July 2007

Monthly Research Progress

Institute for Clean Energy Technology
(formerly Diagnostic Instrumentation & Analysis Laboratory)
Mississippi State University
Dr. Roger King, Interim Director

Prepared for the U. S. Department of Energy
Office of Science and Technology
Cooperative Agreement No. DE-FC01-06EW07040

Task 1 Support of Oak Ridge Site Closure

Characterization of Corrosion for Closure of Oak Ridge Research Reactor

Due to the lack funds for the 2007 federal fiscal year, our Oak Ridge collaborators were unable to provide the support required to enable deployment into the Oak Ridge Research Reactor (ORRR) pool.

Task 2 Support of Hanford Single Shell Tank Waste Disposition

In-tank/At-Tank Characterization for Closure of Hanford Waste Tanks

Stereovision. Progress on the stereovision effort was significantly slowed during July by the unexpected departure in December of the graduate student working on this effort. The retroactive recision of CA07 funds in May have prevented replacement of the graduate student. Lack of manpower significantly limits progress on this and other efforts.

Fourier Transform Profilometry. This month the ICET Fourier Transform Profilometry (FTP) effort concentrated on addressing questions relating to how FTP handles curved (non-perpendicular) background surfaces (such as a curved waste tank bottom). Curved tank bottom will not be encountered in the multi-stage performance evaluation until Stage 3. In an effort to address all unresolved questions, to incorporate increased capabilities/improvements as soon as possible, and to have greater consistency in the FTP system utilized in the different Performance Evaluation stages, this technical issue is being addressed now.

At the request of our Hanford collaborators, a PowerPoint presentation on the FTP analysis procedures was prepared and discussed with our Hanford collaborators during a bi-weekly conference call.

Also during this month, revised ICET CA07 manpower allotments and budgets were finalized. Some additional manpower was gained by reassignment from other ICET CA projects.

Process Chemistry and Operations Planning for Hanford Waste Alternatives

Data obtained during the "dry" Gibbsite to Boehmite transition experiments was evaluated. The primary parameter effecting the transition was temperature, not caustic concentration. Results from XRD analysis were in excellent agreement with those from the TGA experiments. One way to more closely mimic waste contained within site tanks was to perform the experiments in sealed vessels. In this way, evaporation would be minimized. One test commonly used to evaluate waste glass acceptance is the product consistency test or PCT. Vials for the PCT are stainless steel and are equipped with a lid and a screw-cap. Samples were made up such that only a limited volume was available for the vapor phase. Experiments have begun and will be analyzed using TGA and XRD. It is believed that the current "wet" experiments will be more similar to actual tank conditions. Additional experiments are also planned at lower temperatures to determine the conditions at which conversion of Al(OH)3 to AlOOH takes place. The data can then be correlated with historical temperature records from specific tanks thereby providing an indication of the fractions of Gibbsite and Boehmite in current waste inventories. Such information is expected to be useful in the design of sludge leaching and washing flow sheets. Evaluation of the available literature indicates Gibbsite dissolves rapidly in sodium hydroxide solutions whereas the kinetics for Boehmite (AlOOH) dissolution is very slow. Thus, not considering the fraction of Boehmite within the tank waste may well result in difficulties when generating low activity wastes from the parent wastes. Delays in the sludge washing step would be expected to impact downstream processes such as glass production.

Task 3 Disposition of Idaho HLW Calcine

Support of the CH2M-WG Idaho Calcine Disposition Project

A mid-year report was issued July 27.

The fourth pilot-scale run (in mid-June) was designed to have about 10% less water in the batch in an attempt to increase the compressive strength. This was the largest batch tested so far. Feeding time for the powders was approximately 30 minutes. The mixing time (held constant over all four runs) was 20 minutes.

Nine samples were taken, 3 at the beginning (1A, 1B, and 1C), 3 in the middle (2A, 2B, and 2C), and 3 at the end (3A, 3B, and 3C). Results of the compression strength testing are given in the table below. Acceptable strengths are starting to be reached after aging for about a month.

