

FINAL TECHNICAL REPORT  
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Project Title: **PATHWAY TO COMMERCIALIZATION OF THE MOTORLESS ROTORLESS CELL**

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ABSTRACT

With Illinois Clean Coal Institute funding, the ISGS developed and demonstrated a proof-of-concept, lab-scale model of a motorless-rotorless froth flotation cell (MRC) in 2003. The invention was disclosed to the University of Illinois in March 2003, and an updated disclosure was filed in April 2004. Because the MRC does not use rotors, froth-scrapers and electric motors to produce froth concentrate, it occupies a smaller footprint, requires less maintenance, and operates at a lower cost than conventional froth flotation cells. Furthermore, the operating principle of mixing with eductors provides greater throughput capacity than is available in conventional froth flotation cells. The intent of this project was to scale-up the MRC and study the impact of different physical features and device configurations, MRC operational parameters, and effects of feed characteristics on the MRC's ability to produce a marketable product. The project consisted of laboratory work at the facilities of Dynamic Separations Incorporated (DSI), owners of the exclusive license for the technology, and field testing at Freeman Energy's Crown III mine.

The experimental work, conducted by DSI, was done in two phases. In the first phase, initial laboratory data were used to design a 1,000-gallon cell with sixteen 1½-inch feed eductors for scale-up testing. A test circuit with four 350-gallon-per-minute pumps feeding the MRC was assembled at DSI's facilities in Champaign, Illinois. This circuit was used to investigate various operating parameters and determine feed eductor positioning. Most importantly, testing confirmed the feasibility of fine coal recovery at acceptable levels with the 1,000-gallon MRC.

The second phase consisted of moving the 1,000-gallon MRC to Freeman Energy's Crown III preparation plant as part of a system to recover coal from the underflow of sieve bends dewatering the plant's polishing cyclone product. Initial tests were used to optimize the MRC's operating fluid level, air feed rate, static mixer configuration, and dosages for frother and collector. A second test set examined interaction effects of these parameters. Despite poor feed characteristics (19% solids with 28% less than 45-µm, 39.38% ash, 6.65% sulfur), phase two testing determined the 1,000-gallon MRC capable of achieving results in line with flotation release analyses of the feed material. Operating at optimum settings, the MRC product was 12,188 Btu/lb, 13.19% ash and 6.09% sulfur (dry basis) at a yield of 62.2% and a recovery of 83.5%, which compares favorably to the 12,748 Btu/lb, 10.15% ash, 4.17% sulfur (dry basis) product marketed by the mine.

## EXECUTIVE SUMMARY

To process coal fines more effectively while reducing associated processing costs, researchers at the Illinois State Geological Survey developed, tested and demonstrated a lab-scale motorless-rotorless flotation cell (MRC). Like the Imhoflot pneumatic cell, it requires relatively short retention times to process coal, and hence has greater throughput capacity while consuming less energy than conventional flotation cells with mechanically driven mixers. The MRC can also be fitted with the Inclined Froth Washer (IFW), another technology also developed by the ISGS, to further improve the separation of fine coal from mineral impurities. Development and testing of the MRC has been supported by research grants from the Illinois Clean Coal Institute to the University of Illinois.

Feed slurry is pumped through static mixers for aeration and enters the MRC through eductors, which also recycle slurry already within the cell for thorough mixing. Eductor positioning with the eductor jet angled parallel to the cell wall also causes some cyclonic action within the cell. This cyclonic action and the overall shape of the cell positively influence cell throughput by creating a pressure gradient that forces denser slurry with unattached particles to the outer edge of the cell where it is drawn into the eductors and remixed. As separation occurs, heavier mineral impurities drop to the cell bottom and are discharged as waste, while less-dense coal-laden froth is concentrated at the top center of the cell by the cyclonic action and is recovered through the IFW at the top of the MRC.

High throughputs can be achieved in the MRC by processing feeds with relatively high solids content. Unlike flotation columns, which also lack moving parts, mixing of air bubbles and slurry in the MRC is not limited to counter-current flow of ascending air bubbles and descending solid particles. The active and intense mixing achieved in the MRC without the use of rotors and/or electric motors ensures that there are numerous encounters between particles and air bubbles, and this enables the cell to process feeds with greater solids content than flotation columns. In conventional mechanically agitated flotation cells, mixing occurs at a constant rate. Thus, an increase in feed rate results in a decreased probability of any particle colliding with and attaching to an air bubble. In the MRC, mixing by eductors increases in direct proportion to the feed rate within the performance range of the eductors. Thus, increasing the feed rate does not reduce the probability of any particle colliding with and attaching to an air bubble. The effect is shorter retention time and greater throughput with the MRC. Because of relatively short retention times, the rate of clean coal production from the MRC is significantly greater than that of flotation columns. The IFW-equipped MRC occupies a relatively small footprint and, under optimum operating conditions, is expected to provide low cost, maintenance-free fine coal recovery and cleaning.

Early in this project, MHI Energy Partners LLC, a venture capital group holding exclusive licenses on the ISGS fine coal technologies, formed Dynamic Separations Incorporated (DSI) to collaborate with the Principal Investigator (PI) on moving the technologies towards commercialization. Towards that end, the intent of this project was to design, build and test a larger MRC unit in the laboratory and in the field. An additional cooperating partner, Freeman Energy, was engaged to host a field-scale

demonstration of the MRC. Their interest was to determine if the MRC could recover coal fines currently being discarded as waste, a practice common throughout the Illinois coal industry.

Using data generated during the initial 15-gallon lab-scale project and characteristics of the fine coal circuit of Freeman Energy's Crown III coal preparation plant, the MRC was scaled up to 1,000 gallons in size. A test circuit consisting of a 1,000-gallon MRC equipped with sixteen feed eductors, a 3,000-gallon mixing tank, four 350-gallon-per-minute (gpm) feed pumps and all of the associated piping was designed and assembled at the DSI facility in Champaign, Illinois. Fine coal samples from various operations including Freeman Energy's Crown III Mine, CONSOL's Rend Lake Mine, and DSI's Providence One coal recovery operation at a mine site in Kentucky, were tested in the circuit. Without a continuous stream of fresh feed material, testing was limited because of solid recirculation. However, this testing did show that the 1,000-gallon MRC was capable of recovering fine coal at acceptable levels of yield and recovery.

In April 2005, Freeman Energy agreed to allow the 1,000-gallon MRC to be moved to the Crown III mine site for a field test. In May 2005, the research grant was amended to establish DSI as the subcontractor assigned to design and conduct the field testing. Construction at the mine site began in June 2005, operational testing of the MRC circuit commenced in August 2005, and testing was completed in October 2005. Collected samples were analyzed independently by SGS and the ISGS.

Although not an ideal feed material, the underflow from sieve bends dewatering the plant's polishing cyclone product was selected for the feed to the MRC because the pipe carrying that material was conveniently located relative to the MRC test site made available by Freeman Energy. This material presented a challenge as feed slurry samples indicated that 28.1% of the solid material was ultra-fine (0.045mm X 0) and the slurry as a whole had an average ash content of 40.5% by weight. However, flotation release analyses performed at Southern Illinois University Carbondale and University of Kentucky suggested that a concentrate with ash content between 17.4% and 20.4% could be achieved at approximately 85% combustible recovery.

After functionality testing, two engineered experiments were designed and conducted. In the first experiment, critical parameters were examined and an optimum operating point established. These parameters and the optimal operating values determined were as follows:

- operating fluid level at 90 inches
- air feed rate at 30 standard cubic feet per minute (scfm)
- static mixer configuration with both 8-inch and 4-inch mixers installed
- frother dosage at 190 grams per minute
- collector dosage at 450 grams per minute

The second experiment used a Box-Behnken design to examine the interactions between factors. Analysis of Variance (ANOVA) results from the initial test matrix indicated that

only air flow and the air flow-fluid level interaction were significant factors at a 95% confidence level in predicting concentrate ash content. Follow-up testing found that the concentrate flow rate as measured by the difference between feed flow rate and reject flow rate was also a significant factor. These results were used to develop empirical models for predicting concentrate ash content, combustible recovery and ash rejection percentage.

Overall, when the MRC was operating at optimum levels for the critical parameters, it produced a 12,188 Btu/lb product with 13.19% ash and 6.09% sulfur (dry basis) at a yield of 62.2% and a recovery of 83.5%, which compared favorably with the 12,748 Btu/lb, 10.15% ash, 4.17% sulfur product (also on a dry basis) marketed by the mine.

With these results and the known operational parameters for the MRC, the economic feasibility of fine coal production at Freeman Energy’s Crown III Mine was analyzed for three scenarios based on feed stream possibilities. Each scenario assumed drying of the MRC concentrate with a screen bowl centrifuge using operational data supplied by Decanter Machine. The first scenario considered the same feed material used during pilot-scale testing, that being only the sieve bend underflow. In the second scenario, effluent of known quantity and quality from centrifugal dryers was added. The third scenario assumed additional feed sources from the plant sufficient to operate the MRC at a maximum production rate of 12.0 tons per hour. Results of this economic feasibility study, shown in Table 1, clearly indicate that the MRC must run at or near capacity to be economically justifiable. Given the significant amount of fines currently being rejected as waste at most Illinois coal mines, we believe that there is tremendous market potential for the MRC system.

Table 1. MRC Economic Feasibility Analysis for Freeman Energy Crown III Mine

| <b>Parameter</b>                    | <b>Scenario 1<br/>Test Feed</b> | <b>Scenario 2<br/>Available Feed</b> | <b>Scenario 3<br/>MRC Capacity</b> |
|-------------------------------------|---------------------------------|--------------------------------------|------------------------------------|
| MRC Feed (tons/hr)                  | 3.74                            | 16.00                                | 24.96                              |
| <b>Net Production</b> (tons/hr)     | 1.79                            | 7.68                                 | 11.98                              |
| Chemical Cost (\$/ton)              | 10.18                           | 3.42                                 | 3.42                               |
| Energy Cost (\$/ton)                | 11.25                           | 2.63                                 | 1.69                               |
| Capital Cost (\$/ton amortized)     | 34.67                           | 8.10                                 | 5.19                               |
| Labor Cost (\$/ton)                 | 4.65                            | 1.09                                 | 0.70                               |
| <b>Total Cost</b> (\$/ton)          | 60.75                           | 15.23                                | 10.99                              |
| Selling Price (\$/ton)              | 20.00                           | 20.00                                | 20.00                              |
| <b>Incremental Revenue</b> (\$/ton) | (40.75)                         | 4.77                                 | 9.01                               |
| Annual Production (tons)            | 6,996                           | 29,952                               | 46,725                             |
| Net Revenue (\$/year)               | (285,133.59)                    | 142,890.45                           | 421,054.00                         |

## OBJECTIVES

The goal for this research project, the second phase of developing the motorless-rotorless flotation cell (MRC), was to design and build a field-scale model that had been tested both in the laboratory and in the field. To achieve this goal, the following five objectives were planned:

1. Complete the design, fabrication, and assembly of a scaled-up version of the cylindrical MRC with conical bottom and domed top equipped with an ISGS Inclined Froth Washer (IFW).
2. Automate the cell.
3. Determine effects of cell characteristics on throughput and quality of clean coal product from the cell in preparation for industrial scale demonstration.
4. Determine effects of operating conditions such as feed rate, aeration intensity, aeration mechanism and retention time on the quality of clean coal produced by the cell.
5. Determine the impact of the different IFW types fitted to the scaled-up MRC.

MHI Energy Partners, a venture capital group focused on energy technologies, owns an exclusive license to market the MRC and other fine coal technologies being developed by the ISGS. In November 2004, they formed Dynamic Separations Incorporated (DSI) to partner and collaborate with the ISGS on technology commercialization. In May 2005, DSI was appointed by the ICCI as a subcontractor to the ISGS with responsibility for completing all of the project objectives.

## INTRODUCTION AND BACKGROUND

Mechanization of the coal mining industry has increased the amount of fine coal (material less than 1mm in size) being produced. Most Illinois coal preparation plants are able to efficiently recover and clean the coarser fraction (1mm X 0.15mm) of these fines using hydrocyclones and spirals. The method commonly used on an industrial scale throughout the industry to recover and clean the finest coal (0.15mm X 0) is froth flotation, either in subaeration cells or in columns. However, at present, most Illinois coal mine operators are not using any means to recover this ultra-fine product and it is being discarded as waste in slurry ponds or underground mine workings. This tremendous waste of a valuable resource points to the need for more effective processing technology. Fines recovered from some Illinois coals are much lower in sulfur and ash content, and consequently higher in heat content than conventional Illinois coal. These improvements in coal quality would make the total product more competitive with western coal. Improving the effectiveness and decreasing the cost of fine coal cleaning could significantly improve the marketability of Illinois Basin coals. The MRC is an energy efficient, low cost, low maintenance alternative to conventional subaeration and column flotation cells for recovering and cleaning fine coal. It has the potential both to significantly reduce the amount of run-of-mine (ROM) coal presently being rejected and placed in underground workings or surface impoundments that must be maintained and reclaimed, and to generate revenues from a high quality product that has low incremental

costs associated with producing it. The additional revenue stream to a mine combined with reducing associated waste disposal costs will improve profitability. The many Illinois mines that do not currently employ fine coal cleaning are the ones most likely to benefit from installation of a fine coal recovery and cleaning circuit based on MRC technology.

