, but progress has been slow because of the intermittent operating schedule of the Lady Dunn Plant during December.

Centrifuge Dewatering Tests

Consideration is being given to methods for dewatering the clean coal product from the Microcel™ flotation column. Centrifuging and vacuum filtration have been evaluated for this application. In this regard, twelve drums of clean coal froth product slurry were collected from the 30-inch Microcel™ column and shipped to DOE/PETC for centrifuge testing by the Coal Preparation Research Division (CPRD). The column was receiving minus 48-mesh combined screen undersize and cyclone overflow at the time the clean coal froth was collected.

The CPRD conducted the centrifuge dewatering tests using the patented GranuFlow process. Two to eight percent of an asphalt emulsion from Venezuela called Orimulsion was added to the centrifuge feed during the GranuFlow process testing in order to improve the properties of the dewatered product. The properties most effected by the Orimulsion are the moisture retention and handleability (stickiness and potential dustiness) of the cake. Baseline tests were performed without the additive as well.

Two types of continuous-feed centrifuges available at PETC were used for the dewatering tests. The first was a 6-inch diameter, 576 g-force screen-bowl. The second was a 14-inch diameter, high g-force (1789 g-force) solid-bowl. The g-force in the 6-inch screen-bowl was similar to g-forces in screen-bowl centrifuges commonly installed to dewater fine coal, while the g-force in the 14-inch solid bowl was similar to g-forces in recent-vintage high-speed solid-bowl decanter centrifuges.

Figure 3 presents residual moisture in the centrifuge cakes versus the amount of Orimulsion added to the clean coal froth slurry. Without the Orimulsion, the screen-bowl cake contained 39% moisture, and the solid-bowl cake contained 35% moisture. The difference between these values indicates the effect of higher speed centrifuging on product moisture content. In both instances, adding 6-8% Orimulsion decreased the amount of moisture remaining in the cake (to the 35-36% range in the case of the screen-bowl tests, and 31% in the case of the solid-bowl test).

Further results of the centrifuge tests are presented in Table 2 and indicate that GranuFlow processing with Orimulsion had other benefits in addition to moisture reduction. First, the potential dustiness of the cake upon drying was reduced by agglomeration of the fines with the asphalt. This is shown by the change in the Dust Index and the Dust Reduction Efficiency entries in the table. These are measurements of the amount of minus 100-micron material released during sieving of the dried cakes as shown below:

Dust Reduction Efficiency, %:

$$E = \frac{(I_o - I_i)}{I_o} \times 100 \quad \text{Where:}$$

I_o = dust index of feed coal, weight % minus 100 μ m by wet screening and

I_i = dust index of cake, weight % minus 100 μ m in dry cake by Ro-Tapping 5 min

The agglomeration also reduces the stickiness of wet cakes, and test 16-4 (see Table 2) in the solid-bowl centrifuge produced free-flowing granules. A further benefit of the GranuFlow process was the improved solids recovery during screen-bowl centrifuging, also due to the agglomeration of fine particles in the feed slurry. Solids recovery was even better when dewatering with the high-g solid-bowl centrifuge, and the resulting effluent may even have been clean enough for reuse without clarification.

A detailed GranuFlow test report, prepared by George Wu-Wey Wen of PETC, is presented in Appendix A.

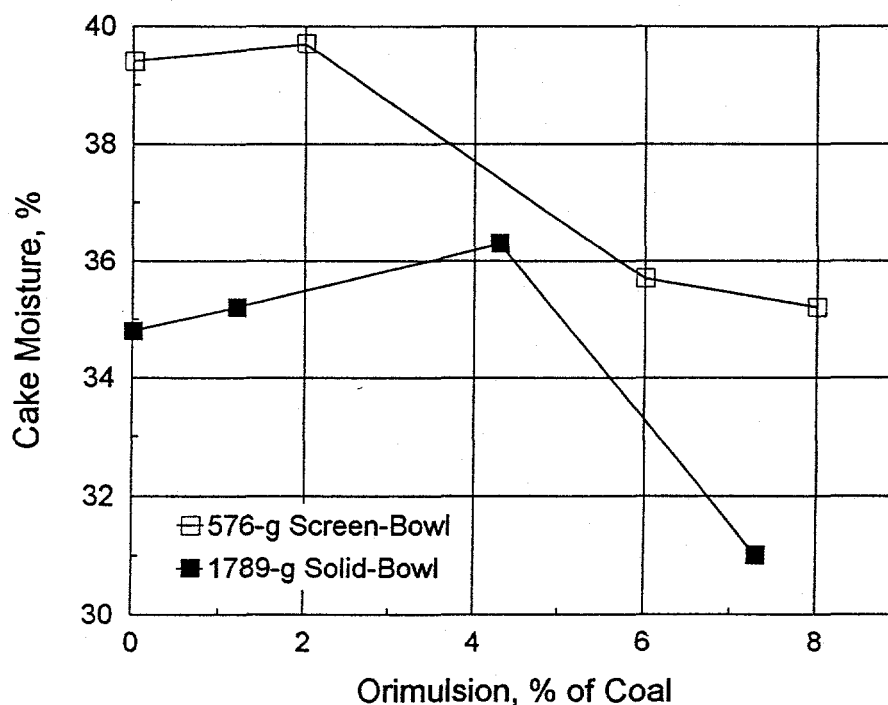


Figure 3. Cake Moisture vs Orimulsion Addition - Microcel™ Product Centrifuge

Vacuum Filter Dewatering Tests

Laboratory vacuum filtration leaf tests were conducted on the froth slurry by Westech Engineering, Inc. personnel [14]. Most of these tests were performed at the Lady Dunn plant, but a few supplementary tests were performed at the Westech laboratory in Salt Lake City. The objectives of the leaf testing was to project the capacity and performance of top feed horizontal belt filters and of bottom feed drum filters. The

laboratory evaluation also included evaluation of the benefits of layering spiral concentrate onto a horizontal filter ahead of the froth slurry to form a deep filter bed.

Table 2. GranuFlow Centrifuge Testing Results

<u>Test</u>	<u>Orimulsion wt %</u>	<u>Cake moist. %</u>	<u>Main Effluent, % solids</u>	<u>Dust Index</u>	<u>Dust Reduction Efficiency wt %</u>	<u>Solids Recovery wt %</u>
6-inch Screen-Bowl Centrifuge (g-force = 576):						
17-1	0	39.4	1.13	83	3	89.5
17-2	2	39.7	1.40	34	0	92.0
17-3	6	35.7	0.91	7	92	95.1
17-4	8	35.2	0.73	4	95	96.5
14-inch Solid Bowl Centrifuge (g-force = 1789):						
16-1	0.0	34.8	0.17	73	15	98.6
16-2	1.2	35.2	0.10	66	23	99.2
16-3	4.3	36.3	0.12	13	85	99.0
16-4	7.3	31.0	0.10	7	92	99.2

Top Feed Procedure - The filter test leaf consisted of a round disk with drainage grooves and a cloth support grid on one side and a filtrate discharge connection on the other side. A filter cloth and a dam high enough to retain the required volume of slurry (typically less than 650 ml) were clamped onto the edge of the disk. The effective filtering area of the leaf was about 0.078 sq ft.

Before the start of each test, the test leaf was placed in position on the vacuum flask with the drain valve closed and the desired quantity of slurry placed in an Erlenmeyer flask. Next, the vacuum was turned on and adjusted to the desired level. After that, flocculant was mixed with the slurry and the flask vigorously swirled to put all of the solids in suspension. Then, in quick succession, the slurry was poured onto the test leaf, the valve beneath the leaf opened to apply vacuum, and the timer started.

Each test run consisted of the two operations of cake formation and final drying, with the cake formation time taken as the time required for all of the free slurry to disappear from the surface of the cake. After the final dry time, the vacuum was turned off and the cake discharged. The following observations were recorded for each test:

- Vacuum level
- Cake formation time
- Final dry time
- Final cake thickness
- Wet and dry cake weights
- Filtrate volume and an evaluation of its clarity

- Quantity of flocculating polymer used
- Volume of air passing through cake during drying period (optional)
- Ease of cake discharge and amount of cake remaining on cloth

Some of the tests involved the application of an initial layer of coarse spiral concentrate followed by a second layer of froth slurry. A special procedure was followed to allow the second layer to form without unduly disturbing the layer of coarse solids and also to allow the initial layer to act as a filter medium for the froth slurry. The spiral concentrate was also mixed directly with the froth slurry prior to filtration for some of the tests.

Bottom Feed Procedure - A typical set-up for the bottom feed testing is essentially the same as the equipment for the top feed testing except that the dam was only slightly deeper than the maximum expected cake thickness.

About one gallon of slurry was placed in a bucket and flocculated with a power stirrer for these tests. Mixing continued for about 30 seconds after the polymer addition was completed. When starting these tests, the hose between the test leaf and the vacuum flask was crimped by hand and the vacuum adjusted to the desired level. The leaf was then immersed in the hand- or paddle-agitated slurry. Time was started as soon as the crimp in the hose was released. After the desired cake formation period, the leaf was removed from the slurry and held with the cake uppermost for the final drying. At the end of the desired drying time, the vacuum was shut off, and all of the same observations noted for the top feed tests were noted for the bottom feed tests as well.

Test Variables - Two different types of froth slurry were tested:

- Froth from the column when receiving "fine feed" (minus 100 mesh cyclone overflow)
- Froth from the column when receiving "coarse feed" (cyclone overflow plus a small amount of minus 48 mesh coal from another stream).

As a practical matter, the particle size distributions of the two froth slurries did not differ very much [12]. A considerable amount of launder water was needed when producing the two slurries so they only contained 10-12 % solids. While most of the tests were carried out on the slurries at this as-received solids concentration, additional tests were completed at the end of the program using slurries thickened to the 15-20% solids range, simulating an operation in which only minimal amounts of launder water would have been used. Unit capacities improved with the higher solids concentration feeds.

The use of the coarse spiral concentrate (basically 10 x 150 mesh) as a filter aid was evaluated for top feed filter tests only. It was added at ratios between 0.37 and 1.71 pounds of spiral concentrate (dry weight) per pound of froth slurry solids (dry weight). As indicated above, it was evaluated as an initial layer (similar to a precoat) for the filter and as a bulking agent mixed in with the froth slurry feed. The weight of the spiral concentrate additive (and accompanying moisture) was deducted from the total weight

of the cake when projecting filter test performance. It was found that the projected filter performance did not change appreciably when changes were made to the spiral concentrate/froth solids ratio.

The froth slurries and the spiral concentrate both contained a considerable amount of residual clay. The use of a low to medium molecular weight anionic polymer was soon found to be beneficial for flocculating the slurries before filtration, and such flocculants were added to both the clean coal froth slurry and the spiral concentrate for most of the testing.

An intermediate-permeability cloth (POPR-859, 100 cfm/sq ft permeability) worked quite well with the flocculated slurries and was used for most of the testing. Sixty and 300 cfm/sq ft cloths were also evaluated. It was necessary to wash the cloth after each test since the cloth would blind after a few cycles. Coarse material from the layered sequence cycles tended to adhere to the cloth and also necessitated a washing step after each cycle. A submerged-blow cleaning procedure was tried but with questionable success.

Cake formation times were selected to bracket formation of 3/8- to 1/2-inch thick cakes, and drying times were selected to bracket practical drying time cycles for commercial horizontal belt and bottom-feed scraper- and belt-discharge drum filters. It should be noted that in most cases, a 3/8-inch cake is about the minimum thickness that will discharge reliably from a filter cloth.

Projections of Filter Performance - A total of 122 laboratory vacuum filtration leaf tests were performed. Westech employed engineering correlations to project filter performance from these data, the principal correlations being from time versus cake thickness, or weight and drying time versus the amount of moisture remaining in the cake. Capacities of operating vacuum filters were then calculated from these correlations using practical cycles of cake formation, drying, discharge, and cloth washing periods for the individual types of equipment. Because of the cloth washing requirement, the performance projections were limited to top feed horizontal and bottom feed drum belt filters. Table 3 provides a summary of the Westech projections when producing 3/8-inch thick cake. Projected capacities were less when producing 1/2-inch thick cake but cake moistures were unchanged.

There were some ambiguities among the capacity and cake moisture projections which may have been due to the differing amounts of flocculant required for each situation. However, it was clear that filtering coarse spiral concentrate along with the froth slurry, either by layering or by premixing, offered little, if any, advantage with respect to unit capacity or moisture removal. While a horizontal belt filter cycle appeared to offer a somewhat higher capacity on a lb/hr/sq ft basis than a drum belt filter cycle, the moisture contents of the resulting cakes were about the same, in the 34-43% range.

After considering these results, the Lady Dunn Plant management decided to forego pilot-scale testing of a horizontal belt filter. They plan instead to dewater the

Microcel™ column flotation froth with a screen-bowl centrifuge as done with the mechanical-cell froth in the existing plant.