A fifth run will be scheduled for early August.

Compressive strength of cubes made during Pilot #4.		
Sample	**Breaking Stress (psi)**	**Cure Time (days)**
1A	375	14
2A	344	14
3A	375	14
1B	360	32
1C	298	32
2B	413	32
2C	548	32
3B	725	32
3C	455	32

Task 4 Support of SRS Salt Disposition and Other SRS Alternatives

Modeling and Experimental Support for High-level SRS Waste Disposition

Modeling of the Sludge Batch 5 leaching process continued. Additions of various volumes, ranging from 100,000 to 150,000gallons of 50% NaOH, indicated complete dissolution of the aluminum either Boehmite or Gibbsite at volumes greater than 140,000 gallons. Temperature surveys were then performed indicating that an additional 6^{o}C of safety would result in using the 150,000 gallon addition. Solids precipitation (Boehmite and then Gibbsite) began at 38^{o}C, as opposed to the stream leached with 140,000 gallons of the 50% NaOH where solids re-precipitation was predicted to commence at 44^{o}C. The next stage of the simulations will be blending the aqueous phase leached solution with fractions from the DDA process and with the effluent treatment facility and DWPF recycle streams. The initial salt dissolution target will be the waste contained in SRS tank 41H. Calculations will be performed to analyze solids re-precipitation and compliance with corrosion and saltstone waste acceptance criteria (WAC).

Process Improvements for the Defense Waste Processing Facility (DWPF): On-line Analysis

The new surrogate batch for LIBS pellet measurements was prepared. This batch contains all the elemental components that will be present in the surrogate feed materials of plutonium disposition project. LIBS data of the new batch were collected at different experimental conditions and compared with the data collected with previous batch. We found that LIBS signals for most analyte lines from the new batch are weaker and a little shifted. We are examining the broadband spectrometer and communicated with the experts at LLA Instruments GmbH to identify possible problems of the broadband spectrometer.

Process Improvements for the Defense Waste Processing Facility (DWPF): Improvement of Waste Throughput

A new series of crucible-scale melts has been prepared to study Sludge Batch 5. We mixed SB5-2 SRAT Product with four different frit compositions:

Frit ID	B2O3	CaO	Li2O	Na2O	SiO2	Total
503	14	0	8	4	74	100
517	17	0	10	3	70	100
519	20	0	9	3	68	100
521	10	1	8	6	75	100

Four different waste loadings, 35 wt%, 38 wt%, 41 wt%, and 44 wt%, have been studied. We had Frit 503 on hand from previous experiments, but needed to make our own frits for the other three.

To make the other three frits, we used the following raw materials:

Glass sand, SiO_2
Sodium Carbonate, Na_2CO_3
Calcium Carbonate, $CaCO_3$
Lithium Carbonate, Li_2CO_3
Boric Acid, H_3BO_3

The frits were prepared by melting the raw materials in a 500mL platinum/rhodium crucible at 1400C for about one hour. Each batch was designed to provide approximately 175g of glass. Four batches were made for each of the three frits (517, 519, and 521). The glass was poured onto a cold steel plate and allowed to cool.

After cooling, the glass was broken up and ground in a mill in order to create a particular particle size. Sieving was done using a Ro-Tap sieve shaker. The particle size used for these experiments was -80 +200 mesh.

The powdered simulant and frits were mixed to provide waste loading of 35 wt%, 38 wt%, 41 wt%, and 44 wt%. The mixtures were placed in 50mL alumina crucibles heated for two hours at five temperatures (700C, 750C, 800C, 850C, and 900C) to study the series of reactions.

All the experiments are now complete. We will analyze the results and write a draft report next month.