### **Description of Technology**

The ISGS developed the MRC concept in 2000 and, with support from the Illinois Clean Coal Institute (ICCI Project Number 02-1/4.1B-1), designed, built and tested lab-scale models that reduced the concept to practice. This new type of froth flotation cell is a high density polyethylene (HDPE) cylindrical vessel with a conical bottom and domed top. It employs off-the-shelf components such as eductors and static mixers. Feed slurry is pumped into the cell through inline static mixers. Air is injected into the slurry upstream of the mixers. The mixers shear the slurry/air mixture several times creating air bubbles within the slurry. Eductors use the venturi effect to agitate and mix slurry. Slurry pumped through the narrow nozzle of an eductor creates a high velocity stream that, when released into the larger flared part of the eductor, forms a low-pressure zone that draws in liquids, solids and gases from the area surrounding the eductor. In the MRC, slurry that enters through eductor nozzles into flared sections of eductors submerged in the cell entrains four times its volume in slurry already present in the cell. The feed and the entrained material are thoroughly mixed inside the flared section of the eductors before being discharged into the flotation cell. The eductors may be spaced and oriented in such a way that their mixing zones overlap, increasing the probability of particle-bubble collisions and the kinetic energy of those collisions. Because of the extremely high mixing rate, the time necessary to provide opportunities for collisions between hydrophobic particles and air bubbles (retention time) is reduced, resulting in greater throughput in the MRC than would be achievable with a conventional flotation cell.

Directing eductor flow tangentially along the MRC walls also creates a rotational flow in the tank. The pressure gradient formed by this fluid motion forces froth to separate and concentrate in the center of the cell. Heavier material migrates towards the cell wall where it is drawn back through eductors providing additional opportunities for mixing and attachment. The heaviest hydrophilic material eventually settles to the bottom of the MRC and is discharged as waste. This cyclonic effect enhances separation, reduces retention time and, hence, increases cell throughput. However, the rotational flow of the slurry-froth mixture combined with the conical bottom of the cell cause the formation of a vortex in the center of the cell. This vortex reduces the capability of the reject pipe to remove waste slurry and also causes some of the froth to short-circuit to the reject. To eliminate these undesirable effects, a vortex breaker was designed and installed in the MRC.

### **Rationale for Accelerated Development**

Typically, development of a new process or technology starts with proof-of-concept work in the laboratory. Scientists and engineers develop ideas that come either as a burst

of inspiration or as the result of incremental improvements to an existing process. Laboratory testing demonstrates concept validity. Next, bench-scale devices are manufactured to identify important parameters influencing the process and examine their impacts on the output and efficiency of the process. Pilot plant tests are then carried out at a larger scale to see how parameters defined in laboratory tests change with scale of the device and the quality of material processed. Successful pilot-scale tests are followed by industrial-scale demonstrations of a prototype unit under continuous operating conditions. At the conclusion of these industrial-scale tests, one-of-a-kind in-plant tests are carried out to subject the process to sustained use. Following all of these steps to commercialize the process or technology can take ten to fifteen years.

Because of the urgent need for improved coal cleaning technology and the success of early tests, the MRC has generated a great amount of commercial interest suggesting that it should be developed as quickly as possible. As a result, MRC development has expedited or bypassed several of the customary development steps. The first phase of development combined proof-of-concept and lab-scale testing, wherein design improvements and the impacts of various operating parameters were being considered simultaneously. This second phase research and development project combined pilot-scale and industrial-scale testing in just the third year of process/technology development with the expectation that the MRC would be ready for one-of-a-kind in-plant testing within five years of conceptualization.

## EXPERIMENTAL PROCEDURES

### **Design, Build and Operate a Scaled-up MRC Test Circuit**

MHI Energy Partners, a venture capital group based in Connecticut and focused on energy related technologies, acquired the technology license for the MRC before this project commenced. They committed significant resources to furthering this project including creation of Dynamic Separations Incorporated in September 2004. By the end of October 2004, DSI had acquired a laboratory in Champaign Illinois with adequate space and electrical service for dealing with the large tanks and electrically driven pumps involved in nearly full-scale operations.

Based on retention times achieved during first phase development of the MRC, the DSI-ISGS research team established targets for a scaled-up MRC to achieve 1,000 gallons-per-minute (gpm) throughput capacity with 1-minute retention time. This set the initial scale-up size for the MRC at 1,000 gallons. Accordingly, two large high density polyethylene (HDPE) tanks were purchased and delivered to the DSI facilities. One tank was a 1,000-gallon cell with a conical bottom to be used for the MRC. The other was a 3,000-gallon flat-bottomed tank to be used for storage, mixing and transfer of material to the MRC.

The primary mixing mechanism in the MRC is a tank mixing eductor (TME). Two criteria were considered in the decision of which venturi-type TME to use. First, TME length had to be kept to a minimum in order to avoid substantial disruption of the

tangential flow in the 1,000-gallon cell. Second, eductor size needed to be as large as possible to minimize the number of input port holes drilled in the cell. Judging proper mixing to be more important than minimizing holes in the cell, Penberthy 1½-inch diameter, 9¾-inch long TMEs capable of delivering 64-gpm at 35-psi were selected for use. Sixteen eductors were required to achieve the desired 1,000-gpm throughput capacity. Eductors were installed by drilling sixteen 1½-inch holes (four sets of four holes) in the side wall of the 1,000-gallon MRC tank. Four feed manifolds with four outlets, one for each hole in a set, delivered slurry through the wall of the cell as shown in Figure 1. Attached to the end of each feed pipe on the inside of the cell was an elbow and a TME. Phase 1 research indicated that the best orientation for the elbow and eductor was horizontal and tangent to the cell wall. The top and bottom of the 1,000-gallon MRC cell also had to be altered. First, to enable sufficient flow, the existing 2-inch drain port at the bottom of the cone was replaced with an 8-inch flanged fitting. A vortex breaker was installed to dampen the cyclonic flow of slurry prior to discharge. Next, the manhole entrance at the top of the cell was sealed except for a slot to which the Inclined Froth Washer (IFW) was attached.



Figure 1. View of MRC (Providence One Installation) Showing Feed Input Arrangement

Outside the cell, the main feed line passes through an 8-inch static mixer for mixing of slurry and chemical frother. The feed line then splits four ways and air is injected into each stream just before a four-inch inline static mixer, which entrains the air with the feed slurry just upstream of the feed manifolds. Static mixers use a series of blades that both divide and cause counter-rotation of the flow (Koflo, 2005) creating much smaller air



bubbles than would normally be formed within a smooth pipe. Combinations of 4-inch and 8-inch static mixers were tested.

Gravity is used to remove waste slurry from the MRC requiring the cell to be elevated. Fluid level in the test circuit cell was controlled by means of a butterfly valve in the waste discharge line. A pinch valve was also installed in parallel with the butterfly valve. Although considerable effort went toward automated control of the pinch valve using a fluid level sensor designed to measure changes in capacitance of the cell, throughout all of the testing done during this project, fluid level in the MRC was manually controlled with the butterfly valve.

The remainder of the test circuit consisted of pumps, motors, piping and a conditioning tank. Four R.S. Corcoran 4X3 pumps, each capable of 350-gpm at 60-psi, supplied feed to the MRC. Each pump was powered by a 40-hp 480-volt motor. Feed slurry was mixed and conditioned in a 3,000-gallon HDPE tank. For the most part, piping was Schedule 40 PVC in 4-inch, 6-inch and 8-inch sizes. The completed test circuit is shown in Figure 2.



Figure 2. MRC Test Circuit at DSI Laboratory in Champaign, IL

Once the test circuit was established, several fine coal samples from various operations, including Freeman Energy's Crown III Mine, CONSOL's Rend Lake Mine, and DSI's Providence One coal slurry pond recovery operation at a mine site in Western Kentucky, were tested. However, without a continuous stream of fresh feed material, testing was limited because of solids recirculation. The testing did satisfy the investigators that the 1,000-gallon MRC was capable of recovering fine coal at acceptable levels of yield and recovery.

### **Design, Build and Test a Scaled-up MRC System in the Field**

Meetings with Freeman Energy management personnel at the Crown III Mine took place during the month of June 2005 to finalize plans for a demonstration of the 1,000-gallon MRC. The mine agreed to reroute the underflow from sieve bends dewatering the plant's

polishing cyclone product to a test site located between the plant and its static thickener. A plan view of the test site is shown in Figure 3. DSI hired contractors approved by mine management to pour a concrete pad, build a support stand for the MRC, install the feed pump and complete electrical terminations. This work commenced on July 18, 2005 and was completed by August 19, 2005. Figure 4 shows the MRC and the pump and conditioning tank located below the MRC at the Freeman Energy demonstration site.

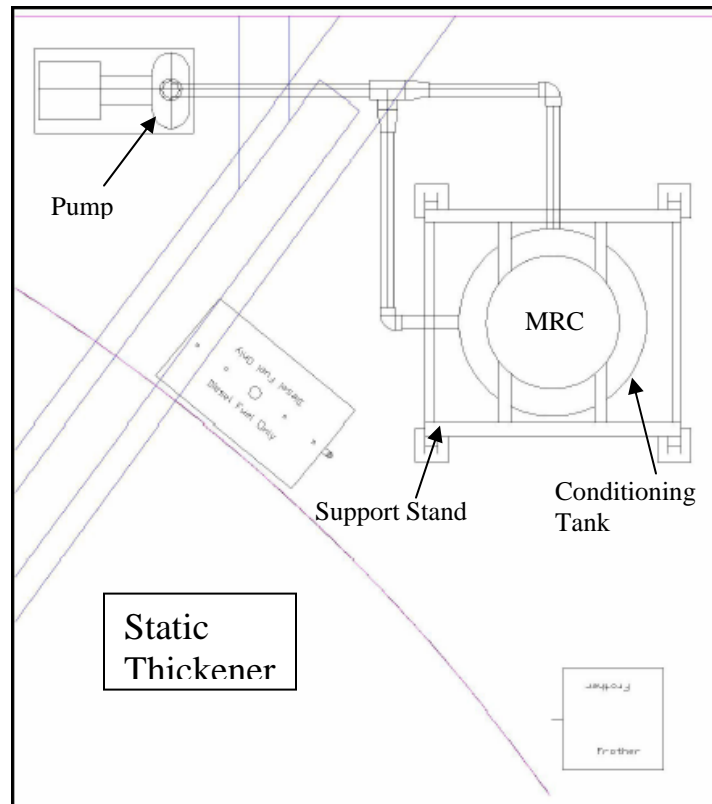


Figure 3. Plan View of the Freeman MRC Demonstration Site

The demonstration circuit included the MRC, the conditioning tank and all of the TMEs and static mixers from the test circuit. Other components such as the MRC support stand, the IFW and the feed pump were fabricated or newly purchased specifically for the demonstration. The ISGS built a 36-inch wide, 4-inch tall, 18-inch deep IFW inclined 40-degree from horizontal based on demonstration results of that technology at another site. A Durco Mk III 75-hp centrifugal pump was purchased for the demonstration. It had a 14-inch impellor and was rated at 1,000-gpm at 60-psi or 140 feet of head. Two chemical reagent pumps and a 300-gallon double-wall diesel fuel tank were purchased to supply flotation chemicals. The mine provided a portable air compressor to supply air.



Figure 4. MRC Installation at Freeman Energy Demonstration Site

The location adjacent to the plant's static thickener allowed the MRC to be positioned so that concentrate and waste streams could gravity flow to the thickener after processing. This required a support stand 18 feet in height. The stand was designed to allow the conditioning tank to fit beneath the MRC in a stack arrangement, which saved floor space. A tee and two valves were inserted into the feed line originating on the third floor of the preparation plant to divert the sieve bend underflow from the thickener to the conditioning tank.

### **Calibration of Instrumentation**

Level in the MRC was measured by a pressure transducer connected to an isolation ring mounted on the flange at the reject port. A 4-to-20-milliamp signal from the transducer corresponded to a range of 0 to 150 inches above the isolation ring. Factory calibration was verified by filling the MRC to the top crease (92 inches) and then noting the transducer signal at the known heights of each row of eductors (79 inches, 67.4 inches, 59.2 inches, and 51.8 inches) as the cell was drained. The isolation ring calibration was verified weekly as shown in Figure 5. As with the test circuit, fluid level in the cell was manually controlled with a butterfly valve in the reject discharge line.

Paddle wheel flow meters were installed in the feed line from the plant to the conditioning tank, in the pump discharge line feeding the MRC, and in the MRC waste discharge line. The paddle wheel flow meter measures the number of pulses generated in a given time by a series of magnets mounted on the paddle wheel. Because of the high percent solids in the plant feed line, that flow meter was rendered unusable by the

accumulation of material around the device. It was later noted that the accumulated material included ferrous particles that had jammed the magnets. Thus, the plant feed rate had to be determined by measuring level change in the conditioning tank. At the beginning of each testing session, after feed material was diverted from the plant and before make-up water was turned on, a conditioning tank level change over a 10-minute period was measured. Each one-inch change in level represents 27.52 gallons of plant feed.

