TECHNICAL REPORT
March 1, 1996, through May 31, 1996

Project Title: INNOVATIVE PROCESS FOR CONCENTRATION OF FINE PARTICLE COAL SLURRIES

DOE Cooperative Agreement Number: DE-FC22-92PC92521 (Year 4)
ICCI Project Number: 95-1/5.2A-3P
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ABSTRACT

Williams Technologies, Inc. And Clarke Rajchel Engineering are developing a technology (patent pending) to produce high quality coal water slurries from preparation plant fine coal streams. The WTI/CRE technology uses the novel implementation of high-shear cross-flow separation which replaces and enhances conventional thickening processes by surpassing normally achievable solids loadings. Dilute ultra-fine (minus 100 mesh) solids slurries can be concentrated to greater than 60 weight percent and re-mixed, as required, with de-watered coarser fractions to produce pumpable, heavily loaded coal slurries. The permeate (filtrate) resulting from this process has been demonstrated to be crystal clear and totally free of suspended solids.

The primary objective of this project was to demonstrate the WTI/CRE coal slurry production process technology at the pilot scale. The technology can enable Illinois coal producers and users to realize significant cost and environmental benefits both by eliminating fine coal waste disposal problems and producing an IGCC fuel to produce power which meets all foreseeable clean air standards. Testing was also directed at concentrating mine tailings material to produce a tailings paste which can be mine-back-filled, eliminating the need for tailings ponds.

During the grant period, a laboratory-scale test apparatus (up to 3 GPM feed rate) was assembled and operated to demonstrate process performance over a range of feed temperatures and pressures. A dilute coal/water slurry from Consol, Inc.’s Rend Lake Preparation Plant was concentrated using the process to a maximum recorded solids loading of 61.9% solids by weight. Analytical results from the concentrate were evaluated by Destec Energy for suitability as an IGCC fuel.

“U.S. DOE Patent Clearance is NOT required prior to the publication of this document.”
EXECUTIVE SUMMARY

It has been estimated that the Illinois coal industry discards over 4,000,000 tons of coal fines into tailings impoundments each year. Much of this coal is of fairly high quality due to a high degree of inherent natural mineral liberation. As such, this coal might be considered "pre-prepared" for a number of various fine coal beneficiation processes. Also, it does not require milling for feed to such processes as IGCC or co-firing with pulverized coal in utility boilers. Coal fines most often go under-utilized because of inadequate means of de-watering and because of associated filter-cake handling problems. Williams Technologies, Inc. and Clarke Rajchel Engineering are developing a technology (patent pending) to produce high quality coal water slurries from preparation plant fine coal streams.

In this engineering study, recovered fine coal from Consol Inc.'s Rend Lake Preparation Plant was concentrated with the WTI/CRE process. The highest recorded slurry concentration was 61.9 weight percent solids slurry. Flotation concentrate feed slurry from the Rend Lake fine coal cleaning circuit which had been cleaned (via flotation) to less than 6.5% ash and 1.3% sulfur and with a nominal concentration of 15 weight percent, and of a size suitable for feed to Destec's slurry fed coal gasification plant was utilized in this demonstration.

The primary objective of this project was to demonstrate the WTI/CRE coal slurry production process technology at a pilot-plant scale. It is hoped that the technology will enable Illinois coal producers and users to realize significant cost and environmental benefits both by eliminating fine coal waste disposal problems while producing an IGCC fuel which will meet all foreseeable clean air standards. In addition, testing was also directed at concentrating mine tailings material to produce a tailings paste which can be mine-back-filled and thus eliminate the need for tailings ponds.

Central to the WTI/CRE process technology is the novel implementation of high-shear cross-flow separation which replaces and enhances the thickening process by surpassing normally achievable solids loadings. Dilute ultra-fine (minus 100 mesh) solids slurries can be concentrated and re-mixed, as required, with de-watered coarser fractions to produce pumpable, heavily loaded coal slurries. The permeate (filtrate) resulting from this process has been demonstrated to be crystal clear and totally free of suspended solids.

The project team was assembled to present a "total technology approach" to provide a package which addresses the interests of the Illinois power generation industry. The project team includes the proposers, Williams Technologies, Inc./Clarke Rajchel Engineering Joint Venture; Consol, Inc. coal company of Illinois; Destec Energy, Inc., and Southern Illinois University. An example of the target interest is represented by the planned Franklin County Industrial Park which has shown interest in providing an environmentally friendly supply of power to their users while maintaining the support of the Illinois Coal industry.
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The specific goals of the effort are:

- To improve the overall plant efficiency of Illinois coal preparation facilities and thus reduce overall costs by reducing wastes and lowering plant capital and operating costs.

- To provide the slurry production technology to produce a fuel suitable for the Destec slurry fed gasification process using recovered preparation plant fines.

- To demonstrate a process which will reduce or eliminate the need for large tailings impoundments.

- To gather data which will provide the engineering information required to design a demonstration-scale process facility for the production of coal-water slurry fuels from preparation plant fines which is suitable for the Destec slurry fed IGCC process.

- To produce a cleaner fuel by taking advantage of the natural mineral liberation associated with preparation plant coal fines.

- To demonstrate the utility of the technology in producing a tailings paste which is suitable for mine back-filling.

The specific tasks involved in achieving these goals included:

A. **Process Set-up**
   
   A model process plant was assembled and operated at the SIUC Coal Research Center, Carterville, Illinois. The test setup included a membrane separator, feed tanks and piping, and a steam generator.

B. **Ambient Temperature Tests**
   
   This testing had the goals both equipment shake-down and base-line testing. Slurries were concentrated and performance data measured.

C. **Elevated Temperature Tests**
   
   Elevation of feed slurry temperature has an effect linked to reduced slurry carrier fluid viscosity. Tests were performed to demonstrate the substantial improvement in unit water removal rates.

D. **Optimum Vibrational Frequency Tests and Feed Pulse**
   
   Optimum shearing frequency was investigated for the pilot scale separator. Also, the effect of disturbing the fines bed associated with the membrane surface was investigated.