Table 3. Projected Filter Performance for Dewatering Microcel™ Froth Slurry

<u>Feed Slurry</u>	<u>Spiral Conc/ Float Coal Ratio</u>	<u>Froth Slurry Coal Portion Only</u>			
		<u>Feed Slurry % Solids</u>	<u>Flocculant lb/ton</u>	<u>Capacity lb/hr/sq ft</u>	<u>Cake Moist, %</u>
<u>Top Feed Horizontal Belt Filter</u>					
Coarse Column Feed Froth:					
Layered Spiral Conc	0.6	9.8	0.03	50.4	35.5-38.5
Mixed Spiral Conc	0.6	9.8	0.03	48.8	41-43
No Coarse Material		9.8	0.08	53.0	34
No Coarse Material		18.1	0.17	57.6	43
Fine Column Feed Froth:					
Layered Spiral Conc	0.6	11.5	0.07	58.9	36.5-40.5
Mixed Spiral Conc	0.6	11.5	0.08	49.2	42-44
No Coarse Material		11.5	0.10	45.1	39
No Coarse Material		15.0	0.04	80.7	37
No Coarse Material		20.0	0.04	90.8	37
<u>Bottom Feed Drum Belt Filter</u>					
Coarse Column Feed Froth		9.8	0.08	32.2	34-35
Fine Column Feed Froth		11.5	0.07	42.6	35

Subtask 3.3 Dewatering Studies

This work, being performed by Virginia Tech, is aimed at developing a novel hydrophobic dewatering (HD) process for clean coal fines. In this process a hydrophobic substance is added to a coal-water slurry to displace water from the coal surface. The hydrophobic substance is then recovered for recycle to the process. Three coals will be tested including the product from near-term testing at the Lady Dunn plant (Subtask 3.2). Subtask 3.3 is further divided into five additional subtasks as follows:

- Subtask 3.3.1 - Project Planning
- Subtask 3.3.2 - Identification of Hydrophobic Substances
- Subtask 3.3.3 - Process Development
- Subtask 3.3.4 - Design/Operation of Continuous Bench-scale Unit
- Subtask 3.3.5 - Economic Analysis

During the previous quarter a detailed work plan was prepared and submitted to DOE under Subtask 3.3.1. This plan contained a description of the test plan, experimental procedures, analytical methods, and reporting guidelines for implementation and completion of the project. Previous work also identified suitable hydrophobic

substances as part of Subtask 3.3.2. Work this quarter focused on Subtasks 3.3.2, and 3.3.3 as discussed below.

Subtask 3.3.2 - Identification of Hydrophobic Substances

Subtask 3.3.2.1, Contact Angle Measurements - To identify suitable hydrophobic substances that can readily displace water from coal, contact angle measurements continued during this reporting quarter. Thermodynamically, when the contact angle, defined as the angle between the liquid and coal surface as measured through the liquid phase, becomes greater than 90° , the displacement of water by the hydrophobic substance on the coal surface becomes spontaneous. And, as the contact angle further increases, the hydrophobic substance performs better as a dewatering agent.

While a number of different contact angle measurement methods are available, this project will primarily utilize the "sessile drop method", in which a hydrophobic droplet is placed over a polished coal surface and the angle subtended by the liquid at the three phase contact is measured.

During this quarter, a Ramé-Hart High-Pressure Apparatus (Model 100-08/30) was obtained for contact angle measurements. This compact high-pressure chamber mounts directly onto the stage of the Ramé-Hart Contact Angle Goniometer, providing a means for determining contact angles at elevated pressures and/or temperatures. With this high-pressure apparatus, the contact angle of butane and other high-pressure substances, on coal, can be measured. Unfortunately, the sample stage holder broke during preliminary measurements and was returned for repairs. It is anticipated that contact angle measurements utilizing this apparatus will be completed during the next reporting quarter.

Subtask 3.3.2.2 , Determination of Free Energy Displacement - The three most suitable hydrocarbons will be used to determine the free energy of displacement for one of the coals to be utilized during the process development work. Work on this task will commence as soon as the identification of suitable hydrophobic substances has been completed.

Subtask 3.3.2.4, Direct Force Measurements - In order to validate the combining rule for long-range hydrophobic forces, the forces between dissimilar solids in water were measured. This work was carried out utilizing an atomic force microscope. For this work, spherical glass samples hydrophobized by ODTCS to an equilibrium contact angle of 109° and flat silica samples hydrophobized to various contact angles in the 80 - 109° range were used. Results from this work indicate that the combining rule is valid for hydrophobic forces, and that hydrophobic force is uniquely determined by contact angle. Further, for similar solids, the hydrophobic force is a function of the contact angles of the forces involved, while for dissimilar solids it is determined by the average contact angle involved.

Calculations of potential curves for the displacement of water on coal surfaces were also carried out. For the dewatering process to be kinetically possible, the potential curve for the interaction of coal-water-hexane must not have a barrier. Based on the results of the present work, it appears that the barrier disappears when, for the coal-water-hexane system, the contact angle of the coal is less than 87°. However since it is possible that the thickness of the water layer on coal is restricted, dewatering may occur at coal contact angles as low as 73°. In a similar manner, this critical contact angle of coal (for dewatering to be kinetically possible) can be calculated for any reagent, other than hexane, with a known contact angle.

Subtask 3.3.3 - Process Development

Under Subtask 3.3.3.2, a batch dewatering unit was designed and constructed to test this HD process during the last reporting quarter. The design incorporates the following:

- A high pressure cell to keep the hydrocarbons in a liquid form
- A variable speed mixer to provide adequate mixing
- A transparent window for visual observation
- A pressurized sampling vessel
- Continuous monitoring of temperature, pressure, and mixer speed

Shakedown testing of the HD process unit was conducted this quarter on Lady Dunn plant samples using butane as the hydrophobic substance. During initial testing, it was found that the inability to drain all of the water from the vessel resulted in remixing of water with the dewatered coal. To resolve this problem, a sampling assembly was constructed and installed on the cover of the high-pressure vessel.

Following this modification, preliminary tests resulted in coal moistures of 20-25%. These relatively high moistures were attributed to several factors including:

- The recovery of some water with the butane phase during sampling
- The freezing of moist air during the evaporation of the butane
- The use of oxidized coal

Additional changes made to the system at this time included:

- Modification of the sampling chamber allowing evacuation prior to sampling to reduce the effect of the moist air
- A decrease in the depth of the sampling tube to prevent recovery of liquid from the water phase
- An increase in the amount of butane used to increase the thickness of the butane phase

- The use of a fresh coal concentrate from Elkview (British Columbia, Canada)

Under these conditions, moisture contents of 0.9-1.6% were achieved indicating that the necessary modifications had been completed, and that the unit was ready for parametric testing.

TASK 4 ENGINEERING DEVELOPMENT OF ADVANCED FROTH FLOTATION

As described in the Subtask 2.1 report, Coal Selection Plan and Recommendations [15] and in previous quarterly reports [3, 4], six coals were identified as good candidate feedstocks for conversion into premium fuel and were selected for testing during Task 4. The six coals selected are described in Table 4.

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

Table 4. Test Coals Selected for Project

<u>Coal</u>	<u>Mine</u>	<u>State</u>	<u>HGI</u>	<u>Ash, %</u>	<u>Sulfur, %</u>
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

Task 4 activity during this reporting quarter involved work under Subtask 4.4, Bench-scale Testing and Process Scale-up.

Subtask 4.4 Bench-Scale Testing and Process Scale-Up

A draft of the Subtask 4.4 topical report containing the bench-scale flotation work and the toxic trace element reduction data was distributed to team members for comment on November 30, 1995. The report contains the flotation performance data from the 1-foot KenFlote™ and Microcel™ column testing and also the toxic trace element analyses and distributions for selected tests on each coal. It is anticipated that the final version will be issued during January, 1996.

TASK 6 ENGINEERING DEVELOPMENT OF SELECTIVE AGGLOMERATION

Task 6 activity during this reporting quarter focused on Subtask 6.4 CWF Formulation Studies, and Subtask 6.5 Bench-scale Testing and Process Scale-up.

Subtask 6.4 Coal-Water-Fuel Formulation Studies

The primary objective of Subtask 6.4 is to evaluate the formulation of coal-water-fuel (CWF) slurries from selective agglomeration products. The slurry feedstock, i.e., selective agglomeration products, used for this work are generated during Subtask 6.5, Selective Agglomeration Bench-scale Testing and Process Scale-up.

While much of this test work will evaluate the effect of various parameters on slurry quality, there are two other objectives for the Subtask 6.4 work. First, this test work will provide a comparison between similar slurries formulated from flotation and agglomeration products. This information will provide some insight into whether one process generates a product inherently more amenable to highly-loaded slurry formulation than the other process. Second, the Subtask 6.4 work will attempt to determine slurry quality guidelines for commercial production. To this end, determinations of required slurry coal loadings, stabilities, and viscosities will be carried out.

The Subtask 6.4 testing began in earnest during the previous (twelfth) quarter of this project. This initial work involved particle size distributions (PSD) characterization of Subtask 6.5 testing final products, i.e., product from the steam stripping circuit used to remove heptane from the recovered agglomerates. This previously reported [12] PSD characterization work was completed for the following coals:

- Taggart 1 (62-mesh topsize) - The Taggart coal utilized for all of the Subtask 4.3 and 4.4 testing, and the initial Subtask 6.5 start-up testing.
- Taggart 2 (62-mesh topsize) - The replacement Taggart coal which will be utilized for the remainder of project testing. While this coal comes from the same seam as the Taggart 1 coal, it is from a different mine.
- Sunnyside (150-mesh topsize) - This coal was used during Subtask 4.3 and 4.4 testing, and the initial Subtask 6.5 testing. This coal has since been replaced with the Hiawatha coal.
- Elkhorn No. 3 (100-mesh topsize) - This coal was used during Subtask 4.3 and 4.4 testing, and is also being used for Subtask 6.5 testing.
- Indiana VII (325-mesh topsize) - This coal was used during Subtask 4.3 and 4.4 testing, and is also being used for Subtask 6.5 testing.

It should be noted that based on Subtask 6.5 testing results, the Taggart 2 coal will require a finer grind to achieve the 1 lb/MBtu product ash specification set for this coal.

As such, no further slurry formulation work will be carried out with this 62-mesh tosize Taggart 2 coal agglomeration product.

Subtask 6.4 testing continued during this reporting quarter with the formulation of slurries utilizing Elkhorn No. 3 coal product from the 25 lb/hr selective agglomeration bench scale unit. Table 5 presents the agglomeration circuit feed and steam stripper product PSD for the Elkhorn No. 3 coal. As before, the steam stripper product material was utilized for the slurry work.

Table 5. Elkhorn No. 3 Coal Selective Agglomeration Feed and Product PSD's

<u>Microns</u>	<u>Feed</u>	<u>Product</u>
	<u>100 mesh</u>	
	<u>Cumulative Percent Passing</u>	
150	100.00	100.00
106	96.86	96.85
75	84.34	85.91
53	70.64	75.55
45	63.73	64.95
38	59.58	60.64
30	50.63	51.62
20	35.84	35.55
15	27.47	28.56
10	18.8	20.09
8	15.78	16.31
6	12.11	12.45
4	8.07	8.17
3	5.73	5.68
2	3.21	3.27
1	0.44	0.67
MMD*	39.43	37.92

* Mass Mean Diameter = Sum of size interval midpoint (microns) times weight fraction of particles in that interval (percent)

Testing was carried out to characterize the formulation of slurries from the "as-received" Elkhorn No. 3 coal agglomeration circuit product. Additional testing was also carried out to provide a comparison between the formulation of slurries from advanced flotation products and from selective agglomeration products. This was accomplished by repeating the formulation of various slurries carried out during Subtask 4.3 testing. As such, slurries were formulated from the following Elkhorn No. 3 coal feedstocks:

- As-received agglomeration product using 0.5 and 1.0% A-23 dispersant. Coal loadings for these slurries were in the 57-60% by weight range.

These results provide a baseline with which to gauge the quality of other slurries formulated from this coal and also provide a comparison with slurries formulated during Subtask 4.3 testing.

- A blend of 70% as-received material and 30% material reground for 30 minutes in the attritor mill, using 0.5% A-23 dispersant. This blend is referred to as the 70/30 regrind blend. Coal loadings for these slurries were in the 58-61% by weight range.

The results for these slurries are shown in Figure 4, which indicates Slurry coal loading vs slurry viscosity. As can be seen from the data in Figure 4, scatter in the data for both sets of slurries formulated from 100% agglomeration product occurred. As such, while it appears that slurries formulated from the 70/30 regrind blend achieved slightly lower viscosities, at the same coal loadings, than the slurries formulated from the 100% as-received agglomeration product, some uncertainty exists.

Also, while no Flocon stabilizer was utilized in the formulation of these slurries, it is expected that its use would result in increased slurry viscosity as observed for all previous slurry formulation studies.