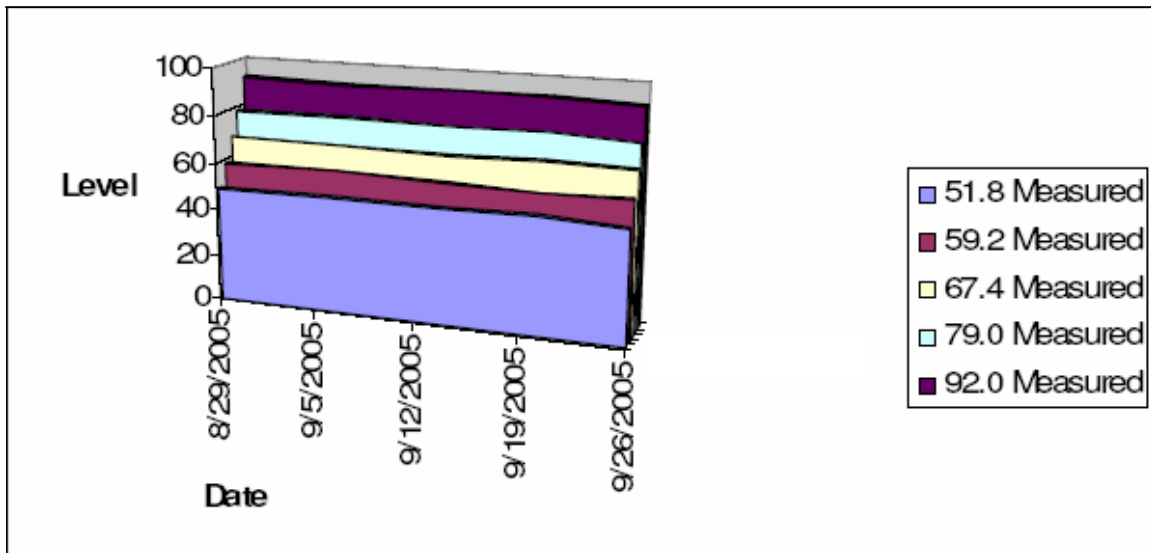


Figure 5. MRC Level Measurement Verification

A transmitter attached to the paddle wheel converts the pulses generated by the flow of fluid through the paddle wheel into gallons-per-minute (gpm) using scaling factor known as a K-value. The number of pulses divided by the K-value equals the number of gallons flowing past the element for a given time period. The K-value for the MRC feed line was determined by a timed measurement of flow from the conditioning tank. First, the conditioning tank was filled with water. The water was then pumped from the conditioning tank for a measured period of time. The level change in the conditioning tank during that time period was measured. Multiplying the level change by 27.52 gallons per inch and dividing by the measured time period yielded a flow rate for the feed pump. The K-value was then determined by dividing the number of paddle wheel pulses during the measured time period by the calculated flow rate. This K-value was programmed into the MRC feed flow meter. The K-value for the flow meter on the MRC reject line was determined by pumping water to the MRC and manually adjusting the discharge valve on the reject line until a steady level was maintained within the MRC. In this state, the MRC feed and reject rates are equivalent and the following equation is used to determine the K-value for the reject flow meter:

$$\text{Actual K-value} = \text{Pre-set K-value} \times (\text{Predetermined Feed Rate} \div \text{Indicated Flow Rate})$$

The actual K-value is then programmed into the MRC reject flow meter and it becomes the pre-set K-value. The process of filling the MRC to a steady level is then repeated and the reject flow meter K-value adjusted until both flow meters indicate the same value.

The clean coal concentrate flow rate proved difficult to measure directly because the IFW attachment did not have uniform flow across its rectangular cross section. Open channel methods of measuring concentrate flow rate were investigated, but in the end, concentrate flow rate was measured indirectly as the difference in flows between the MRC feed and the MRC reject. This required accurate measurement of feed and reject rates, hence the comparative flow rates from both meters at static fluid level in the MRC were checked on a weekly basis during the testing period.

The metering pump supplying the conditioning tank with diesel fuel collector was a variable speed piston pump capable of 20 gallons per hour at 20-psi. The piston stroke distance and speed were manually controlled. Calibration of the pump indicated that at full stroke, increasing the speed past 80% of the maximum speed resulted in decreased flow. A regression analysis of the calibration at full stroke (100% or maximum) produced the following equation for flow rate of the collector pump:

$$\text{Flow Rate (gpm)} = 11.2387868 \times \text{Pump Speed (expressed in \% of maximum)}$$

Another metering pump injected frother to the intake line of the MRC feed pump. This variable speed piston pump was capable of supplying 4.5 gallons per hour at 50-psi and it was also controlled manually. A regression analysis of calibration at 85% of full stroke produced the following equation for flow rate of the frother pump:

$$\text{Flow Rate (gpm)} = 2.5327832 \times \text{Pump Speed (expressed in \% of maximum)}$$

A 1¼-inch hose supplied air from the portable compressor to tees in each vertical feed manifold. The tees were installed just upstream of the static mixers. A ball valve was used to manually regulate the flow of air, which was measured by a pressure regulated flow meter. The mine also provided a fresh water line to supply make-up water to the conditioning tank. Level in the conditioning tank was manually controlled by means of a butterfly in the freshwater line.

### **Sampling Procedures**

Valved sampling points were installed throughout the demonstration circuit to collect samples during testing. Each sample point had its own challenge because of line pressure, flow rates, or the amount of solids present. To keep the sampling process as uniform as possible, DSI developed a standardized sample tracking sheet that was used to record all relevant information for each sample collected.

Feed to the MRC was sampled on the discharge side of the feed pump where the feed slurry was at approximately 60-psi. The sample point consisted of a 2-inch ball valve on

the down side of a pipe tee in the feed line. To collect this sample, the ball valve was opened and the sample line allowed to clear itself of any buildup of solid material. Then, a bucket was placed under the sample pipe and filled without allowing any overflow. Because solids in the MRC feed tended to quickly settle in the sample bucket, any slurry overflow would have distorted the percent solids being measured.

A tee with a 2-inch down-facing port connected to a 2-inch ball valve was installed in a horizontal stretch of the reject line immediately below the MRC. The reject sample was collected by opening the valve and allowing the line to flush before placing a sample bucket to collect the sample. Because of the high volume of slurry at this point, the bucket filled almost instantly and overflow was unavoidable. However, settling could not occur with such rapid, turbulent filling and overflow was not considered a problem.

To sample the concentrate, a 3-inch tee was installed on the line coming from the concentrate trough to the static thickener, with a ball valve and piping to within 5 feet of the ground connected to the down-facing port of the tee. Once test runs began, the valve was left open allowing a continuous flow of froth down the line. This served as a visual indicator that the cell was functioning properly, which was difficult to determine in any other way because of the height of the cell and the fact that the plant only ran in the evening and it was usually dark during test runs. As with the plant feed sample, care was taken not to overflow the sample bucket when collecting this sample so as to not distort the percent solids being measured.

Before each test run, a sample of feed from the plant was collected at a sample port in the line from the plant just before it entered the conditioning tank. This sample was collected before any makeup water was turned on so that the percent solids coming from the plant could be determined. Because this material was typically in the 30% solids range, the sample port had to be carefully flushed before collecting the sample and sample overflow was avoided so as not to distort the percent solids being measured.

Samples were collected in pre-marked containers and immediately sealed, then carefully transported to DSI's facility and sorted by sample type and number. The letters 'F', 'C' and 'R' were used to designate feed, concentrate and reject, respectively. A standard procedure was established and followed in preparation of all samples. The procedure describes methods for decanting, filtering and drying samples as well as documenting solids weight, total volume of sample water, and the pH of sample water. Once dry, a sample was ground to an appropriate standard size and sent off for analysis. All samples were sent to SGS Laboratories for short proximate analysis to determine percent ash, percent sulfur and heat content (Btu per pound) with results typically returned in three days. Duplicates of some of the optimization and Box-Behnken samples were also sent to the ISGS for ash and sulfur determination on a moisture free basis. This provided a comparison check on the SGS results (Manrique et al., 2005).

### **Flotation Release Analysis**

To measure MRC performance, a release analysis of the material being processed was

required. Flotation release analyses are used to determine the ultimate recovery-grade relationship for a given coal (Honaker et al., 1996). It is the most suitable method for measuring flotation device performance because it is based on material surface properties (Adel and Wang, 2005). Comparing MRC test results with the flotation release curve generated by the flotation release analysis gives an indication of MRC efficacy.

Freeman Energy's Crown III plant had two possible sources of feed available for testing in the MRC. Underflow from the sieve bends dewatering the polishing cyclone concentrate was found to have 19% solids, ash content of 39.38% and sulfur content of 6.65%. Effluent from centrifugal dryers dewatering the sieve bend overs was found to have 27% solids, ash content of 30.14% and sulfur content of 4.46%. While the dryer effluent would have provided a better feed material for the MRC demonstration, the decision was made to use the material from the sieve bend underflow as the feed to the MRC because the sieve bend underflow piping in the plant was more easily accessible to the test site than was the centrifuge dryer effluent.

Consequently, two flotation release analyses were conducted on samples of the sieve bend underflow material to determine the ultimate recovery-grade relationship. The first analysis was done at Southern Illinois University in Carbondale, Illinois on a mixture of plant feed samples taken on September 19, 2005 and September 26, 2005. The release analysis results indicated the lowest achievable concentrate ash was 18.49%. Considering this result to be atypical, a second sample was collected on October 10, 2005 and sent to the University of Kentucky for analysis. The results from that analysis indicated that a concentrate ash as low as 14.87% was achievable. Both release analyses are plotted in Figures 6 and 7. As a verification check, a portion of the sample sent to Kentucky was analyzed in DSI's laboratory. This analysis consisted of a simple flotation test using diesel fuel collector and Zinkan's O'Brien 509 frother. Concentrate collected from the first float was re-floated three more times and the tailing removed. The final concentrate contained 12.36% ash and 5.28% sulfur, acceptable values that justified processing sieve bend underflow material in the MRC.

### **Selection of Chemicals**

Four different frothers were tested in a simple two-stage flotation test using a 2-liter Denver flotation cell at the ISGS laboratory to determine which one should be used for the Freeman demonstration. Three of the frothers were provided by Zinkan Industries – O'Brien 504 frother which is glycol based and O'Brien 502 and 509 frothers which are alcohol based. The fourth frother was CM 630, a glycol based frother provided by Stockhausen. The two most common collectors used for coal flotation are No. 2 fuel oil (diesel fuel) and kerosene (Leonard, 1991). Although kerosene was used in tests to select the frother, diesel fuel was selected for the Freeman demonstration based on lower costs and better availability.

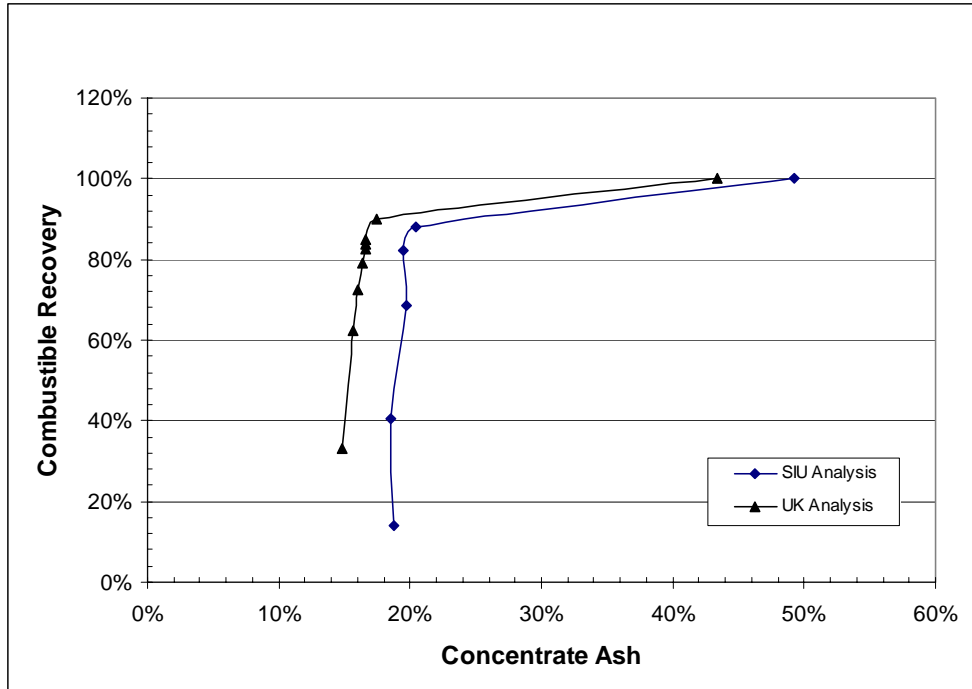


Figure 6. Release Analysis Showing Concentrate Ash versus Combustible Recovery

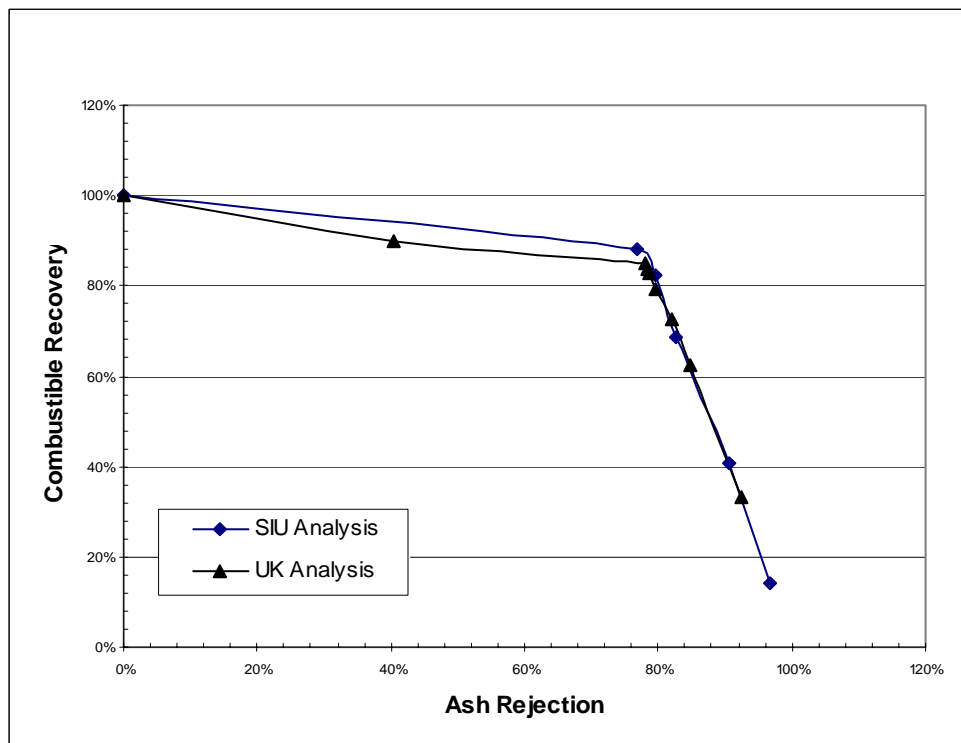


Figure 7. Release Analysis Showing Ash Rejection versus Combustible Recovery



In our exploratory laboratory tests in a 2-Liter Denver Cell the glycol-based O'Brien frother O'B Froth 504, yielded a more stable froth, which is typical for glycol-based frothers. The O'B Froth 502 is a new product that has been tested successfully in southern West Virginia. Stockhausen's CM 630 was tested because it is readily available and costs less than their CM 650.