E. **Process Control Strategy Tests**
   
   Optimal operations in a commercial plant will most likely include some level of instrumentation which will aid in start-up, shut-down, normal run-mode
operation, and product quality control. Process control strategies were developed to provide smooth start-up and reliable operation.

F. **Equilibrium Flux Rate at Elevated Slurry Concentration**
(Formerly “Long Duration Testing”)
Rend Lake Flotation Concentrate feed slurry was concentrated at both ambient and elevated temperatures. Measurements at system equilibrium were taken at various values of feed slurry solids concentration to observe the variation of flux rate versus solids loading.

This task was modified from the original proposed scope during the project. The original objective was to perform a set of long-duration tests to demonstrate equilibrium performance and investigate membrane degradation over a period of 100 hours or more. However, long duration tests proved to be impractical owing to equipment limitations of the test apparatus.

G. **Tailings Paste Production**
Tailings material was concentrated to determine whether a paste suitable for mine back-fill can be produced or if the material can be concentrated such that a better feed for belt filtration and subsequent tailings pile stacking can be achieved.

A dilute tailings slurry from the Rend Lake Preparation Plant was concentrated from 1% to 3.5% solids. Further concentration was not attempted due to insufficient quantities of feed slurry. In addition, the tailings slurry tested was apparently not a representative sample of the Rend Lake waste product.

H. **Destec Slurry Fuel Evaluation**
Samples of coal/water slurry fuel made from the Rend Lake Flotation Concentrate were prepared and analyzed. Analytical results were evaluated by Destec Energy, Inc. to determine gasifier acceptability and estimated IGCC heat rate for the fuels. Destec’s analysis indicated that the best fuel sample, which was 61.9 wt% solids, would make an acceptable gasified fuel with an estimated IGCC heat rate of 8,000 Btu/KWH.
OBJECTIVES

The overall objective of this program was to demonstrate the fine particle slurry de-watering process being developed by the Williams Technologies, Inc.-Clarke Rajchel Engineering Joint Venture (WTI/CRE) through construction and operation of a nominal 5 gallon per minute pilot plant. The specific objectives of this research included:

1. Demonstration of an inexpensive high shear cross-flow membrane separation process for cost effective production of multiple use coal-water slurry (CWS) fuels/IGCC feeds.

2. Generate engineering data required to design a fully automated Coal-Water Slurry Fuel production plant for a commercial-scale demonstration facility. Testing was focused on a cleaned-coal slurry produced at Consol’s Rend Lake Preparation Plant.

3. Prepare fuels for analysis by an IGCC manufacturer (Destec Energy) and determine both the suitability of the fuel for gasification feed and computation of unit power production costs.

4. Investigate process performance and determine economics for dewatering a dilute mine tailings slurry.

5. Generate preliminary process economic data for comparison with other fuel production and dewatering technologies.

INTRODUCTION AND BACKGROUND

The technological approach used to remove water from fine coal slurries in this program utilizes cross-flow membrane separation, heat, and feed pulsation to effect a separation of water from the fine particle slurries. The separation of water from CWS is effected by pumping the slurry across a fine pore membrane surface. The membrane is vibrated radially at high frequency (50-60 Hz) which prevents the pores from being blinded by the finest slurry particles. The product is a thickened slurry. In previous laboratory work, fine particle slurries approaching 60 weight percent coal have been produced. Major advantages of the technology include:

a) Eliminates flocculants required by both filtration and thickening processes.

b) Achieves higher solids concentrations than achievable in conventional thickeners.

c) Slurry concentration equipment is compact in comparison to other fine particle dewatering/thickening equipment. The footprint of the commercial machinery is approximately eight feet by eight feet, including pumps.
d) Equipment is simple requiring maintenance similar to that of a pump.

e) Provides a pumpable fuel for coal gasification and combustion technologies.

f) High potential for use as a NOx reducing re-burn fuel

The approach might also find utility in the de-watering of tailings muids from coal wash plants. Thickening tailings to a paste consistency will allow the waste material to be disposed of in a much smaller area.

**EXPERIMENTAL PROCEDURES**

Previous work (DoE Contract No. DE-FG03-93ER-81503) with the WTI/CRE technology established that it was both technically and economically interesting. The previous work included separation of water from CWS with a laboratory-scale separator which uses a single membrane surface with approximately one-half square foot of membrane separation area. In this work various recovered fine-coal feedstocks were concentrated to solids loadings up to 58.9 weight percent solids.

This program attempted to increase the process throughput to pilot scale with a feed flow rate up to five gallons-per-minute, or about 1.35 tph (slurry basis). The cross-flow separator in the pilot plant utilizes multiple membrane plates stacked vertically to increase the filtration area. The configuration was similar to (but not identical) that of the commercial-scale equipment. The total pilot cross-flow separator membrane area could be configured with up to 15 square feet of separation area.

In previous work, water removal performance has been shown to be enhanced by the addition of heat to the feed. The configuration of the pilot plant is depicted in Figure 1. A package boiler provided heat to the feed by direct steam injection. To reduce the process requirements, heat was recovered by exchange from both product slurry and separated water (permeate) with the feed in double-pipe heat exchangers.

The pilot plant program was divided into separate tasks described below:

**Task A Procurement and Set-Up**

The pilot plant apparatus was constructed at the SIU Coal Research Center in Carterville, Illinois. All equipment, supplies, and slurry feedstocks were delivered to this site and connected by SIUC and WTI/CRE personnel. Major equipment included one New Logic International, Inc. V-SEP "Series P" high-shear membrane separator and a 6.93-horsepower Chromalox process steam generator. Samples of both froth flotation concentrate and fine coal wastes were obtained from Consol’s Rend Lake Preparation Plant. The plant was commissioned and tested first with water and then with slurry.
Task B  **Ambient Temperature Tests**

This testing was performed to accomplish both equipment shake-down and base-line testing. The first work included various concentration tests at ambient temperatures, and was to duplicate earlier, laboratory scale work at the pilot scale.