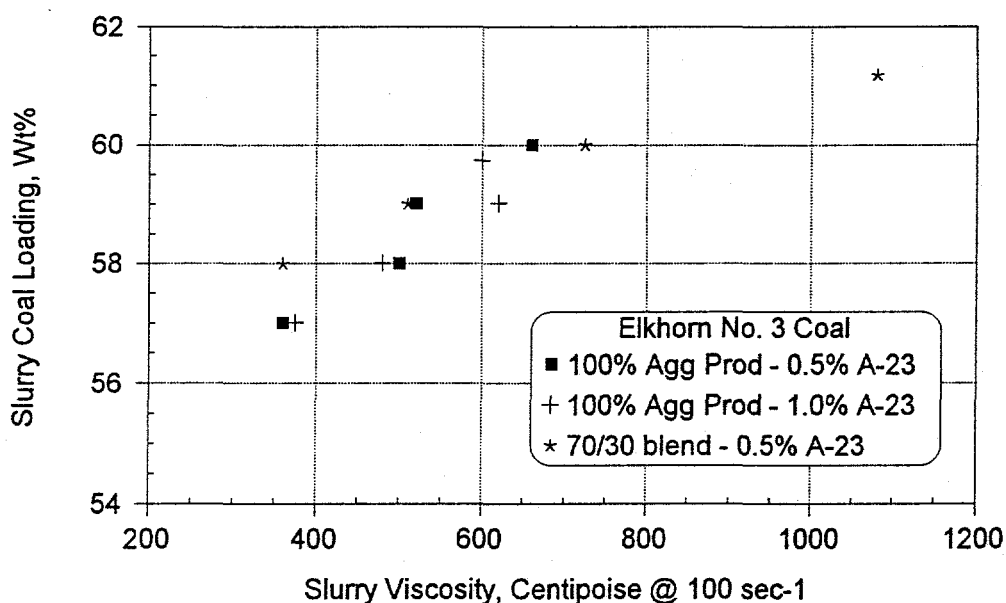


Figure 4. Elkhorn No. 3 Coal Agglomeration Product Slurry Formulation Results

Figure 5 provides a comparison of slurries formulated with selective agglomeration and flotation products.

As this data indicates, while the trends observed as a result of PSD manipulation may be similar, it appears that lower viscosities, at similar coal loadings, are achieved with agglomerated product than with flotation product. It is believed that this may be due to a number of reasons including:

- The presence of aggregates of particles in the steam stripped agglomeration circuit product, effectively coarsening the PSD
- A steam stripping process effect on the surface properties of the coal
- The presence of residual heptane in the agglomeration circuit product

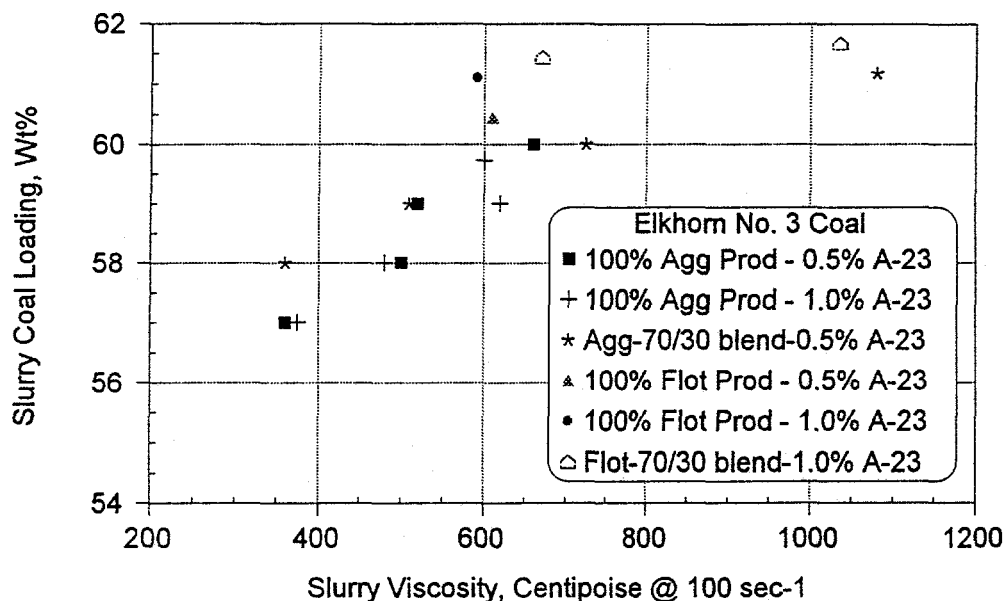


Figure 5. Elkhorn No. 3 Coal Agglomeration and Flotation Slurry Results

All of the slurries formulated with this Elkhorn No. 3 coal had poor stabilities with a rating of "1" after overnight storage. While this rating of "1" indicates that at least 25% of the slurry volume was occupied by a very hard pack sediment, for all of these slurries, at least 65% was very hard packed.

In addition to this test work, discussions were held with Dr. John Doohar of Adelphi University, a consultant being used for the Subtask 6.4 work. Based upon his recommendations, future testing will incorporate a revised viscosity determination procedure in which more time is provided for the slurry to achieve equilibrium. In addition, evaluation of slurries formulated at a pH of 10, rather than the natural pH of 7, used to date, will be carried out.

Subtask 6.5 Bench-Scale Testing and Process Scale-up

During previous testing utilizing the 25 lb/hr bench-scale unit, evaluation of the Taggart 1, Sunnyside, and Elkhorn No. 3 coals were completed. It should be noted, that the Taggart 1 and Sunnyside coals have since been replaced with the Taggart 2 and Hiawatha coals, respectively, and as such will not be tested further. While other previous testing evaluated the Indiana VII coal, indicating that product ash specifications could be met at the selected 325-mesh topsize grind, additional testing

with the Indiana VII coal may be carried out later in this test program. Previous work also included preliminary evaluation of the Taggart 2 coal.

Selective agglomeration bench-scale testing this quarter focused on the Winifrede and Hiawatha coals, with some additional work carried out with the Taggart 2 coal.

Winifrede Coal

To achieve the desired particle size distribution (PSD), the Winifrede coal was initially ground in the 4' x 4' ball mill in open circuit. The product from this grind is then normally subjected to two or three passes through the Drais fine grinding mill to achieve the liberation required to meet the desired product ash specification.

However, during the first two passes through the Drais mill, a cooling water leak resulted in a drastic reduction in solids concentration from approximately 40 to 18% solids. As such, the dilute slurry resulted in a less efficient grind, and therefore, three additional passes through the Drais mill were needed. These last three grinds were carried out at high feed rates to prevent overheating of the mill since the cooling water had to be turned off to avoid additional slurry dilution.

PSD's for the first, second, and fifth Drais mill pass products are shown in Table 6, along with the product ash content from batch agglomeration tests carried out with each. As can be seen from this data, the product ash target of 2.0 lb ash/MBtu was achieved with the fifth pass through the Drais mill.

Sixteen continuous agglomeration tests utilizing the Winifrede coal, ground as shown in Table 6 (fifth pass through Drais Mill), were carried out during this quarter. This test work utilized both fresh commercial grade heptane and recycled commercial grade heptane. Test conditions and results for these sixteen tests are shown in Appendix B.

The following conditions were held constant for all of these tests:

- Feed PSD as discussed above
- 2.4-inch (4-blade) high-shear impeller

As for all previous testing to date, product and tailings ash values, as well as performance calculations, utilized composited samples from froth skimming simulation. This procedure involves reporting the material which floats in the tailings sample as part of the product.

The primary variables changed during the completion of these sixteen agglomeration tests were:

- Feed slurry solids concentration
- Coal feed rate

- Heptane type
- Heptane concentration
- Asphalt addition
- High shear tip speed
- Low-shear tip speed
- Low-shear impeller configuration
- Low-shear residence time
- Vibrating screen inclination
- Vibrating screen spray water rate

Table 6. Winifrede Coal Grinding PSD's and Batch Test Product Ash Values

<u>Microns</u>	<u>Drais Mill Grinding Pass Products</u>		
	<u>First</u>	<u>Second</u>	<u>Fifth</u>
	<u>Cumulative Percent Passing</u>		
75	100.00	100.00	100.00
53	99.84	100.00	100.00
45	99.18	100.00	100.00
38	98.69	100.00	100.00
30	89.01	96.61	99.77
20	77.07	82.42	91.99
15	66.04	75.38	82.22
10	50.70	63.49	78.03
8	42.04	55.64	75.44
6	31.93	44.85	65.90
4	20.45	28.90	48.25
3	14.14	20.39	34.96
2	7.53	10.83	19.59
1	<u>1.45</u>	<u>2.06</u>	<u>4.29</u>
MMD*	13.28	10.36	7.14
Batch Test Prod., <u>lb ash/MBtu</u>	2.45	2.11	1.80

* Mass Mean Diameter = Sum of size interval midpoint (microns) times weight fraction of particles in that interval (percent)

The results for all of these tests are presented in Figure 6 which shows agglomeration Btu Recovery vs Product ash content.

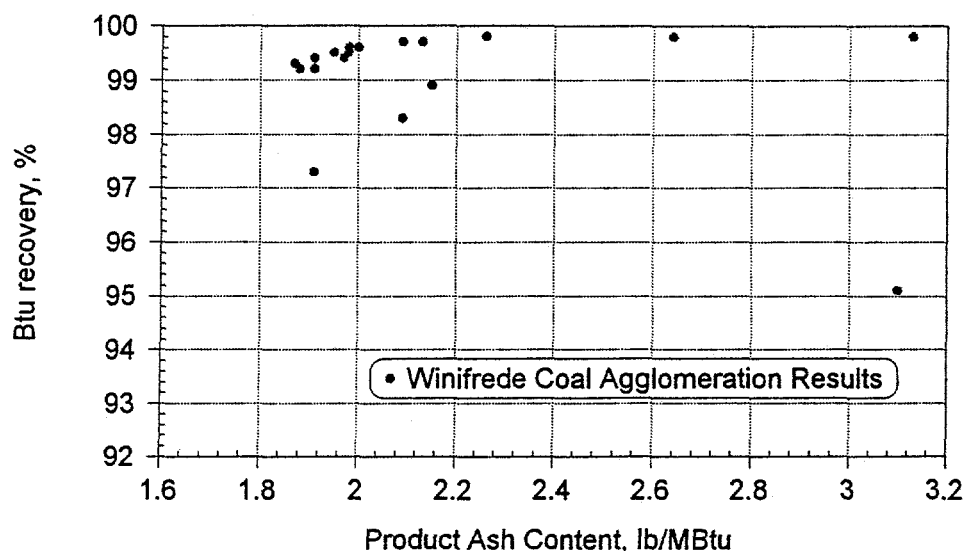


Figure 6. Winifrede Coal Continuous Agglomeration Results

As can be seen from this data, the 2 lb ash/MBtu specification was met for most of the tests completed, indicating that the 7.14 micron mass mean diameter grind utilized, provides sufficient mineral-matter liberation. This data also indicates that very High Btu recoveries, i.e., >97% were achieved for all but one (W1A13) of the tests carried out. While corresponding tailings ash values for all of these tests were in the 47-89% range, most were in the 78-89% range.

No difference in the operation of the continuous agglomeration unit was observed when fresh commercial grade heptane was used (W1A1 and W1A2), as compared to recycled commercial grade heptane.

In virtually all of these tests, only a marginal inversion was achieved during the high shear agglomeration unit operation. This is attributed to the extreme fineness of the grind and the inability of the high-shear reactor to provide higher energy inputs. As a general trend, higher high-shear impeller tip speeds and lower volumetric feed rates, i.e., longer residence times, provided improved inversion. It should be noted, however, that very clear inversions were never achieved. In test W1A4, asphalt was used to improve the inversion without success.

It was found, during this testing, that even though a good inversion could not be achieved during high shear, agglomerate growth during low shear was sufficient to afford good recovery during screening. In fact, as with the majority of previous Subtask 6.5 testing, when sufficient heptane was utilized (55-60% on a dry ash free coal basis), growth during low shear was easily achieved and in fact proved difficult to control.

Generally, when sufficient heptane was used, tests completed with a low-shear impeller tip speed of 4.8 m/s resulted in continuous agglomerate growth, eventually plugging the low-shear discharge. However, when an 8 m/s low-shear impeller tip speed was used,

agglomerate size usually cycled from the <0.5 mm range to the 2-3 mm range. This trend is similar to the bulk of testing completed previously.

Also as observed previously, it was found that when well formed agglomerates were produced, lower product ash contents were achieved. This is due primarily to better drainage of mineral matter bearing process water. This trend is evident in the results for tests W1A8 through W1A10 in which separate product samples were taken at different agglomerate sizes. For each of these tests, as agglomerate size increased, product ash decreased.

For one test carried out with the Winifrede coal this quarter (W1A11), production of well formed 2.5 mm agglomerates was achieved and maintained at steady state, as indicated by the duplicate samples taken approximately one hour apart. For this test, the coal feed rate was 12.5 lb/hr and the solids concentration approximately 7%. These conditions provided 2 minutes of high-shear residence time and 4.6 minutes of low-shear residence time.