The procedure for testing the different frothers consisted of mixing 100 grams of sieve bend underflow sample with tap water for about two minutes in the bench top flotation cell. Kerosene in the amount of 0.06% (600 grams/ton) of solids was then added and allowed to condition the slurry for three minutes. Make-up water was added, as required, to raise the slurry level to 2,000-mL mark in the flotation cell. Frother was then added at the rate of 800 grams/ton of solids and after a brief one minute mixing period, the air inlet was opened. After about one minute of flotation, no additional coal floated and a white froth appeared. The concentrate from this first stage was then repulped in water and re-floated in a second stage. No collector was added to the pulp when O'Brien frothers were used, but Stockhausen's CM 630 frother required additional collector to make the coal float again because coal in the first stage concentrate tended to detach from air bubbles during the second stage. In this particular case, reject produced during cleaning of the rougher concentrate was mixed with half the amount of kerosene added to the raw feed and re-floated producing a second concentrate and middling. Frother was added during all re-cleaning efforts.

Results from the two-stage flotation test, shown in Table 2, were comparable for all of the different frothers, but they raised concerns about the quality of the selected feed material from a floatability standpoint. However, the decision to use the sieve bend underflow material had already been made because that material was more accessible to the test site, and changing it was not an option. Therefore, it was determined to use O'Brien 509 frother because it is Zinkan Industries' mainstay product and it produces a suitable froth at a reasonable cost.

Table 2. Results from Flotation Tests Used to Select Frother

| Frother     | Feed  |          | Concentrate |          | Reject |          |
|-------------|-------|----------|-------------|----------|--------|----------|
|             | % Ash | % Sulfur | % Ash       | % Sulfur | % Ash  | % Sulfur |
| O'Brien 502 | 41.32 | 6.98     | 12.27       | 5.70     | 67.09  | 7.55     |
| O'Brien 504 | 41.97 | 6.38     | 14.14       | 6.44     | 70.82  | 6.11     |
| O'Brien 509 | 42.25 | 6.92     | 12.31       | 5.39     | 68.31  | 7.66     |
| CM 630      | 43.68 | 7.22     | 11.67       | 4.50     | 71.05  | 8.38     |

## RESULTS AND DISCUSSION

### **Task 1. Determine the impact of scaled-up dimensions on MRC performance.**

After reviewing data and results from the initial proof-of-concept, laboratory-scale project, the project team of DSI and the ISGS purchased a 1,000-gallon tank, which was made into a large-scale MRC test unit during the second month of the project. After a brief period of testing the 1,000-gallon MRC in a test circuit at the DSI laboratory in Champaign, Illinois, it was deemed impractical to build multiple 1,000 gallon cells for comparing different shapes and other arrangements. While preliminary test results suggested the 1,000-gallon MRC was functional, convincing qualitative data were difficult to generate with the test circuit as had been hoped. Consequently, building some smaller MRCs in the 85-gallon to 250-gallon size range to do scale-up testing was briefly considered. With opportunities for commercial application of the technology beckoning, the project team decided that the smaller sizes would be impractical and the 1,000-gallon MRC could be made functional with sufficient effort.

### **Task 2. Complete the design, fabrication and installation of a scaled-up version of the cylindrical MRC with conical bottom and domed top equipped with ISGS Froth Washer.**

The 1,000-gallon tank that was purchased was cylindrical with a conical bottom and domed top. The tank was modified to include an 8-inch discharge in the bottom of the conical section, 16 holes (4 sets of 4) in the sides of the cylindrical section, and a slot for the ISGS Inclined Froth Washer to be attached to the domed top. For initial testing of the larger MRC, a test circuit consisting of four 350-gpm pumps, a 3,000-gallon conditioning tank, and all of the associated piping and valves was assembled and tested at the DSI laboratory.

### **Task 2A. Installation of the 1,000 gallon MRC at Freeman's Crown III Prep Plant.**

During the eighth month of the project, Freeman Energy visited the DSI facility and observed the test circuit in operation. This resulted in an agreement with Freeman Energy to host a field demonstration of the 1,000-gallon MRC and the project was extended to allow sufficient time to accomplish this. Over the course of the next three months, the original 1,000-gallon MRC from the test circuit was moved to Freeman Energy's Crown III Mine and a fine coal recovery test circuit was assembled between the mine's preparation plant and static thickener.

### **Task 3. Automation of the cell.**

During the first six months of the project, the ISGS dedicated one of their staff to the task of automating the MRC. Specifically, the intent was to have a level sensor communicating to an automated pinch valve on the waste discharge line of the cell to control the fluid and froth levels in the MRC. When the person assigned to this task left the project, the decision was made to use manual control and eliminate this task.

**Task 4. Determination of the effects of cell characteristics on the throughput and quality of the cleaned coal product.**

Cell characteristics include size and shape of the MRC and positioning of the feed eductors. The initial proof-of-concept project indicated that a cylindrical cell with conical bottom and domed top would function well and that shape was selected for this project. Further investigation of MRC size and shape is necessary but beyond the scope of this project because it is difficult to do in the field. Establishing eductor size, positioning and feed orientation at the DSI laboratory test circuit was necessary because changing those parameters on site at the mine was not practical or safe. Further testing of these parameters is needed but could not be completed as part of this project.

Coal slurry testing with the test circuit while at DSI evaluated eductor positioning at various angles (90°, 45°, 30°, 22.5° and 11.25°) as measured from an imaginary line tangent to the cell wall at the eductor opening. Results indicated that angles greater than 22.5° greatly increased the depth of the vortex within the cell, which was undesirable. It was also determined that the carrying capacity of the MRC was between 10 and 13 tons of coal per hour.

Eductor size is another variable parameter requiring evaluation that could not be made a practical part of this investigation. Eductor size has a direct effect on feed rate to the MRC. To achieve the desired 1,000-gpm feed rate with a minimum number of feed ports in the cell wall, 1½-inch eductors were the smallest size that could be used. Flow rates from eductors up to 3-inch in size were considered but the larger the eductor, the greater the interactive effects within the cell. Fluid dynamics within the cell is an area that requires further exploration beyond the scope of this project. Therefore, the investigators selected the smallest eductor size as a fixed parameter for this project.

**Task 5. Determination of the effects of operating conditions such as feed rate, aeration intensity, aeration mechanism and retention time on the quality of the clean coal product.**

Seven additional controllable parameters were identified for investigation as part of this project. The variable range of each parameter is shown in Table 3.

Table 3. Controllable Operating Parameters and Their Ranges

| <b>Parameter</b>                 | <b>Range</b>                          |
|----------------------------------|---------------------------------------|
| Slurry Feed Rate (gpm)           | 0-1,000                               |
| Solids Content of Slurry (%)     | 0-20                                  |
| Fluid Level in the MRC (gallons) | 0-1,000                               |
| Frother Dosage (lb/ton)          | 0.1-0.5                               |
| Collector Dosage (lb/ton)        | 2.0-6.0                               |
| Air Feed Rate (0-200 scfm)       | 0-200                                 |
| Static Mixer Configuration       | None, 4-inch, 8-inch, 4-inch + 8-inch |

To test different feed rates, the initial plan was to manually adjust butterfly valves on the discharge side of the feed pump. Closing one valve in the pump discharge line directly feeding the MRC would restrict the actual feed rate going to the cell. Opening a second valve at a tee in the discharge line would recirculate some of the feed material back to the mixing tank. Both of these options proved unworkable. Closing the pump discharge valve even a small amount proved too stressful for the pump causing the seals to heat up significantly. Opening the second valve recirculated not only feed material but also frother, promoting froth formation in the mixing tank and making it impossible to accurately measure frother dosage. Consequently, slurry feed rate was eliminated as a test parameter.

Measuring the solids content of the plant feed material indicated that the mixing tank was receiving between 2.5 and 5.0 tons per hour of solid material. Mixing that amount of solids with sufficient water to achieve the desired 1,000-gpm feed rate reduced the percent solids being fed to the MRC to less than 2%, well below the recognized 12% optimum or even the average 3% to 5% usually seen in the US (Leonard, 1991). Because lowering pulp density generally corresponds to lower recovery rates, it was decided that lowering solids content to less than the maximum achievable level of 2% would not yield any useful data. Consequently, solids content was also eliminated as a test parameter (Manrique et al., 2005).

## OPTIMIZATION TESTING

Optimization tests on the remaining five parameters commenced with feed, concentrate and reject samples collected for each test and evaluated for heating value (Btu), ash and sulfur content. Data are provided in Appendix 1. For each test set, one parameter was altered and changes in product ash content, recovery and ash rejection were determined.

### Fluid Level in the MRC

Test conditions for determining the optimum operating fluid level for the MRC are shown in Table 4. During these tests, fluid level in the cell as measured from the bottom discharge flange of the MRC was altered between 90 and 105 inches in non-linear fashion with all other parameters held constant. Test results, shown in Table 5, indicate that maintaining the MRC fluid level at 90 inches produced the best results. The results, plotted in Figure 8, are compared against the release analysis curves described earlier in Figure 7 (Manrique et al., 2005).

Because of the time lag involved in receiving test results and the need to continue optimization testing before they became available, visual observations during the level optimization testing were used to establish a fixed MRC fluid level of 95 inches for the other optimization tests sets. During the last test set, it was discovered that changes in the feed material to the cell also changed the optimum operating fluid level. Consequently, a follow-up series of optimization tests was conducted after all other testing to explore a different avenue for controlling the MRC fluid level. Those results will be reported later.

Table 4. MRC Level Optimization Test Conditions

| Test / Sample# | Fluid level from bottom of MRC | Air Flow. | F* Stk | F* Spd | C** Stk | C** Spd | 4'' & 8'' Mixer |
|----------------|--------------------------------|-----------|--------|--------|---------|---------|-----------------|
|                | (inches)                       | (scfm)    | (%)    | (%)    | (%)     | (%)     |                 |
| FRE-4          | 90                             | 30        | 70     | 100    | 100     | 80      | Present         |
| FRE-5          | 92                             | 30        | 70     | 100    | 100     | 80      | Present         |
| FRE-6          | 97                             | 30        | 70     | 100    | 100     | 80      | Present         |
| FRE-7          | 100                            | 30        | 70     | 100    | 100     | 80      | Present         |
| FRE-16         | 103                            | 30        | 70     | 100    | 100     | 80      | Present         |
| FRE-17         | 105                            | 30        | 70     | 100    | 100     | 80      | Present         |

\* F Stk = Frother pump stroke; F Spd = Frother Pump speed / stroke frequency;  
 \*\*C Stk = Collector pump stroke; C Spd = Collector Pump speed / stroke frequency

Table 5. MRC Level Optimization Test Results

| Sample | Fluid Level | Feed Ash | Concentrate Ash | Combustible Recovery | Ash Rejection | Efficiency |
|--------|-------------|----------|-----------------|----------------------|---------------|------------|
|        | (inches)    | (%)      | (%)             | (%)                  | (%)           | (%)        |
| FRE-4  | 90          | 32.71    | 12.44           | 75.5                 | 77.9          | 61.3       |
| FRE-5  | 92          | 38.74    | 16.33           | 50.9                 | 84.3          | 55.6       |
| FRE-6  | 97          | 35.53    | 19.22           | 52.4                 | 77.4          | 44.2       |
| FRE-7  | 100         | 33.23    | 17.07           | 83.1                 | 65.6          | 56.3       |
| FRE-16 | 103         | 39.22    | 23.10           | 85.9                 | 60.0          | 53.5       |
| FRE-17 | 105         | 33.47    | 23.94           | 93.1                 | 41.8          | 39.3       |

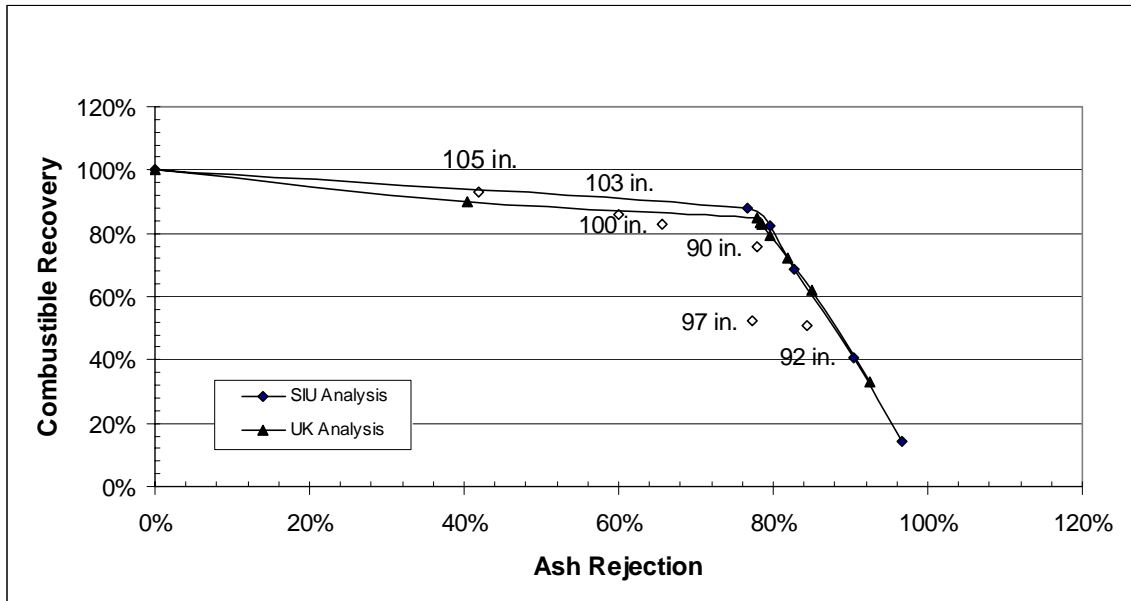


Figure 8. MRC Level Optimization Data Plotted With Release Analysis Curves

## Air Feed Rate

The effect of air feed rate was tested at 15, 20, 25, 30, 50 and 70 scfm delivered at 80-psi with other conditions for these tests shown in Table 6. An aeration rate of 23 scfm, equivalent to 17% of the total cell volume, is consistent with aeration rates used in other subaeration cells. Results of the air feed rate optimization tests are shown in Table 7 and plotted in Figure 9. There was a significant reduction in concentrate ash between 15 and 25 scfm but very little change between 25 and 70 scfm. The highest recovery was obtained at 25 scfm and it declined as air feed rate was increased. These data suggest that the MRC is saturated with air at an air feed rate of 25 scfm, and increasing the air feed rate beyond that amount causes the bubbles to coalesce and detach coal. Ash rejection generally increased as the aeration flow rate increased (Manrique et al., 2005).