Task C  **Elevated Temperature Tests**

Rend Lake Flotation Concentrate (RLFC) was concentrated to maximum solids loading at a temperature of 120 °F. The manufacturer has indicated that the standard V-SEP machine can be reliably operated at temperatures up to 300 °F and 200 psig. Because of some of the materials of construction in the pilot scale unit, temperatures were kept below 200 °F. Flux rate (water removal rate) was measured versus slurry feed temperature at temperatures ranging from ambient to 200 °F.

Task D  **Optimum Vibrational Frequency Tests and Feed Pulse**

Previous test work by the principal investigator suggested that improvement in water removal rates of between 10% and 25% can be achieved if the feed pressure is periodically pulsed between the normal operating pressure and some lower pressure. It has been postulated that the disturbance in the traveling fines bed which forms along the surface of the membrane decreases the cake resistance of the fines bed until the bed re-establishes itself. Also during Tasks B, C, and F, vibrational frequency was varied to produce a range of radial deflection from 0.75 inch to 1.25 inch.
Task E  Process Control Strategy Tests

The effect of start-up and shut-down procedures as well as the anticipated instrumentation required to produce a product of consistent quality were observed. Run-time procedures to detect and prevent plugging and membrane damage were developed.

Task F  Equilibrium Flux Rate vs. Concentration Tests
(formerly “Long Duration Testing”)

The scope of Task F changed during performance of the test program. The original scope included two one-week continuous runs to demonstrate the durability of membrane separator under the seemingly harsh conditions of vibration found in the V-SEP. The focus of the new Task F work plan included runs to measure equilibrium flux rate at varying solids concentration followed by operation of the pilot equipment at elevated concentrations. This change was made in light of:

a. Industrial scale V-SEP units are currently operating at commercial facilities, indicating that the equipment will indeed withstand intense vibrational forces.

b. The initial work plan did not include work which quantified the effect of increasing solids concentration upon unit flux rates.

The new Task F consisted of concentrating one barrel of Consol Rend Lake Flotation Concentrate in steps until a maximum solids loading is achieved. At selected intervals, the apparatus was placed in the full-recycle mode (i.e., concentration of the feed was stopped) and equilibrium measurements of flux rate were taken. Tests were performed at both ambient and elevated temperature. In these tests, operation and relative performance at equilibrium of the multiple-tray system at elevated concentrations was observed. These tests were to demonstrate or give insight on the following:

a. Whether a V-SEP “membrane stack” can function reliably with fine coal flotation concentrate under conditions of high solids loading. Previous experience with a single membrane laboratory-scale separator has shown that a thick paste can be produced, but scale-up to a stack of membranes increases the complexity of operation by increasing the slurry water removal per pass through the machine.


c. Determine through inspection the problems or limitations which are inherent to the New Logic Series-P separator. In particular, does the membrane stack become partially plugged with de-watered paste in areas of low-cross flow?
Task G  **Tailings Paste Production**

A Rend Lake tailings slurry was concentrated to determine whether a paste suitable for mine back-fill can be produced. It was also sought to determine if the tailings material could be concentrated to provide an improved feed for belt filtration and subsequent stacking could be achieved.

Task H  **Destec Slurry Fuel Evaluation**

Fuel samples were prepared in consultation with Destec Energy, Inc. Destec performed a heat rate and gasifier acceptability analysis of two samples taken from test runs in Tasks C and F. Physical data from each sample was used as input for Destec's software gasification model.

**RESULTS AND DISCUSSION**

Task A  **Procurement and Pilot Plant Construction**

Task B  **Ambient Temperature Tests**

Task C  **Elevated Temperature Tests**

Task D  **Optimum Vibrational Frequency and Feed Pulse Tests**

Tasks A through D were completed during the First and Second Quarters.

Task E  **Process Control Strategy Tests**

The following control strategies were developed based on operating experiences with the L-Series and P-Series units. These strategies would be recommended for process automation design in a commercial plant.

a. **Preventing Membrane Failure On Low Differential Pressure**

One way to destroy a membrane(s) is to vibrate the membrane stack with insufficient trans-membrane pressure differential. This happens when either

- the permeate line is closed (which equalizes pressure on both sides of the membrane, causing it to separate from its support with or without vibration)
- when the slurry flow rate is very low or stopped (which can also cause membrane separation during vibration), or
- any time the differential pressure between the slurry-side of the membrane and the water side of the membrane is less than about 25 psi.
The simple solution to this problem is to install a differential pressure switch (or transmitter) between the product slurry outlet and the permeate outlet ports and interlock the signal to the vibration motor and feed slurry pump contactors. When the vibration and slurry flow are stopped, the risk of damage to the membranes vanishes.

b. Preventing Membrane Failure with No Vibration Present

Another way to damage the membranes in the P-Series Unit is to run high cross-flow rates while the vibration is off and permeate flow is blocked off. We found that the turbulence of the cross-flow alone will tend to separate the membrane from the support even when no vibration is present. Simply opening the permeate valve to create a trans-membrane pressure differential did not suffice since this tended to cause plugging. This is not a problem with other, more typical V-SEP applications with very fine slurries, but because of the coarse particle size, coal slurries tend to rapidly de-water and plug under conditions of no vibration.

Cross-flow-with-no-vibration is a start-up situation; that is, one should not start vibration without flow (see “a” above). The solution involves interlocking the pump with the vibration drive contactor such that both must be on. A short delay timer in the process programmable logic controller (PLC) which starts the vibration within a few seconds of the pump start is the best means of control.

c. Preventing Plugging

During operation, there could be events which cause the onset of membrane stack plugging (e.g., upsets in flows, pressures, etc.). If caught early, there are means to “un-plug” the membrane stack reliably and return the unit to original performance. The means of detecting plugging include:

1. Decrease In Vibration Amplitude/Increase In Motor Load
   A decrease in vibration amplitude during steady-state generally means that the stack is getting heavier, which generally means that the stack is beginning to plug. The stack does not plug instantaneously, but it can in a matter of minutes. When this happens, the motor load also increases as a result of the increased stack mass.