Given that this Winifrede coal will not be used during 2 t/hr PDU testing, no additional test work with this coal in the 25 lb/hr bench-scale agglomeration test unit is planned.

Hiawatha Coal

Fourteen continuous agglomeration test were completed with the Hiawatha coal during this reporting quarter. This coal was closed circuit ground in the 4' x 4' ball mill with a 150-mesh screen. This grind size was chosen based on an estimate of liberation requirements to achieve the desired 2.0 lb/MBtu product ash content.

Due to the limited quantity of this coal available, only about 1200 pounds of this coal was ground. An additional 1200 pounds of this coal remains for additional testing.

Table 7 presents the agglomeration feed particle size distribution (PSD) for the Hiawatha coal, along with the product ash content from batch agglomeration tests carried out with the ground product. As can be seen from this data, the estimated product ash target of 2.6 to 2.8% was achieved for the Hiawatha coal at this grind size.

This test work investigated a number of agglomeration operating variables, but has focused primarily on the evaluation of the low-shear unit operation. In particular, the discharge from the low-shear vessel has been of primary concern due to the plugging problems experienced throughout Subtask 6.5 testing.

In an effort to eliminate plugging of the low-shear vessel, two additional sets of discharge ports were installed in the vessel. One set are 2-inches in diameter, and located to allow an overflow discharge. The second set are 1-inch in diameter, and located at the same elevation as the impellers, utilizing an upflowing discharge to maintain the correct operating level in the vessel.

**Table 7. Hiawatha Coal Agglomeration Feed PSD and
Batch Test Product Ash**

<u>Microns</u>	<u>Cumulative Percent Passing</u>
106	100.00
75	96.85
53	88.68
45	77.87
38	74.21
30	63.39
20	45.53
15	35.12
10	23.83
8	19.84
6	15.44
4	10.07
3	7.17
2	4.12
1	0.86
MMD*	27.44
<u>Batch Test Product Ash, %</u>	2.58
* Mass Mean Diameter = Sum of size interval midpoint (microns) times weight fraction of particles in that interval (percent)	

Evaluation of these different discharge arrangements has resulted in the following observations:

- While the use of the 2-inch overflow discharge works well when the vessel is operated half-full, excessive splashing appears to interrupt the desired mixing pattern. As such, no agglomerate growth was observed.
- The 2-inch overflow discharge, when utilized with the vessel full, provides good agglomerate discharge. However, it was found that the lower half of the vessel packs full of agglomerates. As a result, the solids discharge reduces over time, resulting in a buildup of solids and eventual plugging of the vessel.
- Use of the 1-inch discharge port located at the impeller height worked well when the vessel was operated half-full. At these conditions, steady-state operation appears to have been achieved. However, monosized agglomerates were not formed. Instead, agglomerates typically ranged from 0.5-3 mm in size and were oval in shape rather than round.
- Use of the 1-inch discharge port when the vessel was full resulted in plugging of the lower half of the vessel, as was the case for the 2-inch discharge port.

Based on these observations, it appears that the transfer of agglomerates from the lower to upper section of the low-shear vessel is a bottleneck. Attempts will be made to improve this flow during future testing by increasing the opening in, and/or completely removing, the horizontal baffle which separates the two mixing zones.

It should be noted that the location of the discharge ports at the impeller height has resulted in improved agglomerate discharge, confirming the low shear PDU design.

Due to the limited data available for this Hiawatha coal, no ash/Btu relationship has been developed. As such, lb ash/MBtu and percent BTU recovery results are not available at this time and will be reported in a future quarterly report. Operating conditions for these fourteen tests, along with product ash, tailings ash, and yield values, however, are shown in Appendix C.

As can be seen in Appendix C, product ash values for these tests ranged from approximately 2.1-3.2%, indicating that sufficient liberation is achieved at the 150-mesh topsize grind to meet the desired 2 lb/MBtu product ash specification. As such, during future testing of this coal, a 100-mesh topsize grind will be evaluated.

Tailings ash values for all of these tests were consistently in the 82-87% range, indicating that Btu recoveries will fall in the 95-99% range.

Taggart 2 Coal

During previous testing, the Taggart 2 coal was closed-circuit ground to a 62-mesh topsize for evaluation in the continuous agglomeration bench-scale unit. Results from the work carried out utilizing this feedstock indicated that a product ash content of 1.3-1.5 lb ash/MBtu was achieved. As such, the Taggart 2 coal was closed-circuit ground to a 100-mesh topsize this reporting quarter in an attempt to achieve the target product ash content of 1 lb ash/MBtu.

Two agglomeration tests were completed utilizing this feedstock. These two tests resulted in product ash contents of 1.42 and 1.44 lb ash/MBtu, indicating no additional liberation for this finer grind. As such, additional liberation studies will be carried out with the Taggart 2 coal during future testing to determine the grind required to insure that a product ash content of 1 lb ash/MBtu can be met in the 25 lb/hr continuous selective agglomeration bench-scale unit.

Results for all continuous agglomeration testing completed to date with the Taggart 2 coal are presented in Appendix D.

Continuous Steam Stripper Testing

No structured Steam-stripping tests for heptane recovery were carried out during November. Steam stripping of agglomerated coal continued on a regular basis,

however, to allow for coal disposal. Random analysis of steam stripper products confirmed that heptane concentrations in the 5000 ppm (dry coal basis) range were produced.

TASK 7 PDU SELECTIVE AGGLOMERATION MODULE DETAILED DESIGN

Module Detailed Design

Work was essentially completed on the detailed design of the PDU selective agglomeration module during this thirteenth reporting quarter. Sufficient progress was made on the design to allow for the issuing of the construction bid package during this quarter.

Selective agglomeration module detailed design work remaining involves the preparation and issuing of the final task report.

Design Overview

The following sections of this report discuss design features of the plant unit operations with emphasis on the selective agglomeration module. A block flow diagram of the PDU selective agglomeration module is shown in Figure 7.

Coal Grinding and Dewatering

It is not anticipated that any major changes in either the grinding or dewatering circuits will be required for the switch over from the flotation to agglomeration process module. As such, the agglomeration process will use slurry ground by the installed equipment.

The ground product will be pumped to the current Microcel™ flotation column feed tank. The product stream from this tank will be diluted to the desired solids concentration and sent to one of two ground slurry storage tanks. From these storage tanks, the slurry will be metered to the agglomeration process. Once processed, the final steam stripped product and the process tailings will be dewatered utilizing the existing equipment which consists of one vacuum drum filter and two filter presses for the product stream, and two filter presses for the tailings stream.

High Shear

High-shear agglomeration will be carried out in a circuit consisting of two high-shear reactors. These reactors will be sized to provide 0.5 and 1 minute residence times respectively, and each will be powered by a variable speed drive that can achieve up to 18 m/s impeller tip speeds. In this manner, high shear residence times from 0.5 to 1.5 minutes can be achieved by operating either unit individually, or both together in series.

Heptane will be metered to the agglomeration process as required, currently anticipated not to exceed 40% of the coal feed rate. The ability to add asphalt to the high-shear circuit will also be provided for testing of the Indiana VII coal.

Low Shear

Low-shear agglomeration will be carried out in a single vessel divided into two sections via a horizontal baffle. Each section will provide 2.5 minutes of residence time for a maximum of five minutes. The discharge piping will be arranged such that one or both sections can be utilized. The low shear operation will be powered by a single variable speed agitator powering one impeller for each section of the vessel. The impellers will be of the radial-flow type and the drive unit of sufficient HP to achieve tip speeds in the 3-5 m/s range.

Vibrating Screen and Froth Skimmer

The vibrating screen to be used to recover agglomerates from the low shear product will be a 48-mesh dewatering screen approximately 2-feet wide by 6-feet long. Sufficient wash water spray will be provided to insure replacement of mineral matter bearing process water with fresh water.

The vibrating screen underflow (tailings) will then be processed through a froth skimmer. This skimmer will provide approximately 3-minutes of residence time for any carbonaceous material to float. If necessary, nitrogen will be bubbled through the skimmer to help the material float. A continuously rotating paddle will then scrape the floating material to a launder from where it will be combined with the screen product.

Steam Stripper

The combined screen and froth skimmer product will be diluted with hot water to approximately 25% solids. This feed will then be treated in the first stage steam stripper which will provide a 5-minute residence time at 94°C and ambient pressure. The heat source for this stirred vessel will be the vapor product (steam and heptane) from the second stage stripper. This first stage stripper has two primary functions. First it will remove the bulk of the heptane present (about 99%), and second, it will generate a handleable and pumpable product.

This first-stage stripper product will then be pumped to the second-stage stripper which will provide about 10-minutes of residence time at 115-120°C and 15-20 psig pressure. The product from this second stage stripper will then be cooled and sent to the dewatering circuit.

Condenser and Gravity Separator

Vapors from the first stage stripper will contain steam and recovered heptane and be condensed in a two-stage process. Initial condensation and some cooling will be carried out by an air cooler, followed by a shell and tube heat exchanger to provide the necessary sub-cooling. The condensed liquids will then be sent to the gravity separator from which both the heptane and the process water will be recycled to the process.

Equipment Specifications

Work was also completed on the development of equipment specifications for the bulk of the major capital equipment items slated for purchase. As such, the purchasing of major capital items began late in this quarter with the issuing of a number of material requisitions. Plant equipment for which the finalization of specifications remains to be completed includes primarily instrumentation.

Miscellaneous Tasks

Work was initiated to bring the existing GC analyzer on line during this reporting quarter. This will allow trace heptane determinations during the latter part of Subtask 6.5, and throughout Subtasks 9.2 and 9.3, to be completed in-house. Huffman Laboratories was retained as a consultant for this work.

As of the end of this quarter, all required parts (detector, column, etc.) have been obtained, with their installation complete. All other necessary supplies (gases, chemicals, etc.) have also been received. The GC analyzer has been started up and some preliminary testing initiated. Preliminary results indicate that the GC is working well. Work remaining under this task involves the programming of the integrator and final procedure development to allow routine operation of the analyzer throughout the remainder of this project.

TASK 8 PDU AND ADVANCED COLUMN FLOTATION MODULE

The Task 8 work completed this reporting quarter focused on Subtask 8.1 Coal Selection and Procurement and Subtask 8.3 PDU Module Shakedown and Test Plan, as discussed in the following sections of this report.

Subtask 8.1 Coal Selection and Procurement

Four rail cars of washed Indiana VII compliance coal were received in mid November and unloaded at a coal yard in Denver. The coal was ordered for early delivery in order to avoid unloading problems which might arise from any freezing which might

occur while in transit. The Indiana VII coal will be trucked as needed to Ralston Development Company near Amax R&D for further crushing and temporary storage in covered bunkers.

Subtask 8.3 PDU and Advanced Flotation Module Shakedown and Test Plan

Startup and shakedown of the PDU flotation module was completed during the last quarter of 1995. Though some minor operating difficulties were encountered, corrective actions resulted in a fully functional PDU flotation module. Physical and mechanical improvements resulted in the elimination of process bottlenecks which allowed Entech personnel to achieve the desired PDU feed rate of 4,200 lb/hr. The operation of the PDU flotation unit was somewhat limited during December due to the holidays and several Entech personnel on vacation. The test plan was also completed during the quarter and submitted to DOE for review and approval.

PDU Flotation Module Shakedown and Operation

Work related to the PDU module shakedown was completed during the quarter. Because the operation of individual equipment items were checked during the previous quarter, efforts were directed to overall startup and operation of the PDU flotation module. The following is a chronology of PDU startup and shakedown efforts during the last quarter of 1995:

October 2, 1995 - Operation during the previous month indicated that the existing three inch Microcel clean coal transfer line was too small to convey 4,000 lb/hr (dry) of froth to the product sumps. The small line size restricted the flow which resulted in froth overflowing the Microcel launder. Precision Mechanical, Inc. replaced the first 20 feet of the 3 inch line with an 8 inch PVC line and breather vent.

October 4, 1995 - During a material calibration of the Technetics weigh belt feeder, the primary ball mill discharge pump bound up and ceased operation. Entech personnel disassembled the pump and found a 1" ball lodged in the stator. The ball was removed and the pump re-assembled.

October 5, 1995 - During a material calibration of the Technetics weigh belt feeder, the Microcel flotation column was operated to determine the operability of the new 8 inch PVC clean coal transfer pipe. At a feed rate of approximately 4,300 lb/hr, some product "burping" was noticed in the transfer pipe breather vent. As a result, it was concluded that the 8" x 3" restriction is still too small for transporting 4,000 lb/hr of clean coal. Precision Mechanical, Inc. extended the 8 inch line to the product samplers.

October 11, 1995 - Precision Mechanical, Inc. completed the extension of the 8 inch PVC clean coal transfer pipe.

