Project: Study of Soluble Scale Fouling Control in High Solids Black Liquor Concentrators (DE-FG36-99GO10387)

Milestones and Progress, Quarter 4 (July 1, 1999 – September 30, 1999)

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Summary

The quarterly project review meeting was held at IPST on August 2, 1999 and was attended by IPST, Georgia Tech, and ORNL principal investigators, members of the Industrial Advisory Group, and a U.S. DOE representative. Although steady progress is being made, this project is currently behind schedule. The specific tasks that are behind schedule, the reasons for the delays, and the expected completion dates are discussed in the *Performance Variances and Open Items* section below. The remaining tasks are either on schedule, or have not been started.

Performance Variances and Open Items

Task A.1, Solubility experiments – one kraft black liquor (scheduled completion date: 6/99), and Task A.2, Solubility data obtained – other liquors (scheduled completion date: 9/99). Both of these tasks are behind schedule due to needed modifications in the equipment and procedures (described in the Quarter 3 report), and to our technician being unavailable because of an extended illness. We have regained our technical support and added a part time technician. Our plan is to complete Task A.1 in Quarter 6, and Task A.2 by the end of Quarter 7.

Task B.1, Crystallization experiments – inorganic model solutions (scheduled completion date: 9/99). This task is behind schedule because of delays in obtaining funding to buy the Lasentec® FBRM 600L system. We expect this task to be completed by the end of the current quarter (December 1999).

Task C.1, Pilot falling film evaporator operational (scheduled completion date: 9/99). This task is behind schedule due to slower than expected progress in installing the pilot evaporators. We expect to complete the pilot falling film evaporator installation by the end of the current quarter (December 1999), and to have it operational in Quarter 6.

Task D.1.3, Relax simplifying assumptions (scheduled completion date: 7/99), and Task D.1.4, Compare to experimental results: preliminary analysis (scheduled completion date: 9/99). These two tasks are behind schedule because of the change in personnel at ORNL that was described in the Quarter 3 report. We anticipate completing Task D.1.3 by the end of the current quarter (December 1999), and Task D.1.4 in Quarter 6.

Status of Progress by Task

A.1. Solubility experiments – one kraft black liquor.
Scheduled completion date: 6/99.
Current status: **in progress.**

Progress during the quarter:

The modifications to the equipment and the operating procedures, which were described in the Quarter 3 report, have been completed. However, no new experimental work has been performed.

A.2. Solubility data obtained – other liquors.
Scheduled completion date: 9/99.
Current status: **not begun.**

A.3. All solubility data analyzed.
Scheduled completion date: 1/00.

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Current status: **not begun.**

B.1. Crystallization experiments – inorganic model solutions.

Scheduled completion date: 9/99.

Current status: **in progress.**

Progress during the quarter:

A series of experiments have been carried out to study the effect of black liquor content on the metastable zone width of the inorganic model solutions. Black liquor was added at one percent into the solution, and the results showed that the metastable zone width of Burkeite decreased when compared to black liquor free solutions.

Equipment to study the secondary nucleation kinetics of the model solution has been installed and preliminary experiments with this new equipment have been completed. A Lasentec® FBRM 600L system is in place to monitor crystal size, and as the inorganic solutions have been evaporated at constant rates, two critical points have been observed for solutions with high sodium carbonate to sodium sulfate ratios. The distances between the first and the second critical points decrease as the carbonate to sulfate ratios increase. Nucleation rates at the first critical point are greater than those at the second critical point.

B.2. Crystallization experiments – one kraft black liquor.

Scheduled completion date: 9/00.

Current status: **not begun.**

B.3. Crystallization experiments – other kraft black liquors.

Scheduled completion date: 3/01.

Current status: **not begun.**

B.4. Crystallization data analyzed and modeled.

Scheduled completion date: 5/01.

Current status: **not begun.**

C.1. Pilot falling film evaporator operational.

Scheduled completion date: 9/99.

Current status: **in progress.**

Progress during the quarter:

Work has continued on the installation of equipment for the pilot-scale falling film plate evaporator. Additional piping and instrumentation work have been completed during the past quarter. The piping for the main recycle loop has been completed; this includes a Moyno pump with variable speed control and connections for liquor sampling and instrumentation. The black liquor feed system has been installed which uses a gear pump to feed the black liquor from the supply tank to a mix point in the recycle loop. A hot water flush system was added to allow for clean up of the plate and piping after a test has been performed. The piping for the steam side of the evaporator has also been completed. This includes the connection from the building steam supply, with a regulator and the necessary valving. At the condensate side of the evaporator plate outlet, a steam trap was installed followed by piping to a condensate collection tank.

Instrumentation is partially complete for the plate evaporator unit. The temperature probes have been installed throughout the system. Both 100-ohm RTDs and Type K thermocouples are being used, with the RTDs used where higher accuracy is required. Pressure gauges and transmitters have been ordered and should be installed quickly. A magnetic flowmeter is installed on the main re-circulation loop to provide a measure of the total flow to the plate.