   If permeate withdrawal is continued, the stack further plugs and the V-SEP becomes a very small (and rather expensive) filter press. The reliable way to clear the machine is to:

   1. Choke the permeate flow to a very small value while still maintaining the a minimum 20 psid trans-membrane pressure.
   2. Reduce the membrane stack vibration to about 1/2 the original radial deflection.
   3. Take note of the motor load at the reduced vibration amplitude.
4. As solids are cleared from the stack and are replaced by slurry, the motor load (at the reduced amplitude) will decrease.

5. The indication that the plugging has been eliminated is when the motor load has fallen to a new steady state value. When the motor load is at a minimum, normal operation can be resumed.

6. Final assurance that the machine has become unplugged can be determined when the original motor load and radial deflection are achieved after resuming normal operation.

Detection of incipient plugging can be achieved through PLC monitoring of the motor load and/or optical deflection indication. An increase in motor load or decrease in radial deflection could both trigger an alarm and initiate an anti-plugging sequence. The sequence would reduce the permeate flow by positioning the permeate flow valve while maintaining an adequate trans-membrane pressure. Vibration could be reduced by reducing the vibration drive motor speed either automatically or manually.

Decrease in Permeate Flow Rate
A more sophisticated (but more difficult) means of detecting plugging would be to monitor changes in flux rate. Minor fluctuations in permeate flow rate are normal and to be expected; this is what makes this method more problematic. One would have to choose a "threshold" value of permeate flow rate decrease to initiate an automatic membrane stack unplugging sequence. For example, one may encounter normal fluctuations of 10% above or below the normal performance value. In this case, one may choose to have the PLC initiate the un-plugging sequence when the permeate flow rate drops by, for example, 25%.

One advantage of permeate flow monitoring is that various operator alarms could be initiated to signal potential problems to the operator very early. Such alarms would not represent any significant extra costs since permeate flow meters would be included in the installation in any case.

d. Start-Up Sequencing
During operation of the pilot plant, WTI/CRE found that there are numerous operations that must be simultaneously started and monitored during equipment start-up. They include:

1. Feed slurry pump start.

2. Shortly after pump start, vibration must be started.
Control Action: Include a vibration start-up interlock timer to start vibration after feed slurry flow is established.

3. Permeate removal must be started (permeate valve opened). Initially, however, the permeate flow should be controlled since high initial flux
rates can cause plugging on start-up; this is a problem specific to de-watering coal with V-SEP since the particle size is relatively large when compared to other V-SEP applications. Initial flux rates of three to four times the steady-state value are commonly encountered; that is, the coal can be completely dewatered to a filter cake within the machine with an uncontrolled start-up. Once steady-state operation has been achieved, the valve can be fully opened. Steady-state operation is achieved after the machine is full of slurry and a steady-state sludge bed has formed. This can take from several minutes to an hour depending upon the application and the number of membranes in the stack.

Control Action: Limit the permeate flow on start-up to less-than-or-equal-to the expected steady-state permeate flow rate using a flow controller on a permeate flow control valve.

4. During start-up, the first liquid out of the separator is normally water. To maintain the required trans-membrane pressure, the product-slurry outlet flow/pressure control valve will normally be choked-down. Unless the valve is modulated, as thickened slurry begins to appear at the V-SEP product slurry discharge valve, the slurry-side pressure increases dramatically. This causes higher transient flux rates and increases the potential for plugging.

Control Action: Control the slurry side pressure with a pressure control valve on the V-SEP product slurry outlet.

e. Improving Flux Rate
The previously mentioned means of increasing the flux rate in the WTI/CRE process include application of elevated temperatures and flow/pressure pulsation. Temperature control in the pilot plant was manual, but is easily automated. Feed slurry flow pulsation is easily accommodated, assuming the product slurry discharge piping is of sufficient size such that it is not a significant restriction to flow, by automatically modulating the product slurry discharge pressure control valve over a timed interval.

Task F Equilibrium Flux Rate at Elevated Slurry Concentration
(Formerly “Long Duration Testing”)
After discussions with the ICCI Project Manager, it was felt that some crucial questions regarding equilibrium operation of the plant in the P-mode would be left unanswered if the original Task F course were taken. As a result, the Task F work plan was revised.

Formerly, the Task F work was to consist of two weeks of continuous operation utilizing the pilot plant in the P-mode of operation. The test was to consist of feeding slurry to the separator and returning the product slurry and separated water back to the feed tank in a
continuous loop. Testing was to include one week of operation at ambient temperature and one week at an elevated temperature. Operating and performance data were to be taken hourly over the course of the runs. There are several issues which limited the usefulness of such testing. In summary, they include:

a. Some piping within the separator is very small in diameter; about 1/4 inch. At the flow rates used for the test, the velocity of the slurry in these sections ranged from between 22-25 ft/sec. Over the course of testing, this could cause particle size degradation. There is an accompanying decrease in permeate flux rates with decreasing particle size. Since the properties of the feed are continuously changing, the usefulness of any separation performance data is dubious. Therefore, the data obtained would only be useful with regard to physical demonstration of the equipment over the run period.

b. It was demonstrated earlier in the program that the New Logic Series L/P separator was not capable of providing process scale-up data when operating in the “P-mode”. Unit water removal rates in the P-mode are generally lower than that found in L-mode testing by between 20% and 40%.

The particle size degradation problem could have been remedied in a couple of ways: a) change the piping associated with the vendor separator package, or b) feed the plant with a continuous supply of fresh feed and continuous removal of product slurry from the system. Neither of these options was efficable at this point in the test program.