Table 6. Air Feed Rate Optimization Test Conditions

| Test Sample (#) | Air Flow (scfm) | Air Pres. (psi) | F Stk (%) | F Spd (%) | C Stk (%) | C Spd (%) | MRC Fluid Level (inches) | 4'' & 8'' Mixer |
|-----------------|-----------------|-----------------|-----------|-----------|-----------|-----------|--------------------------|-----------------|
| FRE-20          | 15              | 80              | 85        | 100       | 100       | 80        | 95                       | Present         |
| FRE-19          | 20              | 80              | 85        | 100       | 100       | 80        | 95                       | Present         |
| FRE-18          | 25              | 80              | 85        | 100       | 100       | 80        | 95                       | Present         |
| FRE-11          | 30              | 80              | 85        | 100       | 100       | 80        | 99                       | Present         |
| FRE-9           | 50              | 80              | 85        | 100       | 100       | 80        | 95                       | Present         |
| FRE-10          | 70              | 79              | 85        | 100       | 100       | 80        | 95                       | Present         |

Table 7. Air Feed Rate Optimization Test Results

| Sample | Air Flow (scfm) | Feed Ash (%) | Concentrate Ash (%) | Combustible Recovery (%) | Ash Rejection (%) |
|--------|-----------------|--------------|---------------------|--------------------------|-------------------|
| FRE-20 | 15              | 42.34        | 35.19               | -2.9                     | 102.1             |
| FRE-19 | 20              | 47.76        | 31.26               | 80.7                     | 59.9              |
| FRE-18 | 25              | 38.03        | 21.89               | 88.7                     | 59.5              |
| FRE-11 | 30              | 38.58        | 21.83               | 83.8                     | 62.7              |
| FRE-9  | 50              | 38.42        | 20.86               | 71.1                     | 70.0              |
| FRE-10 | 70              | 44.50        | 21.15               | 60.6                     | 79.7              |

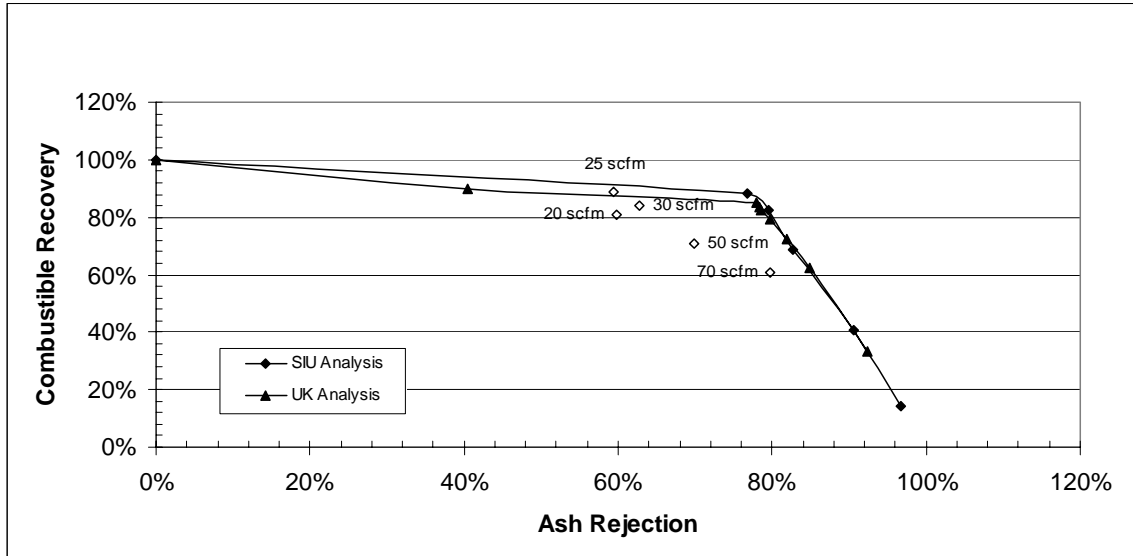


Figure 9. MRC Air Flow Optimization Data Plotted With Release Analysis Curves

#### Frother Feed Rate

After consultation with the chemical supplier, frother dosage was initially set at 0.15 lb/ton or 4.54 g/min based on a feed rate of 2.5 to 5.0 tons of solids per hour being delivered to the MRC. Based on poor run-in test results (see FRE-1 and FRE-3 in Appendix 1), larger dosages were used in optimization testing as shown in Table 8. The greatest combustible recovery occurred at a dosage of 190 g/min as shown in Table 9 and Figure 10.

Table 8. Frother Feed Rate Optimization Test Conditions

| Test Sample (#) | Frother Dose (g/min) | Air Pres. (psi) | F Stk (%) | F Spd (%) | C Stk (%) | C Spd (%) | Fluid Level (inches) | 4" & 8" Mixer |
|-----------------|----------------------|-----------------|-----------|-----------|-----------|-----------|----------------------|---------------|
| FRE-9           | 253                  | 80              | 85        | 100       | 100       | 80        | 95                   | Present       |
| FRE-14          | 190                  | 80              | 85        | 75        | 100       | 80        | 93                   | Present       |
| FRE-15          | 63                   | 80              | 85        | 25        | 100       | 80        | 96                   | Present       |

Table 9. Frother Feed Rate Optimization Test Results

| Sample | Frother Feed Rate (g/min) | Feed Ash (%) | Concentrate Ash (%) | Combustible Recovery (%) | Ash Rejection (%) |
|--------|---------------------------|--------------|---------------------|--------------------------|-------------------|
| FRE-9  | 253                       | 38.42        | 20.86               | 71.1                     | 70.0              |
| FRE-14 | 190                       | 42.43        | 20.96               | 84.9                     | 69.4              |
| FRE-15 | 63                        | 50.83        | 29.13               | 77.2                     | 69.3              |

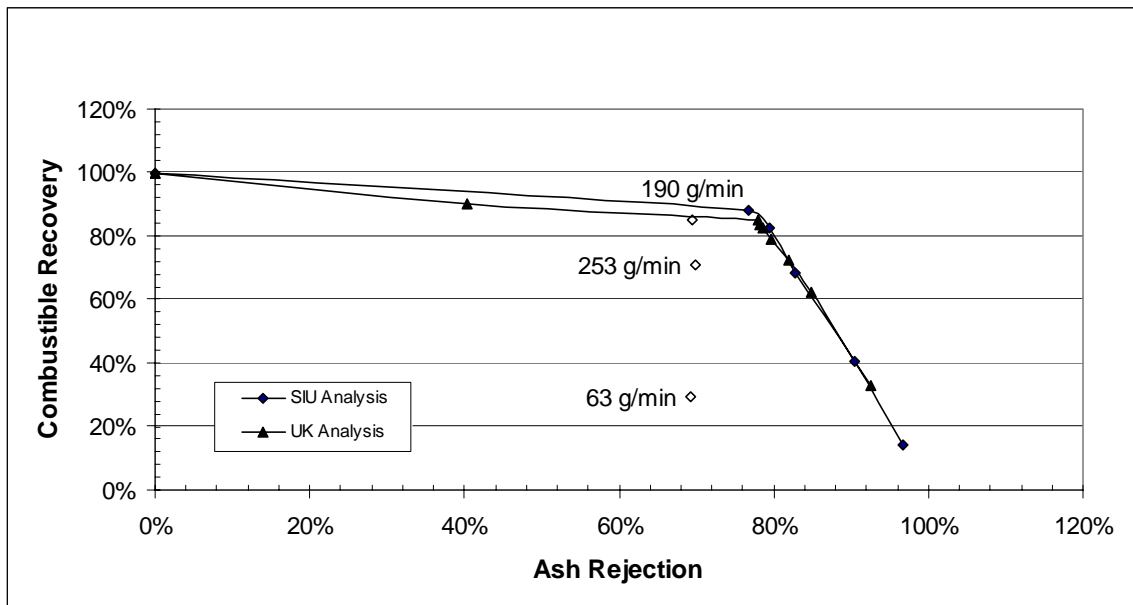


Figure 10. Frother Feed Rate Optimization Data Plotted With Release Analysis Curves

#### Collector Feed Rate

Collector dosages for Illinois coals are typically between 0.5 and 4.0 kg/ton (Leonard, 1991). Again based on run-in test results, collector dosage was tested over a range of 450 g/min to 900 g/min as shown in Table 10. The smallest collector dose yielded the best ash rejection and moderate recovery as seen in Table 11 and Figure 11.

Table 10. Collector Feed Rate Optimization Test Conditions

| Test Sample (#) | Collector Dose (g/min) | Air Pres. (psi) | F Stk (%) | F Spd (%) | C Stk (%) | C Spd (%) | Fluid Level (inches) | 4' & 8" Mixer |
|-----------------|------------------------|-----------------|-----------|-----------|-----------|-----------|----------------------|---------------|
| FRE-9           | 900                    | 80              | 85        | 100       | 100       | 80        | 95                   | Present       |
| FRE-12          | 675                    | 80              | 85        | 100       | 100       | 60        | 95                   | Present       |
| FRE-13          | 450                    | 80              | 85        | 100       | 100       | 40        | 95                   | Present       |

Table 11. Collector Feed Rate Optimization Test Results

| Sample | Collector Feed Rate (g/min) | Feed Ash (%) | Concentrate Ash (%) | Combustible Recovery (%) | Ash Rejection (%) |
|--------|-----------------------------|--------------|---------------------|--------------------------|-------------------|
| FRE-9  | 900                         | 38.42        | 20.86               | 71.1                     | 70.0              |
| FRE-12 | 675                         | 42.72        | 24.72               | 86.2                     | 62.0              |
| FRE-13 | 450                         | 47.09        | 22.56               | 74.8                     | 75.5              |



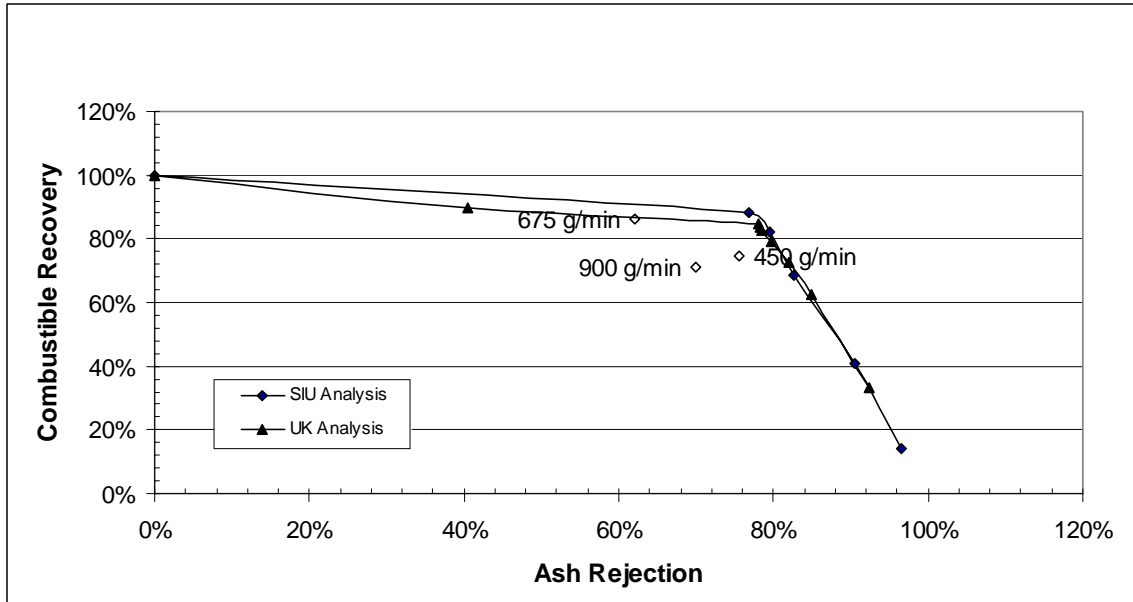


Figure 11. Collector Feed Rate Optimization Data Plotted With Release Analysis Curves

### Static Mixer Configuration

To select the best static mixer configuration, three sets of tests were performed. In the first set, both the 8" and 4" mixers were installed. Because this was the condition for all previous optimization tests, additional tests were dispensed with and optimum data from early tests were used. In the second test set, the 8" mixers were removed leaving just the 4" mixers. In the third test set, the 4" mixers were removed leaving no mixers at all. Table 12 describes test conditions for static mixer optimization testing while Table 13 and Figure 12 show that the lowest ash content in the concentrate and the greatest combustible recovery were achieved when both 4" and 8" mixers were installed.