Process Improvements for the Defense Waste Processing Facility (DWPF): Melter Monitoring

Task 5 DOE Headquarters Support

DOE HQ Road Map

Workshop on Heavy Metal Phytoremediation

HEPA and Regenerable Filter Performance Assurance

Work performed this month consisted of 10 load/backpulse cycles on filter #1. The filter was loaded as close to approximately 15 in w.c. pressure drop during each test. The filter was then weighed in order to quantify mass captured. The filter was then backpulsed 3 times at 100 psi. The pressure drop across the filter was measured. Finally, the filter was weighed again to determine how much salt challenge was removed from the filter during the backpulse. Five load/backpulse cycles were also completed on filter #2. This series of testing followed the same procedure as with filter #1.

Both filters were evaluated at 14 scfm flow rate. Conditions in the main test stand were modified slightly for these tests. The main test stand flow rate increased to 325 scfm from the typical 250 scfm. The aerosol generator was run without the cyclone in place.

Testing planned for next month includes completing additional load/backpulse cycles on the CeraMem filters.

Bio-availability Studies of Mercury and Other Heavy Metal Contaminants in Ecosystems of Selected DOE Sites

During this month, the new manuscript entitled “Phytotoxicity of mercury in Indian mustard (*Brassica juncea* L.)” was drafted. The paper discussed bioavailability and phytotoxicity of mercury in two varieties of Indian mustard.

Open Items (discuss any unresolved issues or items that require action by DOE or DIAL). None.

Status Assessment and Forecast (present analysis of program/project status, proposed solutions to problems, and future expectations regarding the project). We would like to continue summarization of the previous experiments and writing the manuscripts.

Phytoremediation and Long-Term Monitoring of Selected Heavy Metal and Radionuclide Contaminants

Approach Changes (description of any changes from the work plan, including technical changes, the explanation as to why these changes occurred, and what the impact on performance will be). None.

Performance Variances, Accomplishments, or Problems (discussion of accomplishments, problems, and/or variances, their causes, and the effects on the effort). During the month of July, we have started a new round of phytoremediation experiments using our homemade chamber. Boston fern (*N. exaltata*) was grown on clean soil, but the plant shoot was enclosed into a chamber containing mercury contaminated soil (with no direct contact to the plant). Our objective is to investigate the possible leaf uptake of mercury vapor from atmosphere above contaminated soil, and to compare the results with previous experiment with Chinese brake fern. The experiment is expected to finish in coming month(s).

Task 6 Technology Development

Development of New Technologies for DOE Site Applications

Efforts during this month focused on resolving optical issues emanating from the performance of the wavemeter. Suggestions offered by the manufacturer were implemented, and results show that the wavemeter does respond well in the red wavelengths, but not in the UV. The vendor is generating an RMA for the wavemeter to be shipped to them for further evaluation.

The ultimate goal of this project is to develop and deploy a portable and site deployable ringdown spectral technique for tank vapor characterization. Vigorous efforts in this month are focused on the hardware design and assembly of two major parts of the system, optical bench and electro-optical control portion. Each of the two parts has been assembled and tested individually. The electrical switch that controls the multiplexing of the laser diodes has been tested. Ringdown data transfer and processing rates have been tested. Robustness of the optical module has been tested. The parts (optical and electrical portions) are ready to be assembled together.

Development of Fiber Optical Sensor Technologies for DOE Site Applications

DOE reduced the FY 07 funding from $5M to $4M dollars for the Institute for Clean Energy Technology. With the reduction in funding, MSU management had to make hard decisions as to which tasks to continue. After a thorough examination of several factors it was determined that Task 6.2 Optical Sensors would be removed from the scope of work. This information has been conveyed to DOE.

Inquiries may be addressed to:
Dr. Roger King, Interim Director
205 Research Blvd.
Starkville, MS 39759
PHONE: 662-325-2105
FAX: 662-325-8465
E-MAIL: icet@icet.msstate.edu

This and other reports on research at ICET may be accessed at:
www.icet.msstate.edu