The procedure for accomplishing the revised Task F scope was as follows:

a. A one-barrel sample of Consol Rend Lake Froth Flotation Product feed slurry was prepared. The feed barrel had an initial level of approximately 30 inches and a concentration of 12-16 weight percent. An agitator and separator feed pump suction piping were installed. Feed slurry samples were taken.

b. Slurry was fed to the membrane separator, returning both product slurry and permeate water to the feed barrel. When an equilibrium initial flux rate had been established (i.e., a constant flux rate over a 15 minute interval), the concentration test was started.

c. The permeate line to was diverted to a separate barrel while circulating the concentrate back to the feed tank. A sample of the concentrate for each pass was taken.

d. When the level in the feed tank dropped by about five inches, concentration of the feed was stopped by returning permeate back to the feed barrel. The levels in both the feed barrel and permeate barrel were recorded before resuming slurry concentration. This procedure was repeated until the slurry could no longer be concentrated (i.e., became too thick).
e. When the test was completed, a final product slurry sample was taken. All slurry samples were tested for solids concentration.

f. At the end of the slurry concentration, the unit was operated for several hours at the a high slurry loading to observe the unit operation over time at an elevated slurry concentration.

g. The test was repeated at a temperature of 120 °F.

**Task F Experimental Results**

Task F work consisted of four runs denoted Run A, B, C, and D. The first three were to measure the relative change in flux rate as slurry solids concentration increased. The last, and most interesting, was to operate the pilot plant at elevated concentration over time.

**Run A -- Flux Rate vs. Concentration at Elevated Temperature**

For Run A, the V-SEP was operated at the nominal conditions of 120 °F and 60 psi at the V-SEP discharge. Table 3 give Run A results. No attempt was made to achieve ultimate loading since the same membranes were to be used in the next set of experiments.

**Table 3 -- Run A Results**

4-Tray Concentration of Rend Lake Flotation Product  

<table>
<thead>
<tr>
<th>Membrane Area</th>
<th>3.30 sq.ft.</th>
<th>0.5 μ Teflon</th>
<th>No.Trays</th>
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</tr>
</thead>
<tbody>
<tr>
<td>Date</td>
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<td></td>
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<tr>
<td>Run</td>
<td>Time (min.)</td>
<td>Temp (°F)</td>
<td>Feed Flow (gpm)</td>
<td>Flux Rate (GFD)</td>
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<td>199</td>
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<td>119</td>
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</table>
The experiments indicate that the flux rate dropped rapidly in the initial part of the run. It was suspected that the membranes became damaged during start-up. The results of Run B support this.

Run B -- Flux Rate vs. Concentration at Ambient Temperature

It became immediately apparent that the membrane stack was damaged when unusually low flux rates were noted at the onset. Further damage was not suspected upon start-up of this run. The run history for this test is displayed in Table 4.

Table 4 -- Run B Flux Rate vs. Concentration

<table>
<thead>
<tr>
<th>Clock Time (hrs.)</th>
<th>Run Time (min.)</th>
<th>Temp (°F)</th>
<th>Flux TI-06 Rate (GFD)</th>
<th>Product Flow (gpm)</th>
<th>Solids (wt.%)</th>
<th>Slurry Inventory (inches)</th>
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</thead>
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<tr>
<td>835</td>
<td>0</td>
<td>65</td>
<td>64</td>
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<td>17.50%</td>
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<td>45</td>
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<td>60</td>
<td>2.83</td>
<td>23</td>
<td></td>
</tr>
<tr>
<td>950</td>
<td>75</td>
<td>69</td>
<td>61</td>
<td>2.73</td>
<td>18.47%</td>
<td>20</td>
</tr>
<tr>
<td>1000</td>
<td>85</td>
<td>69</td>
<td>60</td>
<td>2.73</td>
<td>20</td>
<td></td>
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<tr>
<td>1030</td>
<td>115</td>
<td>70</td>
<td>60</td>
<td>2.82</td>
<td>19</td>
<td></td>
</tr>
<tr>
<td>1105</td>
<td>150</td>
<td>72</td>
<td>59</td>
<td>2.70</td>
<td>27.66%</td>
<td>16</td>
</tr>
<tr>
<td>1115</td>
<td>160</td>
<td>72</td>
<td>60</td>
<td>2.70</td>
<td>16</td>
<td></td>
</tr>
</tbody>
</table>
Flux rates remained relatively constant throughout the run. This was also observed in Run D; the most successful run. The slurry was concentrated to 55.2 weight percent by the 230th minute of run time. Because the slurry inventory in the feed barrel dropped to about 3-1/2 inches, water was added back to continue the run and observe the concentration behavior again. The run was continued for an additional 3-1/2 hours before termination.

Inspection of the membranes showed that the failures occurred mainly at the slurry outlet to each membrane tray which damaged 1/2 of the total membrane area. This was a problem with the start-up strategy used and was corrected in subsequent runs. This type of the failure is specific to the P-Series V-SEP unit and the mechanism is described earlier in this report.

Run C -- Concentration at Ambient Temperature

Run C was started after replacing the faulty membranes from Runs A and B, but was terminated on start-up due to mechanical failure.

Run D -- Operation at Elevated Concentration Over Time

The optimum start-up strategy for the P-Series machine was demonstrated in this run. The following Table 5, Figure 11, and Figure 12 are from data taken in Run D.

**Table 5 -- Run at Elevated Slurry Concentration (Run D)**

<table>
<thead>
<tr>
<th>Membrane Area:</th>
<th>4.95 sq.ft.</th>
<th>Membrane Material:</th>
<th>0.5 μ Teflon</th>
<th>No. Trays: 6</th>
</tr>
</thead>
<tbody>
<tr>
<td>Date:</td>
<td>22-Jan-96</td>
<td>Temp Time</td>
<td>Pressure Time</td>
<td>1/8 Gallon Flux Rate</td>
</tr>
<tr>
<td>Time (hrs.)</td>
<td>Time (min.)</td>
<td>TI-06 (°F)</td>
<td>PI-06 (psi)</td>
<td>Flux (sec.) (GFD)</td>
</tr>
<tr>
<td>1945</td>
<td>0</td>
<td>61</td>
<td>41</td>
<td>21.3</td>
</tr>
<tr>
<td>2005</td>
<td>20</td>
<td>62</td>
<td>45</td>
<td>24.5</td>
</tr>
<tr>
<td>2015</td>
<td>30</td>
<td>76</td>
<td>45</td>
<td>20.95</td>
</tr>
<tr>
<td>2030</td>
<td>45</td>
<td>92</td>
<td>45</td>
<td>16.75</td>
</tr>
</tbody>
</table>
It is notable that, after achieving the target temperature of 120 °F, the flux rate did not drop off significantly with increased solids loading.