Table 12. Static Mixer Optimization Test Conditions

| Test Sample (#) | Static Mixer Configuration | Air Flow (scfm) | Air Pres. (psi) | F Spd (%) | C Stk (%) | C Spd (%) | Fluid Level (inches) |
|-----------------|----------------------------|-----------------|-----------------|-----------|-----------|-----------|----------------------|
| BB25            | 4" & 8"                    | 30              | 80              | 85        | 100       | 60        | 95                   |
| FRE-13          | 4" & 8"                    | 50              | 80              | 75        | 100       | 80        | 93                   |
| FRE-14          | 4" & 8"                    | 50              | 80              | 100       | 100       | 40        | 95                   |
| FRE-21          | 4" only                    | 30              | 80              | 85        | 100       | 60        | 95                   |
| FRE-22          | 4" only                    | 50              | 80              | 75        | 100       | 80        | 93                   |
| FRE-23          | 4" only                    | 50              | 80              | 100       | 100       | 40        | 95                   |
| FRE-24          | No mixers                  | 30              | 80              | 85        | 100       | 60        | 95                   |
| FRE-25          | No mixers                  | 50              | 80              | 75        | 100       | 80        | 93                   |
| FRE-26          | No mixers                  | 50              | 80              | 100       | 100       | 40        | 95                   |

Table 13. Static Mixer Optimization Test Results

| Sample   | Static Mixer Configuration | Feed Ash | Concentrate Ash | Combustible Recovery | Ash Rejection |
|----------|----------------------------|----------|-----------------|----------------------|---------------|
|          |                            | (%)      | (%)             | (%)                  | (%)           |
| FRE-13   | 4" & 8"                    | 47.09    | 22.56           | 74.8                 | 75.5          |
| FRE-14   | 4" & 8"                    | 42.43    | 20.96           | 84.9                 | 69.4          |
| FRE-BB25 | 4" & 8"                    | 41.59    | 28.35           | 89.9                 | 50.0          |
| FRE-21   | 4" only                    | 40.00    | 28.18           | 84.3                 | 49.6          |
| FRE-22   | 4" only                    | 36.88    | 24.72           | 86.8                 | 51.2          |
| FRE-23   | 4" only                    | 42.09    | 31.14           | 89.3                 | 44.4          |
| FRE-24   | No mixers                  | 45.29    | 30.79           | 72.8                 | 60.9          |
| FRE-25   | No mixers                  | 44.58    | 35.44           | 86.8                 | 40.7          |
| FRE-26   | No mixers                  | 46.12    | 35.47           | 84.9                 | 45.5          |

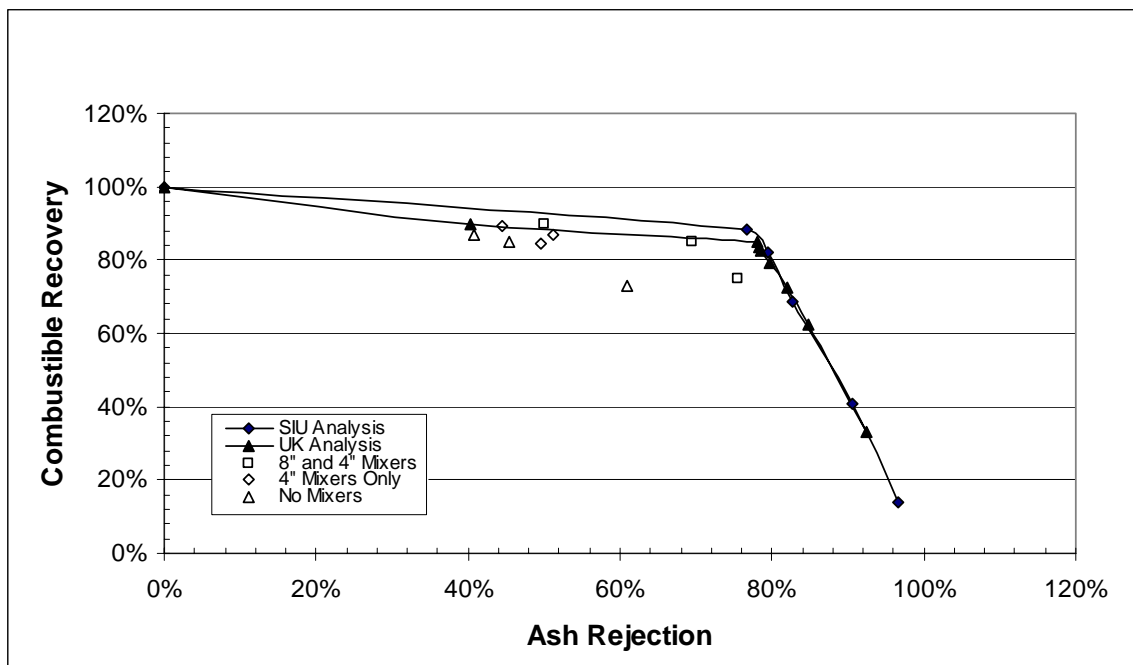


Figure 12. Static Mixer Optimization Data Plotted With Release Analysis Curves

## STUDY OF INTERACTIONS

Following the optimization testing, a Box-Behnken test matrix was designed to examine interaction effects between air flow, froth dosage, collector dosage and fluid level on ash and sulfur rejection and on combustible recovery. The range for each parameter in the Box-Behnken study is shown in Table 14 and the test matrix is shown in Table 15. The thirty tests were conducted in three blocks of ten over a period of two days beginning

October 5, 2005. Samples were sent to SGS Laboratories and to the ISGS Laboratory for analysis. Analysis results are provided in Appendix 1.

Table 14. Design Parameters for Box-Behnken Test Matrix

| <b>Parameter</b> | <b>Units</b> | <b>Low</b> | <b>Middle</b> | <b>High</b> |
|------------------|--------------|------------|---------------|-------------|
| Air Flow         | scfm         | 20         | 30            | 40          |
| Frother Dosage   | gram/minute  | 165        | 190           | 215         |
| Collector Dosage | gram/minute  | 562        | 674           | 787         |
| Fluid Level      | inches       | 95         | 99            | 103         |

Table 15. Box Behnken Test Matrix

| <b>Test #</b> | <b>Air Flow</b> | <b>Frother Dose</b> | <b>Collector Dose</b> | <b>Fluid Level</b> |
|---------------|-----------------|---------------------|-----------------------|--------------------|
| BB1           | 30 (0)          | 165 (-1)            | 562 (-1)              | 99 (0)             |
| BB2           | 40 (1)          | 190 (0)             | 674 (0)               | 95 (-1)            |
| BB3           | 30 (0)          | 215 (1)             | 562 (-1)              | 99 (0)             |
| BB4           | 20 (-1)         | 190 (0)             | 674 (0)               | 95 (-1)            |
| BB5           | 20 (-1)         | 190 (0)             | 674 (0)               | 103 (1)            |
| BB6           | 30 (0)          | 190 (0)             | 674 (0)               | 99 (0)             |
| BB7           | 30 (0)          | 190 (0)             | 674 (0)               | 99 (0)             |
| BB8           | 30 (0)          | 215 (1)             | 787 (1)               | 99 (0)             |
| BB9           | 30 (0)          | 165 (-1)            | 787 (1)               | 99 (0)             |
| BB10          | 40 (1)          | 190 (0)             | 674 (0)               | 103 (1)            |
| BB11          | 40 (1)          | 215 (1)             | 674 (0)               | 99 (0)             |
| BB12          | 30 (0)          | 190 (0)             | 674 (0)               | 99 (0)             |
| BB13          | 30 (0)          | 190 (0)             | 787 (1)               | 95 (-1)            |
| BB14          | 30 (0)          | 190 (0)             | 562 (-1)              | 103 (1)            |
| BB15          | 20 (-1)         | 165 (-1)            | 674 (0)               | 99 (0)             |
| BB16          | 30 (0)          | 190 (0)             | 674 (0)               | 99 (0)             |
| BB17          | 20 (-1)         | 215 (1)             | 674 (0)               | 99 (0)             |
| BB18          | 30 (0)          | 190 (0)             | 562 (-1)              | 95 (-1)            |
| BB19          | 40 (1)          | 165 (-1)            | 674 (0)               | 99 (0)             |
| BB20          | 30 (0)          | 190 (0)             | 787 (1)               | 103 (1)            |
| BB21          | 30 (0)          | 190 (0)             | 674 (0)               | 99 (0)             |
| BB22          | 20 (-1)         | 190 (0)             | 562 (-1)              | 99 (0)             |
| BB23          | 40 (1)          | 190 (0)             | 787 (1)               | 99 (0)             |
| BB24          | 30 (0)          | 165 (-1)            | 674 (0)               | 103 (1)            |
| BB25          | 30 (0)          | 215 (1)             | 674 (0)               | 95 (-1)            |
| BB26          | 30 (0)          | 215 (1)             | 674 (0)               | 103 (1)            |
| BB27          | 30 (0)          | 190 (0)             | 674 (0)               | 99 (0)             |
| BB28          | 20 (-1)         | 190 (0)             | 787 (1)               | 99 (0)             |
| BB29          | 30 (0)          | 165 (-1)            | 674 (0)               | 95 (-1)            |
| BB30          | 40 (1)          | 190 (0)             | 562 (-1)              | 99 (0)             |

Using StatEase™ DesignExpert® software, sample results were entered into an Analysis of Variance (ANOVA) program to assess interaction effects on ash and sulfur content of the concentrate and combustible recovery. ANOVA results for concentrate ash content suggest that only air flow and fluid level-air flow interaction are significant factors in predicting ash content. The analysis showed that at higher MRC fluid levels, more fluid with elevated ash levels was pushed out of the washer with the concentrate. This confirmed observations made during testing that fluid overflowing with the froth concentrate should be minimized. ANOVA results showed no significant correlation between any of the parameters and the sulfur content of the concentrate. This may suggest that, unlike the coarser product marketed by Freeman, a majority of the sulfur present in the fine product is organic sulfur that is bound in the coal and cannot be removed by flotation, or much of the fine pyrite present was also floating and contaminating the product. Finally, ANOVA results indicate that air flow rate is the only significant parameter in predicting combustible recovery.

### FOLLOW-UP TESTING

While running the third block of Box-Behnken tests, the samplers noted that the concentrate flow (determined by measuring the difference in MRC feed flow and MRC reject flow) was significantly greater than in previous tests as shown in Figure 13. The large change in concentrate flow required to maintain a specific level in the cell indicated that the idea of controlling the MRC based on fluid level was fundamentally flawed, and that a better control variable had to be found to regulate the cell.

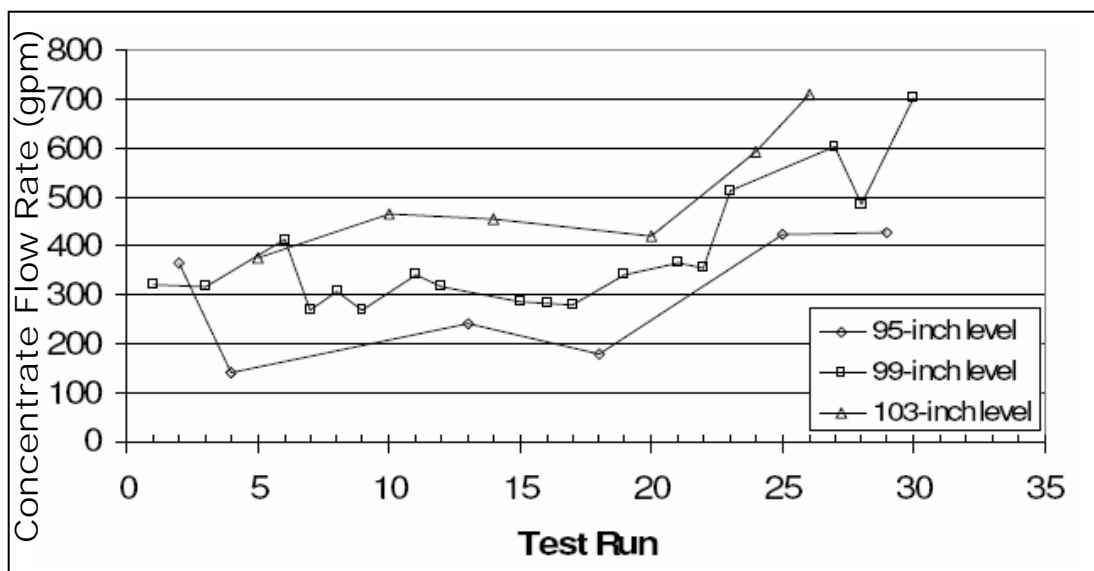


Figure 13. MRC Concentrate Flow Rates During Box-Behnken Testing

Further analysis of the six Box-Behnken tests conducted under identical conditions identified considerable variability in the quantity and quality of the feed. This led to the

hypothesis that maintaining a specific concentrate flow rate would be a better method of control for the cell. To test this hypothesis, eight follow-up tests were conducted with test conditions described in Table 16. All MRC variables were held constant at the previously determined optimum settings while varying the concentrate flow rate between 50 and 200 gallons per minute.

Results of these tests are provided in Table 17 and plotted in Figure 14. They clearly show that when concentrate flow was reduced from 200-gpm to 50-gpm in the presence of optimum doses of frother, collector and air flow, product ash decreased significantly while combustible recovery was maintained at optimum levels.