October 12, 1995 - Operated the PDU Flotation Module at the following conditions:

- Feed Rate: 4,300 lb/hr
- Collector: 0.50 lb/ton*
- MIBC: 1.00 lb/ton*
- Microcel Air: 50 CFM
- Microcel Level SP: 8 inches
- Microcel Wash H₂O: 0 gpm

Under these conditions, froth did not overflow into Microcel launder but remained approximately 2 inches lower than the overflow weir. As a result, froth accumulated inside Microcel unit and discharged through tailings valve.

- * the lack of froth overflow was attributed to a low Microcel level setting but was later determined to be the result of low reagent dosage - see October 26, 1995.

October 24, 1995 - Operated the PDU Flotation Module to generate slurry for calibration of the nuclear density gauge (DIC-203). While running at a rate of 4,000 lb/hr, the screw conveyor plugged at its discharge (primary mill inlet). Mill feed pipe size to be increased from 4 inch to 6 inch diameter.

October 25, 1995 - Cleared screw conveyor and increased mill feed pipe from 4 inch to 6 inch diameter.

October 26, 1995 - Operated the PDU Flotation Module at the following conditions:

- Feed Rate: 4,300 lb/hr
- Collector: 0.50 lb/ton*
- MIBC: 1.00 lb/ton*
- Microcel Air: 50 CFM
- Microcel Level SP: 60 inches
- Microcel Wash H₂O: 0 gpm

Again, under these conditions, froth did not overflow into Microcel launder but remained approximately 2 inches lower than the overflow weir. Investigation revealed that the lack of froth overflow was the result of low reagent dosage. Specifically, the MIBC dosage was found to be 0.33 lb/ton instead of 1.0 lb/ton.

October 31, 1995 (morning) - Entech staff recalibrated Collector and MIBC pumps.

October 31, 1995 (afternoon) - Operated the PDU Flotation Module at the following conditions:

- Feed Rate: 4,000 lb/hr

- Collector: 0.50 lb/ton
- MIBC: 1.00 lb/ton
- Microcel Air: 50 CFM
- Microcel Level SP: 60 inches
- Microcel Wash H₂O: 0 gpm

The increase in reagent dosage corrected the problem with the Microcel overflow. The process operated for approximately 1/2 hour before the Microcel launder started to overflow onto floor. The 8" x 3" restrictions at the clean coal samplers are causing the backup of froth into the launder. Precision Mechanical, Inc. to extend the 8 inch PVC transfer line directly into each product sump.

November 1, 1995 - Operated the PDU Flotation Module at the following conditions:

- Feed Rate: 2,000 lb/hr
- Primary Water: 10 GPM
- Cyclone Water: 10 GPM
- Air Rate: 50 CFM
- Spray Water: 0 GPM
- Launder Water: 30 GPM
- Collector: 0.50 lb/ton
- Frother: 1.00 lb/ton

Operated PDU for about 2 hours (9:45 AM to 11:45 AM). Had to shut down due to raw coal plug at discharge of screw conveyor. New 8-inch line and wide angle launder sprays working well.

November 2, 1995 - Operated the PDU Flotation Module at the following conditions:

- Feed Rate: 2,000 lb/hr
- Primary Water: 13 GPM
- Cyclone Water: 10 GPM
- Air Rate: 50 CFM
- Spray Water: 0 GPM
- Launder Water: 30 GPM
- Collector: 0.50 lb/ton (5cc/min)
- Frother: 1.00 lb/ton (10 cc/min)

Operated PDU for 2.75 hours (10:05 AM to 12:50 PM). Had to shut down due to lack of filter capacity. Also found that previous PDU runs were at 50% of the

desired collector and frother dosages. The reduction in reagent dosage was found to be the result of back pressure in the Microcel. Both Microcel reagent pumps were recalibrated.

November 6, 1995 - Operated the PDU Flotation Module at the following conditions:

- Feed Rate: 2,000 lb/hr
- Primary Water: 10 GPM
- Cyclone Water: 10 GPM
- Air Rate: 50 CFM
- Microcel Level: 80 inches
- Spray Water: 0 GPM
- Launder Water: 30 GPM
- Collector: 0.50 lb/ton (5 cc/min)
- Frother: 1.00 lb/ton (10 cc/min)

Operated PDU for about 1 hour (2:10 PM to 3:10 PM) but had to shut down due to lack of filter capacity.

November 8, 1995 - Tried to run PDU at 2,000 lb/hr but had to shut down after 20 minutes due to plug at discharge of screw conveyor. Raw coal seems to be somewhat moist (>7%). Increased water flow to the primary ball mill will be used for future operations.

November 8, 1995 - WesTech filter representative, John Smith, determined drum filter capacity to be 2,000 lb/hr at 11% feed solids and 1,000 lb/hr at 4.5% feed solids. Found that the launder water being used to push the Microcel froth down the discharge pipe dilutes the froth to a point where the drum filter loses capacity. Reducing or eliminating the launder water should increase filter capacity. Launder water usage may be reduced by using the 300-D-01 sump located adjacent to the Microcel as the clean coal sump.

November 9, 1995 - Operated the PDU Flotation Module at the following conditions:

- Feed Rate: 2,000 lb/hr
- Primary Water: 13 GPM
- Cyclone Water: 10 GPM
- Air Rate: 50 CFM
- Microcel Level: 75 inches
- Spray Water: 40 GPM
- Launder Water: 30 GPM

- Collector: 0.50 lb/ton (5 cc/min)
- Frother: 1.00 lb/ton (10 cc/min)

Operated PDU for about 2 hours (10:25 AM to 12:25 PM) but had to shut down due to lack of filter capacity.

November 13, 1995 - Relocated the Microcel clean coal discharge line from the column launder to the transfer sump (300-D-01) located adjacent to the unit. The close proximity of the sump results in a vertical drop of the discharge line. Not only should this help alleviate any froth backup in the Microcel launder but the expected reduction in launder / push water should increase the product solids concentration and improve the overall PDU filtering capacity.

November 14, 1995 - Tried to run PDU today at 2,000 lb/hr but had to shut down after 20 minutes due to plug at discharge of screw conveyor. The primary ball mill water addition point was moved from the base of the mill feed chute to the top. In addition, the water discharge angle was changed from a horizontal position to a downward vertical position. The downward flow of water should keep the chute clean and improve the flow of coal.

November 15, 1995 - Operated the PDU Flotation Module at the following conditions:

- Feed Rate: 2,000 lb/hr
- Primary Water: 13 GPM
- Cyclone Water: 10 GPM
- Air Rate: 50 CFM
- Microcel Level: 75 inches
- Spray Water: 40 GPM
- Launder Water: 0 GPM
- Collector: 0.50 lb/ton (5 cc/min)
- Frother: 1.00 lb/ton (10 cc/min)

Operated PDU for 8 hours (7:50 AM to 3:51 PM). Everything ran very well. Relocation of primary ball mill push water to top of screw conveyor seems to have eliminated the plugging problem. Did have trouble with the density gauge. Density reading cycles on a regular basis from 0% to 20% solids. These readings are quite unlikely since the feed rate and water addition rates are basically constant. Winn Marion to troubleshoot unit.

November 20, 1995 - Installed WesTech filtrate bypass line. The high solids concentration of the WesTech drum filter (1% solids) contaminated the clean filtrate from the Netzsch and EIMCO pressure filters. As a result, Entech personnel routed the WesTech filtrate in a fashion which allows the solids to be removed in either the

Netzsch or EIMCO filters before discharging the clean filtrate to the filtrate sump. The improved filtrate quality permits this stream to be discharged directly to the clarified water sump.

November 28, 1995 - Operated the PDU Flotation Module at the following conditions:

- Feed Rate: 2,500 lb/hr
- Primary Water: 13 GPM
- Cyclone Water: 10 GPM
- Air Rate: 50 CFM
- Microcel Level: 75 inches
- Spray Water: 42 GPM
- Launder Water: 0 GPM
- Collector: 0.50 lb/ton (5 cc/min)
- Frother: 1.00 lb/ton (10 cc/min)

Operated PDU at the increased feed rate for about 4 hours (11:15 AM to 3:10 PM). Everything ran very well.

November 29, 1995 - Operated the PDU Flotation Module at the following conditions:

- Feed Rate: 3,000 lb/hr
- Primary Water: 15 GPM
- Cyclone Water: 20 GPM
- Air Rate: 50 CFM
- Microcel Level: 75 inches
- Spray Water: 42 GPM
- Launder Water: 0 GPM
- Collector: 0.50 lb/ton (5 cc/min)
- Frother: 1.00 lb/ton (10 cc/min)

Operated PDU at the increased feed rate for almost 3 hours (12:23 PM to 3:10 PM). Everything ran very well.

November 30, 1995 - Started operation of PDU at 3,500 lb/hr. However, Area 200 technician discovered frother pump had been operating for an extended period of time and overdosed the Microcel with MIBC. The flotation column was drained to the water treatment plant.

November 30, 1995 - Operated the PDU Flotation Module at the following conditions:

- Feed Rate: 3,000 lb/hr
- Primary Water: 15 GPM
- Cyclone Water: 20 GPM
- Air Rate: 50 CFM
- Microcel Level: 75 inches
- Spray Water: 42 GPM
- Launder Water: 0 GPM
- Collector: 0.50 lb/ton (5 cc/min)
- Frother: 1.00 lb/ton (10 cc/min)

Started operation of PDU at 3,500 lb/hr but had to shut down momentarily due to excessive frothing in the new clean coal transfer sump. The high level of froth was most likely due to excess MIBC in the Microcel. The feed rate was reduced to 3,000 lb/hr. The flotation module was operated for about 1 hour (2:15 PM to 3:20 PM). Except for some frothing in various PDU sumps, everything ran well.

December 4, 1995 - Operated the PDU Flotation Module at the following conditions:

- Feed Rate: 3,500 lb/hr
- Primary Water: 15 GPM
- Cyclone Water: 30 GPM
- Air Rate: 50 CFM
- Spray Water: 35 GPM
- Launder Water: 0 GPM
- Collector: 0.50 lb/ton (8 cc/min)
- Frother: 1.00 lb/ton (16 cc/min)
- Microcel Level: 75 inches

Operated PDU for about 3 hours (12:20 PM to 3:17 PM).

December 5, 1995 - Tried to run coal today at 4,200 lb/hr but had to shut down after 1 hour due to a lack of pumping capacity with the ground product pump (100-G-04). The added coal volume caused the ground product sump to overflow while the pump operated at 100% of maximum speed. Entech technicians increased the ground product pump speed by changing the motor and pump sheaves. The existing 5 hp motor was replaced with a 7.5 hp motor in order to overcome additional line friction loss.

December 11, 1995 - Operated the PDU Flotation Module at the following conditions:

- Feed Rate: 4,200 lb/hr
- Primary Water: 20 GPM
- Cyclone Water: 30 GPM
- Air Rate: 50 CFM
- Spray Water: 72 GPM
- Launder Water: 0 GPM
- Collector: 0.50 lb/ton (10 cc/min)
- Frother: 1.00 lb/ton (20 cc/min)
- Microcel Level: 75 inches

Operated PDU for about 6 hours (8:59 AM to 3:48 PM). This was the first time that the target feed rate of 4,200 lb/hr was achieved.

December 14, 1995 - Operated the PDU Flotation Module at the following conditions in order to generate slurry for calibration of the nuclear gauge:

- Feed Rate: 4,200 lb/hr
- Primary Water: 20 GPM
- Cyclone Water: 20 GPM
- Air Rate: 52 CFM
- Spray Water: 70 GPM
- Launder Water: 0 GPM
- Collector: 0.50 lb/ton (10 cc/min)
- Frother: 1.00 lb/ton (20 cc/min)
- Microcel Level: 75 inches

Operated PDU for about 1-3/4 hours.

December 21, 1995 - Operated the PDU Flotation Module at the following conditions in order to generate slurry for calibration of the nuclear gauge:

- Feed Rate: 4,200 lb/hr
- Primary Water: 20 GPM
- Cyclone Water: 20 GPM
- Air Rate: 52 CFM
- Spray Water: 70 GPM
- Launder Water: 0 GPM
- Collector: 0.50 lb/ton (10 cc/min)

- Frother: 1.00 lb/ton (20 cc/min)
- Microcel Level: 75 inches

Operated PDU for about 3-1/2 hours (7:35 AM to 11:10 AM). The Netzsch fine grinding mill was bypassed due to a plug at the mill outlet. The increased size consist resulted in a noticeable improvement in the capacity and moisture of the WesTech drum filter.