Run D was interrupted in the 5th hour of operation due to a leak in the permeate line. The pilot plant was shut-down smoothly, the leak repaired, and the run smoothly re-started. Immediately upon restarting the run a low flux rate was observed. Without any special care, the flux rate re-established itself to approximately its pre-shut-down value. This is significant since it shows that operation can be restored to original performance after a major upset when proper start-up and shut-down procedures are followed. The procedure for automating the start-up and shut-down procedure is covered in “Task E -- Process Control Strategy Tests.”

The run was terminated after 11-1/2 hours of run time due to a pump failure.
Task F -- Operation at High Concentration

Flux Rate vs. Temperature

Temperature (F)

Flux Rate (GPD)

Figure 11

Task G -- Tailings Concentration

This Task was completed during the First and Second Quarters.

Task H -- Destec IGCC Fuel Evaluation
Destec evaluates slurry fuels for their IGCC technology by modeling gasifier performance based upon the parameters of slurry concentration and coal properties from the dry coal proximate and ultimate analyses.

The dry coal analyses reported to Destec were as follows:

<table>
<thead>
<tr>
<th>Ultimate Analysis</th>
<th>Proximate Analysis</th>
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<tbody>
<tr>
<td>C</td>
<td>Vol. Matter</td>
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<tr>
<td>78.6 %</td>
<td>35.1 %</td>
</tr>
<tr>
<td>H</td>
<td>Fixed Carbon</td>
</tr>
<tr>
<td>5.0 %</td>
<td>59.2 %</td>
</tr>
<tr>
<td>N</td>
<td>Ash</td>
</tr>
<tr>
<td>1.8 %</td>
<td>5.7 %</td>
</tr>
<tr>
<td>O</td>
<td>HHV</td>
</tr>
<tr>
<td>7.8 %</td>
<td>13,782 Btu/lb</td>
</tr>
<tr>
<td>S</td>
<td></td>
</tr>
<tr>
<td>1.15%</td>
<td></td>
</tr>
</tbody>
</table>

Table H-1 displays the results of two model runs provided by Destec for slurries with solids concentrations of 50.0 wt.% and 61.9 wt. % solids. Both scenarios compare favorably with standard nominal conventional power generation heat rates of about 10,000 BTU/KWHe. There is, however, an obvious benefit to higher loading as can be seen by the 11% improvement in efficiency for the higher solids concentration.

Table H-1 Destee Slurry Fuel Performance Model Results

ESTIMATED PERFORMANCE FOR FLOTATION-CLEANED COAL FINES SLURRY(1)

<table>
<thead>
<tr>
<th>Slurry Solids, wt.%</th>
<th>50.0</th>
<th>61.9</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coal Fines (Dry), TPD</td>
<td>2363</td>
<td>.2107</td>
</tr>
<tr>
<td>Oxygen Required (95%), TPD</td>
<td>2672</td>
<td>2135</td>
</tr>
<tr>
<td>Slag Product (dry), TPD</td>
<td>135</td>
<td>120</td>
</tr>
<tr>
<td>Sulfur Product</td>
<td>2732</td>
<td>24.2</td>
</tr>
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</table>

Syngas Properties

Dry Composition

<table>
<thead>
<tr>
<th>H2</th>
<th>36.05</th>
<th>32.43</th>
</tr>
</thead>
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<tr>
<td>CO</td>
<td>43.34</td>
<td>.51.29</td>
</tr>
<tr>
<td>CO2</td>
<td>16.48</td>
<td>10.84</td>
</tr>
<tr>
<td>CH4</td>
<td>1.15</td>
<td>2.52</td>
</tr>
<tr>
<td>H2S (ppm)</td>
<td>20</td>
<td>23</td>
</tr>
<tr>
<td>COS (ppm)</td>
<td>30</td>
<td>30</td>
</tr>
<tr>
<td>N2</td>
<td>1.69</td>
<td>1.73</td>
</tr>
<tr>
<td>Ar</td>
<td>1.28</td>
<td>1.18</td>
</tr>
</tbody>
</table>

Dry Molecular Weight | 20.31 | 20.42 |
Moisture Content, vol. % | 30.05 | 23.50 |
Heating Value (dry), BTU/SCF

| HHV | 267 | 295 |
| LHV | 248 | 276 |
Net Power, MW
301.2 302.4

Heat Rate (HHV), BTU/KWHR
9013 8003

(1) Based on Destec's coal gasification process supplying Syngas to a combined cycle plant utilizing an advanced 200 MW gas turbine.