Table 16. Concentrate Flow Optimization Test Conditions

| Test Sample (#) | Concentrate Flow Rate (gpm) | Air Flow (scfm) | Air Pres. (psi) | F Spd (%) | C Spd (%) | 4" & 8" Mixer |
|-----------------|-----------------------------|-----------------|-----------------|-----------|-----------|---------------|
| FRE-29          | 28                          | 20              | 80              | 75        | 60        | Present       |
| FRE-30          | 77                          | 20              | 80              | 75        | 60        | Present       |
| FRE-31          | 162                         | 20              | 80              | 75        | 60        | Present       |
| FRE-32          | 195                         | 20              | 80              | 75        | 60        | Present       |
| FRE-33          | 201                         | 30              | 80              | 75        | 60        | Present       |
| FRE-34          | 151                         | 30              | 80              | 75        | 60        | Present       |
| FRE-35          | 104                         | 30              | 80              | 75        | 60        | Present       |
| FRE-36          | 51                          | 30              | 80              | 75        | 60        | Present       |

Table 17. Concentrate Flow Optimization Test Results

| Sample | Concentrate Flow Rate | Feed Ash | Concentrate Ash | Combustible Recovery | Ash Rejection |
|--------|-----------------------|----------|-----------------|----------------------|---------------|
|        | (gpm)                 | (%)      | (%)             | (%)                  | (%)           |
| FRE-29 | 28                    | 37.66    | 12.67           | 61.8                 | 85.1          |
| FRE-30 | 77                    | 38.11    | 14.97           | 56.3                 | 83.9          |
| FRE-31 | 162                   | 38.42    | 19.33           | 79.0                 | 69.7          |
| FRE-32 | 195                   | 44.33    | 19.63           | 69.3                 | 78.8          |
| FRE-33 | 201                   | 36.69    | 16.67           | 84.7                 | 70.8          |
| FRE-34 | 151                   | 32.47    | 18.35           | 83.7                 | 60.9          |
| FRE-35 | 104                   | 34.32    | 14.43           | 78.8                 | 74.6          |
| FRE-36 | 51                    | 35.29    | 13.19           | 83.5                 | 76.7          |

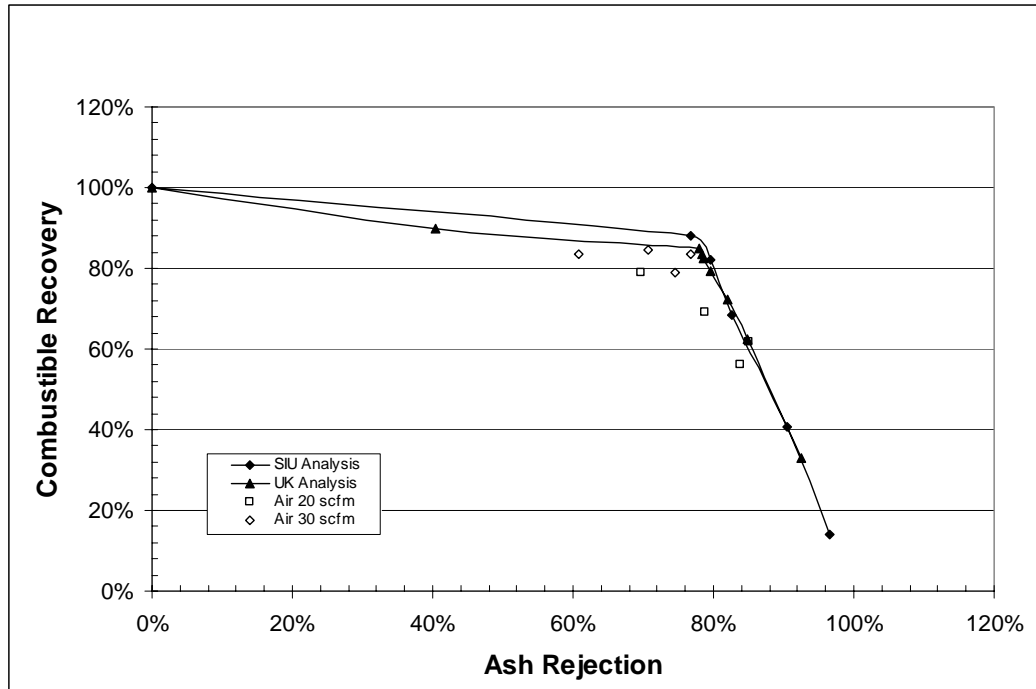


Figure 14. Concentrate Flow Optimization Data Plotted With Release Analysis Curves

To further investigate the effect of controlling the MRC based on the difference between feed and reject flow rates, an ANOVA analysis was performed using the concentrate flow rates from all previous tests, both optimization and Box-Behnken. Results of this analysis were perhaps the most significant finding of this research project. They found that the concentrate flow rate was indeed a significant factor in determining concentrate ash content, ash rejection rate and combustible recovery. Equations produced in the ANOVA results for predicting concentrate ash content, ash rejection rate and combustible recovery are as follows (Manrique et al., 2005):

$$\begin{aligned} \text{Concentrate Ash Content} = & + 0.24 \\ & - 0.039*A \\ & + 3.095E-003*B \\ & + 0.013*C \\ & + 0.093*D \\ & - 0.085*A*D \\ & - 0.032*B*C \\ & - 0.057*B*D \end{aligned}$$

$$\begin{aligned} \text{Ash Rejection Rate} = & + 0.61 \\ & + 0.013*B \\ & + 0.052*C \\ & - 0.18*D \\ & - 0.10*B*C \\ & - 0.10*C*D \end{aligned}$$

$$\begin{aligned}
 \text{Combustible Recovery} = & + 0.92 \\
 & + 0.16*A \\
 & - 0.12*B \\
 & - 0.13*C \\
 & - 0.085*D \\
 & - 0.21*A*B \\
 & - 0.061*A*C \\
 & - 0.19*A*D \\
 & + 0.15*B*C \\
 & + 0.20*B*D \\
 & + 0.13*C*D
 \end{aligned}$$

where A, B, C and D represent coded values for air flow, frother feed rate, collector feed rate and concentrate flow rate respectively.

#### **Task 6. Determination of the impact of the type of ISGS Washer fitted to the cell.**

Simultaneous to this project, a demonstration of the ISGS Inclined Froth Washer (IFW) was being conducted at The American Coal Company's Galatia Mine. Results from the Galatia Demonstration were used to design and construct an optimum washer configuration for this project essentially eliminating this task from the project.

#### **Task 7. QA/QC**

Samples from every test were sent to SGS Laboratories in Henderson, Kentucky for analysis using ASTM methods. Laboratory certifications of the results are on file at SGS. Duplicate samples were made from a majority of tests and these backup samples were sent to the ISGS for analysis, also using ASTM methods. The ISGS results served as a verification check on the SGS results. No discrepancies were noted.

#### **Task 8. Reporting and Technology Transfer**

During the latter months of this project, DSI began installing a 1,000-gallon MRC at their tailings pond recovery operation in Providence, Kentucky. That installation utilizes all of the data generated over the course of this project and they have implemented an automated level control system for the MRC. The Providence site provides the opportunity for continued development of the MRC at full-scale production rates.

With known operational parameters for the MRC from the Freeman Energy project and the Providence site, an economic feasibility analysis was developed. The analysis was based on three possible scenarios related to the demonstration at Freeman Energy. The first scenario considered just the current system as is. The second scenario considered adding all of the additional feed material potentially available from the Crown III plant. The final scenario assumed sufficient feed availability to operate the MRC at full capacity. The economic analysis incorporated a screen bowl centrifuge drying system to

dewater the MRC product into a salable commodity.

The results of this economic feasibility analysis are shown in Table 1 of the Executive Summary. This economic analysis makes it clear that the additional feed stream examined in the third scenario would be required for Freeman Energy to consider permanent installation and operation of an MRC at their Crown III Mine. Possibilities may include intermediate and coarse dryer refuse and the classifying cyclone overflow stream. Plans are being made to conduct a second demonstration that will add an additional feed stream to the MRC feed and incorporate a screenbowl centrifuge test unit to investigate how well the MRC product dries.

## CONCLUSIONS AND RECOMMENDATIONS

The goal of this project was to take the MRC from laboratory scale to field scale. Furthermore, the project intended to demonstrate the MRC in the field by installing a functional unit on site at a coal preparation plant where it could receive a continuous feed stream of fine material. This field demonstration combined with further laboratory work would be used to define the operating characteristics of the MRC and determine its ability to efficiently recover and clean fine coal waste. In so doing, the pathway to commercialization could be charted.

With significant support from Freeman Energy, the objectives of this project were accomplished when a fine coal cleaning circuit consisting of a 1,000-gallon MRC was installed adjacent to the Crown III preparation plant and tested on a fine coal slurry currently being discarded by the mine. Despite limited access to a less than desirable feed stream and the inherent inability to control the characteristics of that feed, by the end of the project the MRC proved itself capable of producing a clean coal concentrate at the most efficient point on the release analysis profile for the feed material.

The data generated during this project support the idea that a fine circuit utilizing MRC technology could produce a marketable product at the Crown III Mine with a payback period of less than five years. While more work is necessary to bring this technology to maturity, this project has shown that the MRC is a viable alternative to existing froth flotation technologies. Using the experience gained during this project, a commercial unit has been constructed and installed at a tailing pond recovery operation. This unit is producing a clean fine coal product of excellent quality at the rate of 6 to 7 tons per hour.

In addition to the successes achieved during this project, some additional avenues for investigation have been exposed. The idea of controlling the MRC based on maintaining a fixed fluid level in the cell proved to be erroneous. A preferred method of regulating the cell by maintaining a constant concentrate flow rate has been investigated, but further work is necessary as a simple fluid flow measuring device will not work with the concentrate froth. Regulation of flow rates based on direct measurement of froth height in the cell may also produce a workable system but requires investigation.

The extent of this project did not include drying the product. Obviously, determining the



drying characteristics of the MRC product will be essential to further forward movement on the pathway to commercialization.

The MRC tested in this project was equipped with an Inclined Froth Washer, also developed by the ISGS. The design was based on another field demonstration of that concept. However, that demonstration was conducted with subaeration flotation cells. These cells generally have little turbulence or motion of the froth at the collection point. The MRC is a pneumatic cell that utilizes some cyclonic action and moving fluid as a mixing mechanism. This motion translates to the froth at the collection point and when high fluid levels were used, it was observed that the IFW actually channeled fluid out of the cell suggesting the need to explore other methods of removing froth from the MRC.

Finally, it should be recognized that this is just the second phase of developing the MRC concept and the technology is still in its infancy. There are still many unknowns such as the hydrodynamics within the cell, what constitutes steady state for sampling purposes, the effect of bubble size, the method of air and chemical injection, just to name a few. With two operational units now in the field, the research group moving this technology towards commercialization has shown that it may be used in the field if one is willing to take some risk while learning to operate the system in an existing coal cleaning plant.

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## APPENDIX 1. Analysis Results for Samples Collected During Testing

Table A1.1. SGS Laboratories Analysis Results for Optimization Tests

| Test Sample (#) | Feed Ash (%) | Feed Sulfur (%) | Feed Btu | Conc. Ash (%) | Conc. Sulfur (%) | Conc. Btu | Reject Ash (%) | Reject Sulfur (%) | Reject Btu |
|-----------------|--------------|-----------------|----------|---------------|------------------|-----------|----------------|-------------------|------------|
| FRE-1           | 49.83        | 9.43            | 5,932    | 13.08         | 6.16             | 12,341    | 72.14          | 8.57              | 1,351      |
| FRE-3           | 41.21        | 7.54            | 7,373    | 15.42         | 6.38             | 11,821    | 68.78          | 7.5               | 2,218      |
| FRE-4           | 32.71        | 6.05            | 9,045    | 12.44         | 5.61             | 12,305    | 60.76          | 7.55              | 3,864      |
| FRE-5           | 38.74        | 8.43            | 7,609    | 16.33         | 15.41            | 11,508    | 52.07          | 11.2              | 5,848      |
| FRE-6           | 35.53        | 6.94            | 8,382    | 19.22         | 6.89             | 11,466    | 47.25          | 8.32              | 6,009      |
| FRE-7           | 33.23        | 6.90            | 8,782    | 17.07         | 5.85             | 11,660    | 65.93          | 8.57              | 2,590      |
| FRE-8           | 42.43        | 9.06            | 6,674    | 19.22         | 7.85             | 11,251    | 52.07          | 11.2              | 4,803      |
| FRE-9           | 38.42        | 7.39            | 7,766    | 20.86         | 6.45             | 11,075    | 60.16          | 8.43              | 3,165      |
| FRE-10          | 44.50        | 6.91            | 7,064    | 21.15         | 6.83             | 10,846    | 61.86          | 8.20              | 4,133      |
| FRE-11          | 38.58        | 6.36            | 7,989    | 21.83         | 5.99             | 10,902    | 70.86          | 6.49              | 2,624      |
| FRE-12          | 42.72        | 6.78            | 7,289    | 24.72         | 8.41             | 10,205    | 77.05          | 6.24              | 1,239      |
| FRE-13          | 47.09        | 7.99            | 6,293    | 22.56         | 7.66             | 10,327    | 72.70          | 9.67              | 1,691      |
| FRE-14          | 42.43        | 6.55            | 7,398    | 20.96         | 6.75             | 11,112    | 77.26          | 7.03              | 1,209      |
| FRE-15          | 50.83        | 9.37            | 5,423    | 29.13         | 6.04             | 9,597     | 75.86          | 8.48              | 834        |
| FRE-16          | 39.22        | 6.86            | 8,059    | 23.10         | 6.63             | 10,555    | 73.31          | 8.24              | 1,625      |
| FRE-17          | 33.47        | 5.75            | 8,805    | 23.94         | 5.75             | 10,555    | 75.18          | 5.67              | 1,490      |
| FRE-18          | 38.03        | 6.47            | 7,977    | 21.89         | 6.71             | 10,809    | 76.35          | 5.12              | 1,161      |
| FRE-19          | 47.76        | 8.92            | 6,416    | 31.26         | 8.69             | 9,205     | 73.94          | 5.01              | 1,221      |
| FRE-20          | 42.34        | 7.02            | 7,301    | 35.19         | 5.83             | 8,532     | 42.16          | 7.16              | 7,335      |
| FRE-21          | 40.00        | 6.93            | 7,616    | 28.18         | 7.05             | 9,682     | 68.12          | 6.91              | 2,257      |
| FRE-22          | 36.88        | 5.67            | 8,132    | 24.72         | 6.08             | 10,084    | 69.35          | 5.23              | 2,427      |
| FRE-23          | 42.09        | 6.67            | 7,083    | 31.14         | 6.90             | 8,914     | 75.15          | 5.85              | 979        |
| FRE-24          | 45.29        | 4.25            | 6,870    | 30.79         | 4.58             | 9,314     | 64.92          | 4.67              | 3,541      |
| FRE-25          | 44.58        | 4.14            | 7,120    | 35.44         | 4.18             | 8,826     | 71.35          | 4.36              | 2,149      |
| FRE-26          | 46.12        | 4.20            | 7,129    | 35.47         | 4.18             | 8,599     | 72.10          | 4.44              | 2,384      |
| FRE-29          | 37.66        | 6.65            | 7,915    | 12.67         | 5.62             | 12,307    | 57.41          | 8.19              | 4,176      |
| FRE-30          | 38.11        | 6.85            | 7,709    | 14.97         | 6.15             | 11,903    | 54.16          | 7.51              | 4,867      |
| FRE-31          | 44.33        | 8.18            | 6,649    | 19.63         | 7.21             | 11,132    | 67.11          | 9.92              | 2,240      |
| FRE-32          | 38.42        | 7.45            | 7,625    | 19.33         | 7.12             | 11,124    | 67.39          | 9.19              | 2,269      |
| FRE-33          | 36.69        | 6.72            | 8,241    | 16.67         | 6.68             | 11,622    | 72.87          | 7.53              | 1,310      |
| FRE-34          | 32.47        | 6.59            | 8,776    | 18.35         | 6.61             | 11,368    | 64.20          | 7.45              | 2,836      |
| FRE-35          | 34.32        | 6.41            | 8,535    | 14.43         | 6.09             | 12,079    | 64.76          | 6.54              | 2,988      |
| FRE-36          | 35.29        | 7.17            | 8,042    | 13.19         | 6.09             | 12,188    | 71.6           | 7.16              | 1,467      |
| FRE250DF        | 45.88        | 5.12            | 6,914    | 21.13         | 5.27             | 10,965    | 75.99          | 5.54              | 2,093      |