PDU Flotation Module Plant Improvements

Operation of the PDU Flotation Module during the quarter indicated that some plant changes were required to improve overall plant reliability and reduce any potential downtime. The following is a list of plant modifications completed during the last quarter of 1995:

- Increased Microcel clan coal transfer line from 3 inch to 8 inch diameter to improve throughput
- Increased screw feeder discharge pipe from 4 inch to 6 inch to prevent plugging
- Repaired / relocated filtrate pump level switch
- Installed throttle valve on WesTech filter feed line to prevent filter overflowing
- Installed protection screen inside of primary ball mill discharge sump to protect pump (100-G-01) from grinding media
- Installed protection screen inside of cyclone feed sump to protect pump (100-G-02) from grinding media
- Raised Microcel spray water header rings to improve froth washing
- Western Industrial Contractors, Inc. replaced failing gear reducer on secondary ball mill
- Replaced WesTech faulty drum filter blower
- Installed Microcel tailings sampler
- Increased quantity of Microcel launder sprays from 4 to 8
- Installed air booster on Microcel spray water flow control valve (FV-206) to improve operation
- Generated CDAS status indication of thickener underflow air valve
- Generated CDAS indication of Microcel spray water flow
- Relocated Microcel launder spray water line upstream of flowmeter FIT-206 to improve flow measurement
- Setup CDAS operation of emergency tank air pump (400-G-10)
- Setup CDAS operation of tailings filter feed air pump #1 (400-G-01)

- Setup CDAS operation of tailings filter feed air pump #2 (400-G-02)
- Setup thickener level probe indication on CDAS screen
- Mech-EI connected emergency tank agitator to prevent solids settling in tank
- Mech-EI connected level sensor on primary ball mill discharge pump (100-G-01)
- Mech-EI connected level sensor on cyclone feed pump (100-G-02)
- Mech-EI connected level sensor on Netzsch FGM feed pump (100-G-03)
- Installed thickener feed bypass line from filtrate to clarified water sump to improve clarified water quality
- Increased sampler discharge opening to prevent overflowing
- Installed recirculation line in Microcel feed sump to improve nuclear density gauge calibration
- Installed bubble tube level indicators to better determine Microcel froth interface
- Installed check valve on Netzsch fine grinding mill fresh cooling water line to prevent clarified water contamination
- Installed flow restriction valve downstream of Microcel air flow indicator (FIT-209) to prevent air flow fluctuations.
- Relocated Microcel clean coal discharge line to transfer sump (300-D-01) located adjacent to the flotation column. The close proximity of the sump results in a vertical drop of the discharge line. Not only should this help alleviate any froth backup in the Microcel launder but the expected reduction in launder / push water should increase the product solids concentration and improve the overall PDU filtering capacity.
- Relocated the primary ball mill water addition point from the base of the mill feed chute to the top. The water discharge angle was changed from a horizontal position to a downward vertical position. The downward flow of water should keep the chute clean and improve the flow of coal.
- Installed WesTech filtrate bypass line. The high solids concentration of the WesTech drum filter (1% solids) contaminated the clean filtrate from the Netzsch and EIMCO filters. Therefore, Entech personnel routed the WesTech filtrate in a fashion which allows the solids to be removed in either the Netzsch or EIMCO filters before discharging the clean filtrate to the filtrate sump. The improved filtrate quality permits this stream to be discharged directly to the clarified water sump.
- Increased speed of ground product pump by changing sheaves and increasing motor horsepower to 7.5 hp. This will eliminate overflowing of the ground product sump when processing 4,200 lb/hr of coal.

- Received and installed a new electronic console on the Berthold nuclear density gauge. The new console eliminated the cycling problem which was sometimes experienced.
- Installed horizontal baffles inside the Microcel feed sump (200-D-01) directly under the discharge of the sump feed and makeup water pipes. The baffles help eliminate small bubbles which are sometimes entrained in the slurry and cause erroneous values from the nuclear gauge.
- Installed a heater on the 2nd floor of Area 100. This will eliminate the chance of pipe freezing and potential downtime.

PDU Flotation Module Instrument Calibration

In order to better operate and control the PDU Flotation Module, all process control and display instrumentation was double-checked for measurement accuracy. The following is a list of all instrument calibration work performed during the last quarter of 1995:

- Calibrated Technetics weigh belt feeder (WIC-103)
- Calibrated Area 100 clarified water flowmeter (FIT-104)
- Calibrated primary ball mill feed push water flowmeter (FIT-105)
- Calibrated cyclone feed sump dilution water flowmeter (FIT-114)
- Calibrated Sisetec push water flowmeter (FIT-116)
- Calibrated ground product sump dilution water flowmeter (FIT-117)
- Calibrated Microcel feed flowmeter (FIT-202)
- Calibrated Area 200 clarified water flowmeter (FIT-204)
- Calibrated Microcel density control flowmeter (FI-205)
- Calibrated Microcel spray water flowmeter (FI-206)
- Calibrated Microcel launder flowmeter (FI-207)
- Calibrated Microcel air flowmeter (FIT-209)
- Calibrated feed bin level indicator (LIT-101)
- Calibrated cyclone feed sump level indicator (LIT-114)
- Calibrated ground product sump level indicator (LIT-118)
- Calibrated Microcel feed sump level indicator (LIT-201)
- Calibrated Microcel tailings sump level indicator (LIT-213)
- Calibrated emergency tank level indicator (LIT-401)
- Calibrated clarified water sump level indicator (LIT-402)
- Calibrated tailings filter sump level indicator (LIT-411)
- Calibrated Netzsch filter sump level indicator (LIT-421)
- Calibrated WesTech sump level indicator (LIT-422)
- Calibrated Microcel bubble tube level indicators

PDU Flotation Module Test Plan

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

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

Table 8. Parametric Test Matrix - Taggart Coal

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

Table 9. Parametric Test Matrix - Indiana VII Coal

<u>Test #</u>	<u>Collector (lb/ton)</u>	<u>Frother (lb/ton)</u>	<u>% Solids</u>	<u>Air Rate (CFM)</u>	<u>Wash H₂O (GPM)</u>	<u>Recirculate (GPM)</u>	<u>Feed Rate (lb/hr)</u>
1	5	2.5	7.50	55	142	800	3200
2	7	2.5	7.50	55	142	800	3200
3	3	2.5	7.50	55	142	800	3200
4	5	3.5	7.50	55	142	800	3200
5	5	1.5	7.50	55	142	800	3200
6	5	2.5	10.00	55	142	800	3200
7	5	2.5	5.00	55	142	800	3200
8	5	2.5	7.50	75	142	800	3200
9	5	2.5	7.50	35	142	800	3200
10	5	2.5	7.50	55	142	800	3200
11	5	2.5	7.50	55	180	800	3200
12	5	2.5	7.50	55	100	800	3200
13	5	2.5	7.50	55	142	1000	3200
14	5	2.5	7.50	55	142	600	3200
15	5	2.5	7.50	55	142	800	2500
16	5	2.5	7.50	55	111	800	2500
17	5	2.5	7.50	55	142	800	3900
18	5	2.5	7.50	55	173	800	3900
19	5	2.5	7.50	55	142	800	3200

Table 10. Parametric Test Matrix - Hiawatha Coal

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

Miscellaneous Activities

The following miscellaneous activities were performed during the quarter:

- Public Service Company contractor completed paving of saw-cut areas of pavement east of pilot plant
- Installed torque sensor on thickener drive
- Performed general housekeeping of PDU
- Replaced frequency motor on Sizer screen #1
- Installed additional structural support to beam in Area 100 as per Bechtel instructions
- Filtered contents of emergency tank. Found 3-mm glass beads in the tailings filters. Ran Netzsch fine grinding mill but found no beads in the mill discharge stream (filtered mill discharge stream with 16 mesh screen)
- Unplugged transfer line from Area 100 to emergency tank

- Installed siding on raw coal storage bunker
- Calibrated Techweigh belt feeder
- Calibrated nuclear density gauge
- Installed a catch basin at the inlet of each ball mill. The basin catches all slurry leaking from the mill inlet and directs it to floor drain. This reduces the amount of labor needed during clean up of Area 100
- Cleaned / unplugged Sizetec screen spray nozzles
- Unplugged piping from ground product sump to emergency tank
- Technicians checked and adjusted I/P on cyclone feed sump makeup water valve (LCV-114)
- Technicians checked and adjusted I/P on Microcel air flow control valve (FCV-209)
- Technicians checked and adjusted I/P on Microcel density control valve (DCV-203)
- Technicians checked and tried to adjust I/P on Microcel spray water control valve (FCV-206). Was unable to improve performance due to faulty air volume booster
- Technicians performed a volumetric flow comparison on the discharge of the Microcel feed sump. At a pump speed of 15% and a sump level of 45%, a portable Polysonics flowmeter indicated 57.3 gpm while flowmeters FIT-205 and FIT-202 both read 59 gpm
- Continued to disassemble and dispose of scrap steel
- ADT installed fire detection system in pilot plant
- Moved clean coal sampler actuator from the drum filter feed sump (400-D-03) to the inlet of the new clean coal transfer sump (300-D-01).
- Installed new fresh water header to the 2nd floor of Area 100. This will enable the PDU to use fresh water / makeup water as needed without impacting the other areas of the facility also using fresh water

TASK 9 SELECTIVE AGGLOMERATION MODULE

Phase III of this project involves the construction and operation of a 2 t/hr selective agglomeration (SA) PDU module. This SA module will be integrated with the existing PDU facility constructed during Subtask 8.2 and currently being operated under Subtask 8.3.

During operation of the SA module, the existing coal handling and grinding circuits will be used to generate ground slurry feed for the selective agglomeration process. Similarly, the existing product and tailings dewatering circuits will also be used. As such, the SA module will essentially replace the Microcel™ flotation column, with the remainder of the plant remaining intact.

Just like the advanced flotation PDU, selective agglomeration process performance will be optimized at the 2 t/hr scale, and 200 ton lots of ultra-clean coal will be generated for each of the 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 or some other user for end-use testing.

Subtask 9.1 Construction

The initial task under Phase III of the project involves the construction of the SA module. This work was initiated during Task 7 (Phase II) with the completion of the detailed design of the SA module.

Utilizing this detailed design, a construction bid package was prepared and submitted to the following Colorado based contractors on December 15, 1995:

- The Industrial Company, Steamboat Springs, CO
- Read Industrial Corporation, Wheatridge, CO
- Western Industrial Contractors, Denver, CO
- Mech EI, Inc., Aurora, CO

As of the end of this reporting quarter, the following schedule is planned for completion of Subtask 9.1:

- | | |
|-------------------------------|--|
| • January 3, 1996, Wednesday: | Site Inspection by Interested Bidders |
| • January 15, 1996, Monday: | Proposal Due at Amax R&D |
| • January 22, 1996, Monday: | Selection of the Subcontractor |
| • February 1, 1996, Thursday: | Letter of Intent to Award the Contract |
| • February 12, 1996, Monday | Signing of the Subcontract |
| • February 15, 1996, Thursday | Mobilization |
| • June 30, 1996 | Construction Completion |

Material Requisitions

Material requisitions (MR) for the bulk of the capital equipment to be purchased for the construction of the SA module were issued during December and early January. Items for which MR's were issued along with the number of vendors to which they were submitted are shown in Table 11.

Material requisitions for the following equipment will be issued early next quarter:

- One sump pump
- One Heptane pump

- One Nitrogen package (rental)
- One Carbon Drum Filter
- One Boiler (rental)
- One conditioner storage tank mixer
- One mist eliminator

Table 11. Selective Agglomeration Module Capital Equipment Material Requisitions

<u>Equipment Description</u>	<u>Number of Items</u>	<u>Number of Vendors</u>
Horizontal centrifugal slurry pumps	2	5
Horizontal centrifugal pumps	3	5
Progressive cavity pumps	1	4
Diaphragm pumps	1	4
Air coolers	2	4
Shell and tube heat exchangers	5	5
Air cooled liquid chiller	1	3
Vibrating screen	1	3
Process vessels	12	2
Gas holder	1	3
Flare stack	1	3
Agitators and impellers	5	2

In addition to those items listed above, the following used equipment, currently on-site, will be utilized during the construction of the SA module:

- One vessel for boiler feed water
- One vessel for conditioner storage tank
- One centrifugal pump for agglomeration circuit slurry feed delivery
- One gear pump for conditioner delivery
- One vessel with agitator for high shear reactor A
- Two vessels for recirculating cooling water
- One vessel for once through cooling water

Any reconditioning or modifications required for this used equipment will be initiated early next quarter.

Water Supply

In anticipation of Task 9 utility water requirements, and to reduce the occurrence of drops in the pilot-plant water pressure during on-going plant operations, installation of

a 6-inch water line was completed during this reporting quarter. This water line enters the building in the southeast corner of Area 100 (grinding plant). Plans for this water supply include a 2-inch line to tie into the existing plant utility water system, a 2-inch line for Microcel™ wash water, and several other 2-inch supplies to be used during PDU selective agglomeration module operation.