5.0 Preliminary Economics

<table>
<thead>
<tr>
<th>Plant Operating Criteria</th>
<th>Feed (tpy, coal basis)</th>
<th>Un-Heated Feed (tpy)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coal</td>
<td>114.2</td>
<td>114.2</td>
</tr>
<tr>
<td>Water</td>
<td>456.8</td>
<td>76.1</td>
</tr>
<tr>
<td>Total</td>
<td>571.0</td>
<td>190.3</td>
</tr>
<tr>
<td>Solids Content</td>
<td>20%</td>
<td>60%</td>
</tr>
<tr>
<td>Water Removed</td>
<td>1,521 gpm</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th></th>
<th>Heated Feed</th>
<th>Un-Heated Feed</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flux Rate</td>
<td>200 GFD</td>
<td>100 GFD</td>
</tr>
<tr>
<td>Area Required</td>
<td>10,954 sq.ft</td>
<td>21,909 sq.ft</td>
</tr>
<tr>
<td>No. of Machines</td>
<td>13</td>
<td>26</td>
</tr>
</tbody>
</table>

Installed Cost of Major Equipment
$3,191,971 $5,783,942
(Including pumps, valves, instruments, tanks, agitators)

Amortized Capital Cost
No. years 5
Interest Rate 10.0%

<table>
<thead>
<tr>
<th>Operating Costs</th>
<th>Feed</th>
<th>Un-Heated</th>
</tr>
</thead>
<tbody>
<tr>
<td>Labor, Utilities</td>
<td>$1,000,000</td>
<td>$1,000,000</td>
</tr>
<tr>
<td>Heat ($/yr)</td>
<td>$305,268</td>
<td>$0</td>
</tr>
<tr>
<td>Coal Fines (at $0.80/MMBtu)</td>
<td>$21,608,467</td>
<td>$21,608,467</td>
</tr>
<tr>
<td>Fuel Revenue (@ $1.00/MMBtu)</td>
<td>$27,010,584</td>
<td>$27,010,584</td>
</tr>
<tr>
<td>Net Revenue</td>
<td>$3,254,815</td>
<td>$2,876,327</td>
</tr>
</tbody>
</table>

A rough economic analysis of the technology is provided here to give an indication of the order-of-magnitude cost for a WTI/CRE facility to produce CWSF from coal fines from a generic source (e.g., prep plant fines, pond fines, milled slurry). Such a plant could have a near infinite number of configurations, so it has been assumed that major equipment includes V-SEP's, pumps, tanks, agitators, and instrumentation. Application specific equipment such as cyclones or other classifiers are not included. These costs have been lumped into one capital cost per installed V-SEP. A summary of all the assumptions and results are displayed in Table 5.1 below.

Excluded from the costs are engineering, building and infrastructure, freight, taxes, permitting, and contractor costs.
Included in the estimate is a comparison of the cost of heating the feed slurry from 68 to 102 °F. In this estimation, it is assumed that the cost of a 375 bhp boiler and associated plate and frame heat exchangers add $300,000 to the capital cost. This cost is offset by a $2.9 MM savings in V-SEP and associated equipment. A net annual amortized capital cost savings of about $675,000 is offset by an increase in operating cost (boiler fuel) of $305,000 per year for a net annual revenue increase of about $370,000/year. If waste heat is readily available, the heated feed option becomes even more attractive.

CONCLUSIONS AND RECOMMENDATIONS

1. The application of heat to improve de-watering performance was demonstrated in this program. Figure 5.1 below indicates the relative improvement in performance with temperature.

![Image of Flux Rate vs. Temperature graph]

*Figure 5.1*

Using the equation shown in Figure 5.1, a flux rate of 100 GFD is predicted at 68 °F. Equipment costs are cut in half by elevation of operating to 102 °F. In an installation to produce 1.0 MMTPY (coal basis) CWSF, the cost to raise the feed to this temperature is generally more than offset by the savings in capital equipment. If waste heat is available, the decision to heat the feed is unquestionably justified. In a new installation, direct steam injection and heat recovery are recommended.

2. The New Logic Series "P" is not an appropriate unit for obtaining scale-up data for larger slurry concentration facilities.
There are significant differences between the Series “P” (denoting pilot scale) and both the Series “L” (laboratory) and the Series “I” (industrial scale) equipment. The flow patterns in the membrane stack do not match either unit. The flow patterns in the Series “P” create operating problems which are distinctly different and than either of the other models.

The Series “L”, which has a flow regime which is similar to that of the “I” unit is much easier to operate. It is also claimed by New Logic International that the laboratory unit is scaleable to the commercial scale, whereas the Series “P” is not. This conclusion could only reasonably be justified by further testing at the vendor facility.

3. It was found that vibrational frequency (i.e., vibration amplitude) had little effect on de-watering performance in either the pilot or laboratory apparatus above a minimum value. In the pilot unit, it was found that when vibration was decreased below about 0.5 inches (as measured at the full diameter of the membrane stack), flux rates fell off dramatically. This was attributed to the inability of the low vibration to clear solids cake formation along the membrane surface. When vibration was increased above this value, the membranes would disrupt the cake and flux rates could be re-established. In general, smoothest operation was achieved above 0.75 inches of stack deflection.

The maximum allowable membrane stack vibration is a function of the standard vendor design. The vendor recommends operation at or below the maximum allowable design value. No further investigation into the effect of vibration appears warranted.

4. The effect of feed pulsing could not be established in the pilot configuration during this test program. The main cause of this was the small diameter piping included with the Series “P” V-SEP package which would not allow for rapid pressure/flow pulsations because of flow restriction. Improved water removal performance as a result of feed pulsing has, as yet only been observed in the laboratory (Series “L”) configuration.

It is recommended that future pilot programs or commercial development address the need for larger system piping and flow paths in the vendor supplied equipment to take advantage of the potential benefits of feed/pressure pulsation.

5. Observations during operation of the pilot plant suggested several operational issues which could be addressed by process control. Included were:

a. Means of preventing membrane failure as a result of general plant upsets.
b. Means of preventing membrane failure during routine start-up and operation.
c. Automated Start-Up Procedures
Means of Preventing Machine Plugging
Automated Means of Improving De-watering Performance

Future pilot plant and commercial development projects should include, at a minimum, the necessary instrumentation and controls for smooth automatic start-up and shut-down procedure as outlined in “Task E” of this document.

6. Task F investigated equilibrium flux rate at elevated concentrations. In the last, and most significant test, it was found that:

a. Flux rate did not drop dramatically as concentration increased to high loading. In other V-SEP applications, where the particle size is normally much smaller, a steady drop in flux rate is usually observed. The vendor claims that this drop is normally associated with increasing slurry viscosity. It is felt that, because of the relatively large particle size of the coal feedstock, this effect was not evident.

b. Flux rates could be re-established if orderly shut-down and re-start-up procedures are followed.