Table A1.2. SGS Laboratories Analysis Results for Box-Behnken Tests

| Test Sample | Feed Ash | Feed Sulfur | Feed Btu | Conc. Ash | Conc. Sulfur | Conc. Btu | Reject Ash | Reject Sulfur | Reject Btu |
|-------------|----------|-------------|----------|-----------|--------------|-----------|------------|---------------|------------|
| (#)         | (%)      | (%)         |          | (%)       | (%)          |           | (%)        | (%)           |            |
| BB1         | 39.33    | 6.45        | 7,614    | 25.55     | 7.09         | 10,098    | 75.69      | 6.63          | 1,246      |
| BB2         | 38.46    | 7.98        | 7,985    | 37.51     | 7.02         | 7,940     | 76.31      | 7.23          | 1,173      |
| BB3         | 39.34    | 8.36        | 7,625    | 26.68     | 8.18         | 10,120    | 69.92      | 8.63          | 2,024      |
| BB4         | 43.87    | 7.76        | 7,538    | 19.08     | 7.19         | 11,306    | 67.18      | 10.18         | 2,521      |
| BB5         | 43.74    | 7.81        | 6,649    | 27.14     | 7.49         | 9,991     | 74.55      | 8.21          | 1,510      |
| BB6         | 48.45    | 9.10        | 5,694    | 28.19     | 6.58         | 9,621     | 74.01      | 8.41          | 1,307      |
| BB7         | 44.70    | 8.23        | 6,407    | 24.20     | 6.31         | 10,434    | 69.23      | 7.90          | 2,284      |
| BB8         | 43.40    | 8.36        | 6,844    | 24.48     | 7.06         | 10,349    | 73.87      | 8.44          | 1,400      |
| BB9         | 40.83    | 7.47        | 7,515    | 24.99     | 6.60         | 10,464    | 73.71      | 7.49          | 1,669      |
| BB10        | 28.62    | 6.59        | 8,081    | 24.75     | 6.48         | 10,230    | 75.68      | 6.29          | 1,688      |
| BB11        | 44.30    | 8.60        | 6,978    | 24.83     | 7.75         | 10,244    | 75.64      | 8.17          | 733        |
| BB12        | 39.72    | 6.99        | 7,586    | 24.34     | 6.97         | 10,442    | 74.64      | 8.32          | 1,484      |
| BB13        | 41.08    | 6.96        | 7,386    | 21.44     | 7.56         | 10,858    | 74.82      | 7.51          | 1,414      |
| BB14        | 38.90    | 6.95        | 7,772    | 25.40     | 6.88         | 10,136    | 75.16      | 7.67          | 1,412      |
| BB15        | 40.51    | 6.00        | 7,747    | 15.15     | 5.83         | 11,320    | 71.28      | 5.95          | 2,475      |
| BB16        | 41.50    | 7.56        | 7,537    | 23.74     | 6.95         | 10,619    | 74.44      | 8.37          | 1,460      |
| BB17        | 43.33    | 7.26        | 7,205    | 21.87     | 6.81         | 10,847    | 70.51      | 7.70          | 2,164      |
| BB18        | 39.94    | 6.53        | 7,747    | 17.78     | 6.18         | 11,677    | 71.62      | 6.99          | 2,282      |
| BB19        | 41.47    | 7.08        | 7,523    | 22.64     | 7.46         | 10,755    | 75.57      | 6.81          | 1,293      |
| BB20        | 43.63    | 7.94        | 7,094    | 28.09     | 7.96         | 9,602     | 72.00      | 8.71          | 1,732      |
| BB21        | 41.81    | 7.21        | 7,279    | 29.10     | 8.30         | 9,593     | 76.34      | 8.07          | 1,077      |
| BB22        | 43.59    | 7.02        | 7,188    | 26.89     | 7.07         | 9,929     | 76.29      | 6.74          | 1,384      |
| BB23        | 46.68    | 7.58        | 6,544    | 29.12     | 6.69         | 9,654     | 79.15      | 6.24          | 908        |
| BB24        | 44.86    | 6.46        | 6,810    | 34.49     | 7.09         | 8,718     | 77.00      | 6.18          | 1,141      |
| BB25        | 41.59    | 5.35        | 7,495    | 28.35     | 5.96         | 9,838     | 77.99      | 5.12          | 1,505      |
| BB26        | 45.69    | 6.32        | 6,730    | 32.79     | 6.17         | 8,934     | 74.64      | 7.51          | 1,334      |
| BB27        | 47.21    | 5.98        | 6,789    | 34.68     | 7.18         | 8,777     | 74.76      | 6.30          | 1,150      |
| BB28        | 47.19    | 5.84        | 6,870    | 28.97     | 5.83         | 9,577     | 72.66      | 4.46          | 2,454      |
| BB29        | 47.22    | 5.47        | 6,779    | 28.92     | 5.70         | 9,766     | 74.54      | 5.83          | 1,948      |
| BB30        | 47.94    | 3.90        | 6,667    | 30.22     | 6.15         | 9,273     | 75.06      | 4.35          | 1,998      |

Table A1.3. ISGS Laboratory Analysis Results for Optimization Tests

| Test Sample | Feed Ash | Feed Sulfur | Conc. Ash | Conc. Sulfur | Reject Ash | Reject Sulfur |
|-------------|----------|-------------|-----------|--------------|------------|---------------|
| (#)         | (%)      | (%)         | (%)       | (%)          | (%)        | (%)           |
| FRE-9       | 46.25    | 7.59        | 21.63     | 6.98         | 66.94      | 8.79          |
| FRE-10      | 42.53    | 7.41        | 22.94     | 6.90         | 70.39      | 8.34          |
| FRE-11      | 41.39    | 6.55        | 23.95     | 5.97         | 73.65      | 7.45          |
| FRE-12      | 45.76    | 6.86        | 26.55     | 9.49         | 76.44      | 4.60          |
| FRE-13      | 50.75    | 8.01        | 24.71     | 7.39         | 78.89      | 6.66          |
| FRE-14      | 48.79    | 7.48        | 22.09     | 6.75         | 80.51      | 7.02          |
| FRE-15      | 54.08    | 8.43        | 32.31     | 6.51         | 79.76      | 4.34          |
| FRE-16      | 40.04    | 6.03        | 24.74     | 6.62         | 77.69      | 7.76          |
| FRE-17      | 36.87    | 6.00        | 25.00     | 5.63         | 77.34      | 6.83          |
| FRE-18      | 42.43    | 6.93        | 23.10     | 6.39         | 79.49      | 5.55          |
| FRE-19      | 48.95    | 7.00        | 33.15     | 9.27         | 76.67      | 7.55          |
| FRE-20      | 44.73    | 7.17        | 39.82     | 5.49         | 55.14      | 7.81          |

Table A1.4. ISGS Laboratory Analysis Results for Box-Behnken Tests

| Test Sample<br>(#) | Feed Ash<br>(%) | Feed Sulfur<br>(%) | Conc. Ash<br>(%) | Conc. Sulfur<br>(%) | Reject Ash<br>(%) | Reject Sulfur<br>(%) |
|--------------------|-----------------|--------------------|------------------|---------------------|-------------------|----------------------|
| BB1                | 46.75           | 7.40               | 26.26            | 7.66                | 79.22             | 8.67                 |
| BB2                | 32.00           | 8.28               | 40.19            | 7.26                | 78.96             | 7.13                 |
| BB3                | 53.68           | 8.24               | 29.59            | 7.77                | 78.84             | 8.98                 |
| BB4                | 45.68           | 7.44               | 18.71            | 6.45                | 74.25             | 10.30                |
| BB5                | 48.71           | 7.67               | 29.18            | 6.51                | 77.22             | 9.42                 |
| BB6                | 53.06           | 9.87               | 31.60            | 7.10                | 77.24             | 10.65                |
| BB7                | 51.29           | 8.43               | 26.45            | 6.28                | 72.99             | 7.39                 |
| BB8                | 48.36           | 7.88               | 26.54            | 7.61                | 78.42             | 8.96                 |
| BB9                | 46.76           | 7.77               | 25.90            | 7.40                | 78.89             | 8.94                 |
| BB10               | 43.37           | 6.76               | 26.39            | 6.15                | 78.83             | 6.59                 |
| BB11               | 51.33           | 8.78               | 27.82            | 8.49                | 76.67             | 9.00                 |
| BB12               | 45.49           | 7.10               | 26.82            | 7.15                | 76.69             | 8.10                 |
| BB13               | 45.99           | 7.22               | 23.68            | 7.15                | 77.41             | 8.01                 |
| BB14               | 44.61           | 6.42               | 27.74            | 7.10                | 78.25             | 7.46                 |
| BB15               | 43.48           | 6.28               | 20.61            | 5.06                | 75.88             | 7.24                 |
| BB16               | 45.89           | 7.42               | 26.21            | 7.13                | 78.06             | 8.60                 |
| BB17               | 46.87           | 7.23               | 22.77            | 6.87                | 76.85             | 8.08                 |
| BB18               | 41.35           | 6.27               | 18.47            | 5.68                | 75.05             | 7.52                 |
| BB19               | 48.99           | 7.38               | 24.58            | 6.99                | 78.38             | 7.40                 |
| BB20               | 51.10           | 8.01               | 30.70            | 7.91                | 79.43             | 8.74                 |
| BB21               |                 |                    | 31.25            | 8.27                |                   |                      |
| BB22               |                 |                    | 29.19            | 7.23                |                   |                      |
| BB23               |                 |                    | 32.59            | 6.51                |                   |                      |
| BB24               |                 |                    | 37.22            | 7.77                |                   |                      |
| BB25               |                 |                    | 31.42            | 5.83                |                   |                      |
| BB26               |                 |                    | 37.07            | 6.25                |                   |                      |
| BB27               |                 |                    | 37.15            | 8.03                |                   |                      |
| BB28               |                 |                    | 32.68            | 6.11                |                   |                      |
| BB29               |                 |                    | 30.59            | 5.92                |                   |                      |
| BB30               |                 |                    | 35.27            | 6.53                |                   |                      |

EQUIPMENT INVENTORY  
September 1, 2004, through November 30, 2005

Project Title: **PATHWAY TO COMMERCIALIZATION OF THE MOTORLESS ROTORLESS CELL**

ICCI Project Number: 04-1/2.1A-2  
Principal Investigator: Latif A. Khan, ISGS  
Other Investigators: William Roy, ISGS  
ICCI Project Manager: Joseph Hirschi

List of Equipment Purchased

| Account     | Date<br>Acquired | Description                 | Cost  | Bldg      |
|-------------|------------------|-----------------------------|-------|-----------|
| 04-1/2 1A-2 | 7/28/2005        | 300 Gallon Double Wall Tank | 1,388 | DSI       |
| 04-1/2.1A-2 | 7/28/2005        | MRC Tank Stand (Material)   | 5,550 | Crown III |
| 04-1/2 1A-2 | 11/28/2005       | Reagent Pump                | 928   | Crown III |
| 04-1/2 1A-2 | 11/28/2005       | Reagent Pump                | 1,997 | Crown III |

PUBLICATIONS AND PRESENTATIONS REPORT  
September 1, 2004, through November 30, 2005

Project Title: **PATHWAY TO COMMERCIALIZATION OF THE MOTORLESS  
ROTORLESS CELL**

ICCI Project Number: 04-1/2.1A-2  
Principal Investigator: Latif A. Khan, ISGS  
Other Investigators: William Roy, ISGS  
ICCI Project Manager: Joseph Hirschi

List of Publications and Presentations

None