PLANS FOR NEXT QUARTER

Following are the activities anticipated for continued work and/or completion during the fourteenth quarterly reporting period, January - March, 1996:

- Subtask 3.2 - Near Term Applications testing will continue as follows
 - Complete the parametric tests planned for the 30-inch Microcel™ column at the Lady Dunn Preparation Plant.
 - Complete coal-water slurry fuel preparation tests at Amax R&D on samples of the spiral concentrate and the Microcel™ froth from the Lady Dunn Plant.
 - Preparation of a paper describing the joint DOE/Cannelton near-term application effort at Lady Dunn for presentation at the CoalPrep 96 conference.
- Subtask 3.3 - Dewatering Studies testing will continue with additional contact angle measurements and the start of free energy of displacement determinations. Parametric testing with the HD batch dewatering unit will also commence.
- The final version of the Subtask 4.4 topical report will be issued.
- Test work will be completed on Subtask 6.4, Selective Agglomeration CWF Formulation Studies.
- Test work will be completed on Subtask 6.5, Selective Agglomeration Bench-scale Testing.
- Work will be completed on the Task 7 detailed design of the selective agglomeration module of the 2 t/hr PDU and the design package issued.
- Under Subtask 8.1, arrangements will be made for purchase of Hiawatha coal for PDU operations.
- Subtask 8.4 efforts will be directed toward the following:
 - Complete parametric testing of Taggart coal
 - Complete parametric testing of Indiana VII coal
 - Commence parametric testing of Hiawatha coal
- Under Subtask 9.1, the PDU Selective Agglomeration Module construction will begin.

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APPENDIX A

GranuFlow Test Report - DOE/PETC

The Application of the GranuFlow Process to Column Flotation Froth from the Lady Dunn Coal Preparation Plant

Introduction

Under the Department of Energy's (DOE) Premium Fuel Development program--a sub-program of the Clean Fuels from Coal Program, Entech Global arranged with the Lady Dunn Coal Preparation Plant of the Cannelton Coal Company, a subsidiary of Cyprus Amax Coal Company, in West Virginia to ship 12 drums of clean coal froth (product of Microcel flotation column) to the Pittsburgh Energy Technology Center (PETC) for dewatering and reconstitution testing. The PETC-developed and -patented GranuFlow Process, a fine-coal dewatering and reconstitution process, is a recent concept that combines dewatering and reconstitution into one step. This process is aimed at improving the effectiveness of mechanical dewatering and reducing overall moisture. At the same time, it minimizes coal losses and dust emissions during transportation, handling, and storage, and produces an economical reconstituted fine-clean-coal product that is easy to handle. The process requires the addition of a small amount of specially selected binding material to the fine clean-coal slurry before filtration or centrifugation. This report describes the centrifugal testing results of using Orimulsion in the GranuFlow process on the Lady Dunn clean coal froth.

Experimental Procedures

Coal Samples

The clean coal froth received from the Lady Dunn Coal Preparation Plant was in slurry form with 10% solids content and 7.75% ash in the solids. According to the size distribution analysis, as shown in Figure 1, it was a 28 mesh x 0 coal, but the amount of minus 400 mesh material was about 60% which is more than twice the amount in a nominal 28 mesh x 0 coal.

Binding Agent

Orimulsion is a low-cost and high-Btu bitumen-in-water emulsion from Venezuela. It is being used as a fuel for power generation. The emulsion contained about 30% bitumen, 70% water, and a trace amount of surfactants.

Dust Index and Dust Reduction Efficiency

To evaluate the performance of the GranuFlow Process, PETC adopted a simple Ro-Tap dry screening process to experimentally measure the dust index (li) of the cakes with a constant amount of stress applied. A dust reduction efficiency is therefore calculated based on the following equation.

$$E = \frac{l_o - l_i}{l_o} \times 100$$

where, E = percent efficiency of dry cake dust reduction, %.
 l_o = dust index of coal, cumulative weight percent of feed coal finer than 100 microns by wet screening.
 l_i = dust index of cake, cumulative weight percent of dry cake finer than 100 microns after Ro-Tapping for 5 minutes.

Test Equipment

High-Gravity Solid-Bowl Centrifuge: A 14-inch Sharples high-gravity solid-bowl centrifuge was used for continuous fine-coal dewatering. The rated capacity of the centrifuge is around one to two tons of coal per hour, and the maximum speed is 3000 rpm, which provides a force field of 1800g. Previous data indicated that as long as there was a 12-foot pipe between the Orimulsion addition point and the centrifuge, a static mixer was not necessary.

Screen-Bowl Centrifuge: A 6-inch diameter continuous screen-bowl centrifuge manufactured by Bird Machine Co. was used. A KOCH SMXL-B static mixer, ½-in diameter and 6-in long, with 6 elements was installed in the dewatering system to provide adequate mixing of coal and emulsion before centrifuge dewatering.

Coal Preparation Process Research Facility(PRF): The Sharples centrifuge test work was conducted in the PRF. This facility is used for bench-scale development and testing of emerging equipment/technologies at rates of between 100 and 2700 lb. of coal per hour.

Test Plan

The fixed test conditions were 10% feed solids concentration (as received froth) and 300 lb/h centrifuge feed rate. Emulsion dosages were 0, 2, 6, and 8% for screen-bowl centrifuge tests and 0, 1.2, 4.3 and 7.3% for the high-gravity solid-bowl centrifuge. The percentage are based on the amount of bitumen versus the weight of coal. The test duration per set of conditions was about 15 minutes for the high-gravity solid-bowl centrifuge, and 12 minutes for the screen-bowl centrifuge. Samples collected per setting were slurry feed, dewatered coal, and slurry effluent. Timed effluent samples were measured for a material balance. Samples were analyzed for product moisture content, product dust reduction efficiency, effluent solid and hydrocarbon contents, and size distribution of the emulsion-treated feed slurry.

Screen-Bowl Centrifuge: The feed rate selected was 3 gpm, and the Orimulsion dosages were 1, 2, 6 and 8%. For each centrifuge dewatering test, a drum of slurry was mixed for 15 minutes and the slurry was fed through a static mixer into the

centrifuge for dewatering. Orimulsion was added to the feed line before the static mixer.

High-Gravity Solid-Bowl Centrifuge: To provide for the continuous bench-scale high-gravity solid-bowl centrifuge testing, 250 gallons of Lady Dunn clean coal froth were pumped into the slurry mixing tank with a conventional mixer. The slurry was then pumped to the centrifuge for dewatering. An emulsion addition system was attached to the centrifuge feed line. It consisted of an emulsion pump and container, and two static mixers. A conveyer received the centrifuge product to transfer it to a trailer for collection.

Summary

The GranuFlow Process reduced the fine coal moisture content, enhanced the handleability of the dewatered fine coal, reduced the dustiness of the fine coal, improved the product ash content and increased the fine coal production. Results and discussion are summarized in the following.

Centrifuge dewatering results are shown in Table 1 and Figure 2. The screen-bowl centrifuge product moistures were 39.4, 39.7, 35.7 and 35.2% with Orimulsion additions of 0, 2, 6 and 8% respectively. The high-gravity solid-bowl centrifuge product moistures were 34.8, 35.2, 36.3 and 31% with Orimulsion additions of 0, 1.2, 4.3 and 7.3% respectively. Compared to a screen-bowl product moisture with no Orimulsion the high-gravity solid-bowl centrifuge test with 8% Orimulsion reduced the product cake moisture by a total of 8 percentage points, of which 4 percentage points were contributed by the higher gravity of the high-gravity centrifuge alone and about 4 percentage points were contributed by the Orimulsion treatment. The difference in dewatering efficiency of the two centrifuges could be due the difference in G-force, which for the high-gravity solid-bowl centrifuge and screen-bowl centrifuge is 1789 and 576 respectively. The role of Orimulsion on fine coal dewatering could be due to the increased particle agglomeration and particle surface hydrophobicity.

In this series of tests the product moisture reductions were not pronounced compared with previous tests PETC has conducted. This could be due to the low slurry solids content and the higher amount of fine particles in the slurry. However, the handleability of the high-gravity solid-bowl centrifuge product improved with the additions of Orimulsion. Free flowing granules were produced at an Orimulsion addition of 7.3%.

The particle size distributions of the clean coal froth mixed with Orimulsion additions of 0, 2, 6 and 8% are shown in Figure 1. It clearly indicates that fine coal agglomeration occurred with the addition of Orimulsion at 6 to 8%. This enlarged particle size of the fine coal slurry could contribute to the product moisture reduction.

The dust reduction efficiency indicated that above 2% Orimulsion addition, more than 60% of the particles finer than 100 microns were agglomerated for both high-gravity solid-bowl and screen-bowl centrifuges. The dust reduction efficiency reached 92% and 95% at 7.3 and 8% Orimulsion addition, respectively.

The screen-bowl product ash contents (avg. 6.26%) were lower than the solid-bowl ash contents (avg. 7.27%) due to the selective removal of fine clay with the screen-bowl main effluent. The Orimulsion treatments further reduced the product ash contents for the high-gravity solid-bowl centrifuge from 7.73% to 6.84% and for the screen-bowl centrifuge from 6.71% to 6.01%.

The material balance calculation indicated that the addition of Orimulsion at 2, 6, and 8% could increase coal production from the screen-bowl centrifuge by 2.5, 5.6, and 7 percentage points, respectively. The increase in coal production was accomplished by reducing the coal losses in the effluent stream as a result of fine coal agglomeration.

Table 1. Test Results for Centrifuge Dewatering of Flotation Froth from Lady Dunn Coal Preparation Plant

Test No.	Orimulsion wt%	Static Mixer No.	Product		Main		Screen Effluent		Dust Index	Dust Reduction Eff. wt%	Solid Balance, wt%			
			Solid Moist.%	Solid Ash %	Effluent Solid %	Effluent Ash %	Solid %	Ash %			Feed	Product	Main Effluent	Screen Effluent
Lab 6"-Screen-Bowl Centrifuge Testing (G-Force = 576)														
17-1	0	1	39.4	6.71	1.13	16.3	33	9.35	83	3	100	89.5	5	5.5
17-2	2	1	39.7	6.31	1.4	15.1	13.2	9.3	34	60	100	92	6.3	1.7
17-3	6	1	35.7	5.99	0.91	21.2	5.15	9.1	7	92	100	95.1	4	0.9
17-4	8	1	35.2	6.01	0.73	22.3	1.28	14.7	4	95	100	96.5	3.3	0.2
HI-G 14"-Solid-Bowl Centrifuge Testing (G-Force =1789)														
16-1	0	0	34.8	7.73	0.17	57.1	NA	NA	73	15	100	98.6	1.4	NA
16-2	1.2	0	35.2	7.41	0.1	53.6	NA	NA	66	23	100	99.2	0.8	NA
16-3	4.3	0	36.3	7.1	0.12	54.7	NA	NA	13	85	100	99	1	NA
16-4	7.3	0	31	6.84	0.1	55.7	NA	NA	7	92	100	99.2	0.8	NA

Figure 1. Lady Dunn Coal Preparation Plant Coal Particle size Distribution with Orimulsion Treatment

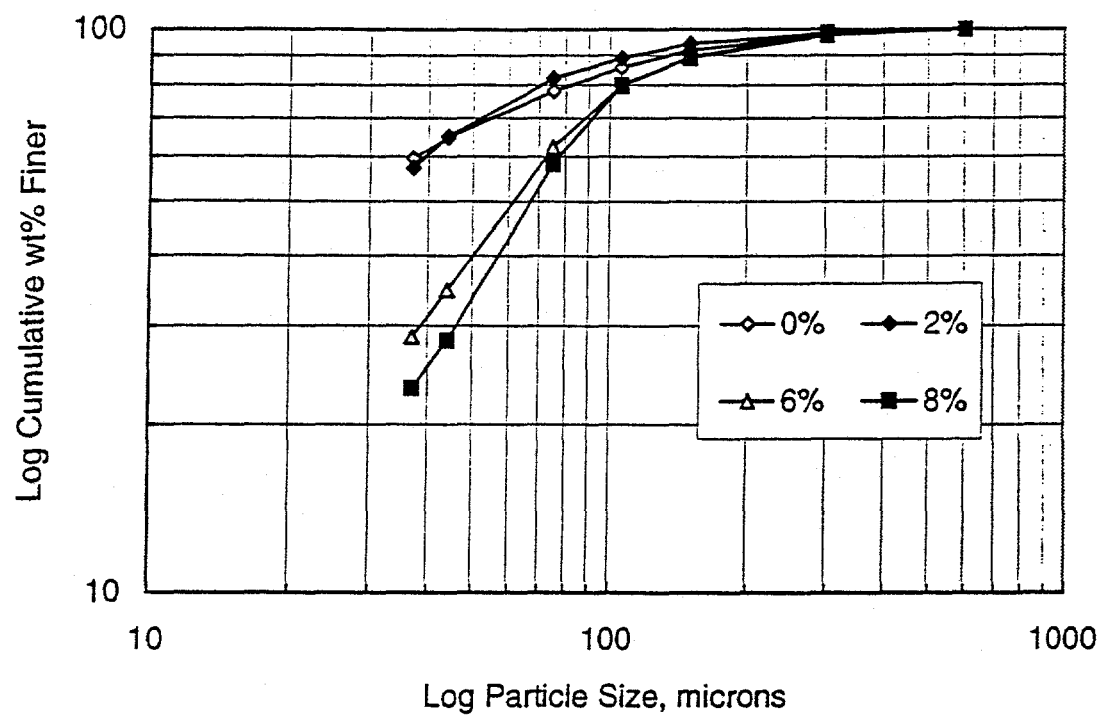
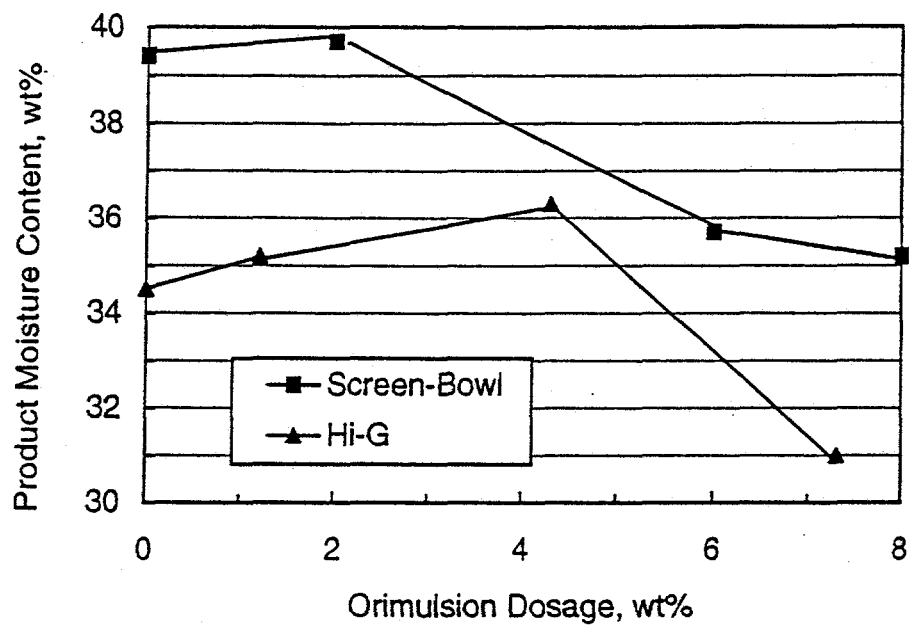