7. No significant effect of increased system pressure on flux rate was found. This confirms the results found in earlier “L” Series test work. One explanation for this is that increased system pressure leads to increased slurry density at the membrane surface which increases the “cake” resistance to permeate flow. The effect is not well understood and could be a function of the coal particle geometry.

This effect may be slurry specific. The vendor indicates that there is some benefit to increased pressure with certain other materials they have tested.

It is recommended that the effect of pressure be investigated for each new slurry to be processed before designing a plant.

8. Consol suggested that this technology may prove useful in de-watering tailings muds to either provide a tailings paste for mine backfill or as an improved feed to existing filtration equipment. Testing of tailings mud in this program was not successful due to the ultra-low solids content (less than 1.0 wt.% ) and apparent fineness of the muds received from Consol’s Rend Lake preparation plant. Limited testing with the material showed that de-watering performance of the equipment did not appear to be economically interesting. However, this assertion is somewhat dubious in the absence of testing with a more representative sample.

9. Model results of CWSF performance in Destec’s IGCC technology at solids contents of 50 and 62 weight percent solids both showed high heat rates as compared to normal pulverized coal power plant performance. IGCC heat rates of
about 9,000 and 8,000 Btu/KWH respectively compare favorably with 10,000 Btu/KWH or more found in conventional power plant practice.

For a given coal fines stream, the maximum attainable solids loading may only be 50-55 wt. %. If so, there are numerous ways to augment the solids loading to achieve a 60% or better coal loading. Normally, the ultimate solids loading is a function of both solids density and particle size distribution. For example, suppose the particle size of a particular recovered fines slurry is such that only a 55% loading is attainable. Coarser size fractions (e.g., -28 mesh x 100) could be mixed with the slurry to increase the solids loading. In this case, the addition of 0.12 lb of coarse coal per pound of slurry would bring the loading up to 60%.

DISCLAIMER

This report was prepared by Marcus Rajchel representing Williams Technologies, Incorporated of Tulsa, Oklahoma with support in part by grants made possible by the U.S. Department of Energy Cooperative Agreement Number DE-FC22-92PC92521 (Year 4) and the Illinois Department of Commerce and Community Affairs through the Illinois Coal Development Board and the Illinois Clean Coal Institute. Neither Marcus Rajchel nor Williams Technologies nor any of its subcontractors nor the U.S. Department of Energy, the Illinois Department of Commerce and Community Affairs, Illinois Coal Development Board, Illinois Clean Coal Institute, nor any person acting on behalf of either:

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PROJECT MANAGEMENT REPORT  
March 1, 1996, through May 31, 1996

Project Title: **INNOVATIVE PROCESS FOR CONCENTRATION OF FINE PARTICLE COAL SLURRIES**

DOE Cooperative Agreement Number: DE-FC22-92PC92521 (Year 4)  
ICCI Project Number: 95-1/5.2A-3P  
Principal Investigator: Marcus Rajchel, Williams Technologies, Inc./Clarke Rajchel Engineering Joint Venture (WTI/CRE JV)  
Other Investigators: Henry P. Ehrlinger, Consultant; Dan Harnett, WTI; A. Fonseca, Consol; R. Maurer, Destec Energy  
Project Manager: Dr. K. Ho, ICCI

COMMENTS

**Project Schedule**

Work during the Quarter consisted of data reduction and report preparation. A draft Final Report was submitted to ICCI. A revised Final Report will be issued as soon as the changes are made.

**Projected Expenditures**

Remaining ICCI funds were spent in this Quarter.
### Projected and Estimated Expenditures

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<th>Quarter*</th>
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<th>Direct Labor</th>
<th>Fringe Benefits</th>
<th>Materials &amp; Supplies</th>
<th>Travel</th>
<th>Major Equipment</th>
<th>Other Direct Costs</th>
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<td>4,960</td>
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<td>--</td>
<td>22,550</td>
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<td>--</td>
<td>23,050</td>
<td>6,287</td>
<td>77,956</td>
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<tr>
<td>Feb. 29, 1996</td>
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<td>6,215</td>
<td>1,926</td>
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<td>75,484</td>
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<td>Sept. 1, 1995 to</td>
<td>Projected</td>
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<td>8,119</td>
<td>2,000</td>
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<td>2,000</td>
<td>21,050</td>
<td>6,287</td>
<td>77,956</td>
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<td>May 31, 1996</td>
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<td>7,931</td>
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<td>6,600</td>
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<td>21,281</td>
<td>6,522</td>
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<td>Sept. 1, 1995 to</td>
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<td>21,050</td>
<td>6,287</td>
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* Cumulative by Quarter
COSTS BY QUARTER
Innovative Process for Concentration of Fine Particle Coal Slurries

Projected + Estimated Actual

Total ICCI Award $77,956.
## SCHEDULE OF PROJECT MILESTONES

<table>
<thead>
<tr>
<th>TASK</th>
<th>1995</th>
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<tbody>
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<td>OCT</td>
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<tr>
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<tr>
<td>L</td>
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</tbody>
</table>

### RESEARCH TASKS

- A. Project Kick-Off & Planning
- B. Procurement & Set-Up
- C. Ambient Temperature Tests
- D. Elevated Temperature Tests
- E. Optimum Frequency & Pulse Tests
- F. Process Control Strategy Tests
- G. Long Duration Testing
- H. Tailing Paste Production
- I. Destec Slurry Fuel Evaluation
- J. Analytical Testing
- K. Technical Reporting
- L. Project Management Reporting

### COMMENTS

Problems with the operation of the test apparatus led to extension of the laboratory phase of the project and changes in the scope of work. All laboratory work was completed in January, 1996.

A draft Final Report has been written. The revised Final Report will be issued as soon as all changes have been made.