The annular test cell (ATC) unit for measurement of fouling rates has also been installed and is nearly complete. With one exception (replacement of the power control SCR), all of the equipment for this unit has been assembled in the high bay, and all of the instrumentation is in place. The entire system has been insulated to reduce the heat loss and increase the operating efficiency and safety. Preliminary testing has been performed on the system to

verify that: 1) the piping is correct and there are no leaks, 2) the pumps all work at the designed flow-rate, and 3) the heaters and heat exchangers are working correctly.

C.2. Pilot evaporator data for fouling rate model completed.

Scheduled completion date: 9/00.

Current status: **in progress.**

Progress during the quarter:

An experimental technique is under development to quantify the appearance and potentially the magnitude of black liquor fouling at higher solids. This technique involves slowly concentrating black liquor past the critical solids level and measuring the resultant dramatic increase in the fouling rate within the evaporator.

C.3. Fouling rate model complete.

Scheduled completion date: 5/01.

Current status: **not begun.**

C.4. Pilot evaporator data – to evaluate fouling rate model.

Scheduled completion date: 5/01.

Current status: **not begun.**

D.0.1. Model survey.

Scheduled completion date: 1/99.

Current status: **complete.**

D.0.2. Literature review.

Scheduled completion date: 1/99.

Current status: **complete.**

D.0.3. Understand evaporation technology and multiple effect evaporation process.

Scheduled completion date: 1/99.

Current status: **complete.**

D.1.1. Assemble solution properties.

Scheduled completion date: 2/99.

Current status: **complete.**

D.1.2. Devise flow dynamics model with simplifying assumptions.

Scheduled completion date: 5/99.

Current status: **complete.**

Progress during the quarter:

Graphical flow field results from three-dimensional simulations, with simplifying assumptions in place and using the FLUENT 5.0 computational fluid dynamics code, were presented to project, industry, and U.S. Department of Energy representatives at the August 2, 1999 project review and coordination meeting.

D.1.3. Relax simplifying assumptions.

Scheduled completion date: 7/99.

Current status: **in progress.**

Progress during the quarter:

Selected references from the literature were identified which examined potential complicating factors that can cause departures from the simplifying falling film assumptions. Expressions were developed to estimate relations between film Reynolds number and film thickness for approximated black liquor evaporator conditions.

D.1.4. Compare to experimental results: preliminary analysis.

Scheduled completion date: 9/99.

Current status: **in progress.**

Progress during the quarter:

Evaluations of the ability of the selected relations to predict experimentally observed film thickness from references were initiated. Subsequent discussions with IPST indicated an interest in online measurement of black liquor film thickness on the surface of the pilot falling film plate evaporator. Inquiries were made concerning expertise available at ORNL to implement an accurate, reliable, and inexpensive method to accomplish this task.

Instrumentation personnel at ORNL have developed liquid film measurement techniques based on electrical admittance principles, and devices employing these techniques have been fabricated at ORNL for specialized application to water-cooled nuclear reactor accident simulation experiments. At this point, it does not appear that such devices could be adapted easily or inexpensively to the indicated black liquor evaporator situation.

D.2. Heat transfer effects incorporated into CFD model.

Scheduled completion date: 9/00.

Current status: **not begun.**

D.3. CFD model for falling film evaporator fouling evaluated.

Scheduled completion date: 7/01.

Current status: **not begun.**

E.1. Monograph completed.

Scheduled completion date: 9/01.

Current status: **not begun.**

Goals For The Period October 1 - December 31, 1999

Task A.1.

1. Repeat the equilibrium study to establish the time required to reach equilibrium--evaporate the liquor to three or four different solids level and then sample as a function of time.
2. Run a critical solids evaporation experiment to as high a solids as possible.

Task B.2.

1. Develop a method to convert the chord length distribution data provided by the Lasentec® FBRM 600L system to the particle size distribution data that is required for correlating crystallization kinetics.
2. Begin experiments on crystallization studies in black liquor.

Task C.1.

1. For the pilot-scale falling film plate evaporator:
 - Complete Piping for the pilot plate evaporator system. The remaining sections include:
 - 1) The black liquor vapor collection system, consisting of a water-cooled condenser, a vacuum pump, a condensate collection tank and a diverter valve to recycle the condensate.
 - 2) The black liquor product discharge system. The black liquor product piping will include either a gear pump or a control valve to maintain the correct product flow for steady evaporator operation.
 - Finish the instrumentation and data acquisition system for this pilot unit.

- Perform preliminary testing on the system to verify the operation of the pumps, piping, heaters, instrumentation, and control systems.
2. The remaining work on the ATC system includes:
- Installation of a new SCR or Variac to provide uniform control of the heat flux in the annular region, as measured by the existing power meter.
 - Calibration of the instrumentation and data acquisition system.
 - Final check out of the system with water.
 - Initial testing of the system with a CaSO_4 solution which has a known fouling rate.

Task D.1.

1. Continue using literature references to estimate falling film parameter relations.
2. Continue evaluations of relations to predict reference experimental data.