Figure 2. Lady Dunn Preparation Plant Coal Product Moisture Content with Orimulsion Treatment



APPENDIX B

Winifrede Coal Agglomeration Results

Appendix B - Winifrede Coal** - 25 lb/hr Agglomeration Test Conditions and Results

System Configuration										Without Froth Skimming										With Froth Skimming																			
High Shear					Low Shear					Scrn		Feed		Screen		Prod Tails				Agg Perform				Product				Tails				Agg Perform				ROM Perform			
Run	Imp	Tip	RT	In	Tip	Imp	RT	In	deg	Incl	% Sol	% lb/hr	% ash	% Hept	% Size	% ash	% ash	% Yield	% Rec.	% Btu	% AR	% ash	% #ash/ MBtu	% ash	% Rec.	% Yield	% Rec.	% Btu	% AR	% Yield	% Rec.	% Btu	% AR	% Yield	% Rec.	% Btu	% AR	Comments	
W1A1	2.4	15.0	1.4	4.8	4.8	6.9	40	9.9	25.2	8.68	60.2	43.2	2.5	2.89	82.2	92.7	99.2	69.1	2.90	2.00	85.8	93.0	99.6	68.9	64.7	89.1	93.4	Continuous growth											
W1A2	2.4	18.0	1.4	4.8	4.8	6.9	40	8.9	22.7	8.70	62.4	19.5	<5	4.60	39.8	88.3	92.7	53.3	4.47	3.13	87.7	94.9	99.8	51.3	66.0	89.3	89.7	No growth											
W1A3	2.4	18.0	1.4	4.8	4.8	6.8	40	9.9	25.5	8.69	56.5	37.3	1	3.09	85.2	93.2	99.5	66.9	3.09	2.13	87.3	93.3	99.7	66.8	64.9	89.2	93.0	1mm monosize, slow continuous growth											
W1A4*	2.4	18.0	1.4	4.8	4.8	7.1	38	9.8	24.7	8.67	53.2	46.8	2	2.88	83.1	92.8	99.3	69.2	2.88	1.98	86.4	93.1	99.6	69.1	64.7	89.2	93.5	2.5 mm monosized, slow continuous growth											
W1A5	2.4	18.0	1.4	4.8	8.0	3.2	40	10.0	25.5	8.67	56.3	40.6	4	2.76	57.0	89.1	95.5	71.6	2.77	1.91	84.6	92.8	99.4	70.3	64.5	89.0	93.7	4mm monosize, cyclic growth											
W1A6	2.4	18.0	1.4	4.8	8.0	3.3	38	9.9	25.2	8.68	55.2	38.4	1.5	2.87	80.9	92.6	98.1	69.4	2.87	1.97	84.2	92.9	99.4	69.3	64.5	89.0	93.5	1.5 mm monosized, cyclic growth											
W1A7	2.4	18.0	1.8	4.8	8.0	4.3	38	10.2	19.5	8.67	53.2	40.7	5-3.5	2.83	81.6	92.6	99.2	69.8	2.83	1.95	85.4	92.9	99.5	69.7	64.6	89.1	93.6	Cyclic growth											
W1A8	2.4	18.0	1.4	4.8	8.0	3.3	38	12.8	32.3	8.69	54.7	31.2	<5	3.84	42.3	87.4	92.5	61.4	3.80	2.64	88.5	94.2	99.8	58.8	65.5	89.3	91.3	Cyclic growth											
W1A9	2.4	18.0	1.4	4.8	8.0	3.4	38	6.9	17.2	8.53	55.7	27.2	2	2.87	81.5	92.6	98.1	69.4	2.87	1.98	85.0	92.9	99.5	69.3	64.6	89.0	93.5	Continuous growth											
W1A10	2.4	19.8	1.4	4.8	9.6	3.3	38	6.9	17.5	8.42	57.4	35.2	2	2.72	60.2	90.1	96.3	70.9	2.73	1.87	82.7	92.9	99.3	69.9	64.6	88.9	93.6	Continuous growth											
W1A11	2.4	18.0	2	4.8	8.0	4.6	38	6.9	12.6	8.49	58.5	37.6	2.5	2.78	77.7	92.4	98.8	69.8	2.78	1.91	82.3	92.8	99.2	69.6	64.5	88.8	93.6	Steady state 2.5 mm product											
W1A12	2.4	19.1	1.2	4.8	4.8	6.2	38	6.9	19.7	8.45	61.3	38.8	2	2.74	78.3	92.4	98.8	70.2	2.74	1.88	82.2	92.8	99.2	70.0	64.5	88.8	93.7	Continuous growth, no spray water											
W1A13	2.4	18.0	1.4	4.8	4.8	7	38	12.9	32.4	8.45	54.5	24.1	2s	4.13	33.2	85.1	89.6	58.4	4.42	3.10	47.6	90.7	95.1	52.6	63.0	85.1	90.0	Product degraded to 2 mm sludge											
W1A14	2.4	18.0	1.4	4.8	4.8	3.3	38	9.9	25.0	8.38	57.2	33.9	1.5	3.02	67.0	91.6	97.6	67.0	3.04	2.09	72.3	92.3	98.3	66.5	64.1	87.9	92.9	Cyclic growth, LS impeller change											
W1A15	2.4	18.0	1.4	4.8	4.8	7	38	10.0	25.2	8.37	51.8	42.5	0.5	3.27	70.8	92.4	98.2	63.9	3.27	2.26	88.6	94.0	99.8	63.3	65.3	89.3	92.2	Minimal growth with cycling											
							34				36.1	0.5	3.37																										
							30				36.2	0.5	3.32																										
W1A16	2.4	18.0	1.1	4.8	4.8	5.5	38	10.0	32.4	8.36	54.4	49.6	2	3.03	84.6	93.5	99.5	66.1	3.03	2.09	87.4	93.7	99.7	66.0	65.1	89.3	92.8	Continuous growth											

* Utilized 5 lb Asphalt per ton feed coal

** Feed particle size distribution with 7.14 micron mass mean diameter

APPENDIX C

Hiawatha Coal Agglomeration Results

Appendix C - Hlawatha Coal - 25 lb/hr Agglomeration Test Conditions and Results

Run		System Configuration																Screen				W/out Froth Skim				With Froth Skim				Comments		
		High Shear				Low Shear				Vibrat. Screen				Feed				Product		Prod Tails		Prod Tails		Prod Tails								
		Tip	Imp	RT	min	Tip	Imp	RT	min	Mesh	deg	lb/hr	%	Solids	lb/hr	ash	%	Heptane	%	Sol	mm	Size	%	ash	%	ash	%	ash	%			
H1A1		150	2.4	8.3	1.4	4.8	8.0	3.3	48	38	47.6	10.5	26.9	8.50	22.3	24.4	52.5	5-2	2.43	70.0	91.0	2.49	83.2	92.6							Cyclic growth	
H1A2		150	2.4	18.0	1.4	4.8	8.0	3.4	48	38	47.6	10.3	26.1	8.21	22.6	24.6	37.4	0-1	3.14	48.4	88.8	3.18	81.8	93.6							Steady-state, or very slow growth	
H1A3		150	2.4	15.0	1.4	4.8	4.8	7.2	48	38	30	10.0	25.4	8.09	22.8	24.9	56.5	5-1	2.61	71.6	92.1	2.64	84.6	93.4							Slow continuous growth, low shear filling	
H1A4		150	2.4	15.0	0.7	4.8	4.8	3.7	48	38	47.6	10.0	50.1	8.13	22.1	24.1	38.4	0-5	3.12	53.9	90.1	3.14	84.4	93.9							Semi steady-state with some cycling	
H1A5		150	2.4	15.0	0.7	4.8	4.8	3.6	48	38	47.6	10.0	50.5	7.89	22.6	24.6	42.9	0-1	2.83	69.2	92.4	2.83	86.5	94.0							Similar to H1A4	
H1A6		150	2.4	15.0	1.4	4.8	4.8	7.1	48	38	47.6	9.9	25.3	7.96	23.3	25.3																2" discharge port, continuous growth, low shear plugged
H1A7		150	2.4	15.0	1.4	4.8	6.4	3.4	48	38	47.6	9.8	24.5	7.69	26.3	28.5	39.2	0-1	2.88	66.6	92.5	2.89	84.9	94.1							New 1" port, semi steady-state	
H1A8		150	2.4	15.0	1.4	4.8	6.4	3.3	48	38	47.6	10.0	25.6	7.47	26.7	28.8	49.2	0-3	2.56	81.0	93.7	2.57	86.0	94.1							Same as H1A7, oval agglomerates, steady-state	
H1A9		150	2.4	15.0	1.4	4.8	6.4	3.3	48	38	47.6	10.1	25.8	7.66	26.0	28.2	55.4	0-3	2.36	82.6	93.4	2.40	86.2	93.7							New 1" port, LS discharge problems	
H1A10		150	2.4	15.0	1.4	4.8	4.8	3.4	48	38	47.6	9.9	25.3	7.68	23.7	25.6	40.8	5-1.5	2.66	55.1	90.4	2.67	81.8	93.7							New 1" port, steady state	
																	40.6		2.62													
H1A10R		150	2.4	15.0	1.4	4.8	4.8	3.4	48	38	40	10.0	25.3	7.45	23.6	25.5	42.9	0-1.5	2.62	77.7	93.6	2.63	85.1	94.2							Same as H1A10	
H1A11		150	2.4	15.0	1.4	4.8	4.8	7.2	48	38	0	9.9	24.8	7.39	24.2	26.1	71.4	3-6	2.06	81.3	93.3	2.13	86.9	93.8							Continuous growth, low shear plugged	
H1A12		150	2.4	15.0	1.4	4.8	4.8	3.4	48	38	47.6	10.0	25.5	7.40	24.6	26.5	49.1	0-3	2.34	76.1	93.1	2.36	84.2	93.8							New 1" port, steady state	
H1A13		150	2.4	15.0	1.4	4.8	3.0	3.4	48	38	47.6	10.1	25.5	7.35	24.6	26.5	42.7	0-3	2.68	72.4	93.3	2.69	84.3	94.3							Same as H1A12	

APPENDIX D

Taggart 2 Coal Agglomeration Results

Appendix D - Taggart 2 Coal - 25 lb/hr Agglomeration Test Conditions and Results

Grind	System Configuration										Screen										Without Froth Skimming										With Froth Skimming																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																	
	High Shear					Low Shear					Scrn					Feed					Product					Prod Tails					Agg Perform					Product					Tails					Agg Perform																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																		
	Imp	Tip	RT	Imp	Tip	RT	Incl	Scrn			Solids	%	lb/hr	ash	%	Hept	%	daf	Sol	%	Size	mm	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash	%	ash

Comments
Low shear plugging
Low shear plugging
Screened well
Screened well
Screened well
Screened in clusters
Screened in clusters