

## Monthly Research Progress

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

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### **Task 1 Support of Oak Ridge Site Closure**

#### *Characterization of Corrosion for Closure of Oak Ridge Research Reactor*

Due to the lack of 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 May by the unexpected departure in December of the graduate student working on this effort. Several graduate students interested in working on this effort were interviewed during April. The sudden and unexpected retroactive recision of 20% of the CA budget meant not only that a graduate student could not be hired for this project, but also that eleven Cooperative Agreement-supported graduate students lost their graduate research assistantships. Lack of manpower significantly limits progress on this and other efforts.

Fourier Transform Profilometry. At the beginning of May, the ICET Fourier Transform Profilometry (FTP) effort learned that two of the seven members of the FTP development team were being terminated at the end of May due to the retroactive recision of 20% of the CA07 budget. Those terminated are a Ph.D. mechanical engineer who has had responsibility for a comprehensive investigation of all factors relating to determination of uncertainty with regard to the FTP technique and instrumentation, and a technician whose duties have included fabrication and volume determination of the non-descript targets used in our multi-stage FTP Performance Evaluation testing. Some time was spent evaluating how these changes are going to affect the FTP effort in terms of both manpower and budget.

Efforts to validate our stitching software continued using synthetic images; a variety of tilt angles were simulated and the stitching results compared. A software error was discovered and corrected. The influence of FTP phase unwrapping procedures on the ability to obtain unsupervised volume determinations was reviewed.

*Process Chemistry and Operations Planning for Hanford Waste Alternatives*

Experiments continued on the Gibbsite to Boehmite transition at elevated temperatures. Samples of the Alcoa gibbsite were placed in vials with a volume of caustic. These were then heated at 120, 135, or 150°C and sampled as a function of time. Samples were then filtered at ambient conditions and the solid subjected to thermogravimetric analysis (TGA). Based on the starting material and the transition from Gibbsite to Boehmite to Alumina it was possible to analyze the weight loss data in terms of composition. This then allowed for an estimation of the amount of Gibbsite that reacted to form Boehmite during the experiments. Plans are to supplement the TGA data with XRD analysis on selected samples.

A data package was prepared and submitted to OLI Systems. The data will be incorporated in a mixed-solvent electrolyte database for permanent archival.

**Task 3 Disposition of Idaho HLW Calcine**

*Support of the CH2M-WG Idaho Calcine Disposition Project*

The results of the compression strength testing of the samples taken during the second run (completed in April) are:

|          |         |
|----------|---------|
| 7 days:  | 150psi. |
| 35 days: | 415psi  |
|          | 685psi  |

A third run was made May 9. The size of the batch was increased to about 15 gallons and the mixing process was altered so that as the materials were being added, the mixing was continuous. The compressive strength after 14 days was 371psi. Testing of the other two samples will be done after about 28 days.

The next run, set up for early June, will be a much larger (about 40 gallons). We are disappointed in the strength results and so will probably take three sets of three samples during this run to try to get more data. We will also begin thinking about what we should do to try to increase the strength without disrupting the rest of the process.

#### **Task 4 Support of SRS Salt Disposition and Other SRS Alternatives**

##### *Modeling and Experimental Support for High-level SRS Waste Disposition*

The  $\text{KNO}_3$  /  $\text{NaNO}_3$  and  $\text{CsNO}_3$  /  $\text{NaNO}_3$  systems in water, 1m and 3m NaOH solutions at 50°C have been filtered and are awaiting analytical results. Negotiations with site personnel have begun and review of the current work scope is underway.

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

For implementing LIBS in glove box at SRNL, a sample press will be used to make a solid surface from the powder. Pellet of powder sample will remain in the holder/die in which it is pressed. The sample press holder/die will be mounted on a rotating plate during LIBS measurement. During this work period, we have applied different pressures on the die that hold the surrogate powder to determine the minimum pressure needed to hold sample together without any binder. We found a pressure of 3000 psi is needed to make a solid surface pellet. We have also made pellet from various amount of surrogate powder to determine minimum weight of powder required for reproducible results. The preliminary study shows that the minimum weight of the powder for a 1 inch internal diameter die that can produce reproducible results is 5g.

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

A second series of crucible-scale melts has been started examining the behavior of mixtures of materials for Sludge Batch 4 (SRAT Product and Frits 418 and 503) at higher waste loadings (40 wt% and 45 wt%).

First impressions of the results appear to lead to the conclusion that higher waste loadings would be possible with these frits combined with this waste simulant. Further analysis will be done and a report evaluating both sets of experiments will be issued.

*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*

Air back-pulse cleaning studies using large CeraMem filters were continued. A set of three filters was loaded (2 in. w.c. above baseline) and back-pulsed at 25 psi, 50 psi, 75 psi, and 100 psi. This was done in an effort to find a back-pulse pressure that resulted in most efficient filter cleaning. It was discovered that varying the air back-pulse pressure had negligible effect on filter cleaning efficiency. This observation may suggest that the filters should be loaded to a greater extent prior to cleaning. Therefore, a change in the testing process was needed. It was decided that modifications to the test stand were in order to allow the test volumetric flow rate to be increased 7 scfm to 14 scfm. This modification will allow for loading more challenge on the filters per unit time, and may increase the level of pressure drop recovery after the back-pulse cleaning.

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

During this month, we focused on summarizing the previous experimental results. The first draft of the manuscript entitled “Stability and Bioavailability of HgS in Oak Ridge soils” was prepared. Due to the shortage of students, the project moves very slowly.

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 May, we have been analyzing plant and soil samples from previous experiments. We have also been processing data and preparing manuscripts for publication.

**Task 6 Technology Development**

*Development of New Technologies for DOE Site Applications*

Efforts during this month focused on obtaining mercury signals from elemental mercury naturally evaporating from spiked soil and solution samples. Due to an inconsistency between the stepper motor readout for the dye laser and the Burleigh wavemeter, attention was given to optimizing the experimental parameters to circumvent this discrepancy. The mercury peak was then readily located. Due to extensive use of the pulsed laser systems, the Nd:YAG laser and the dye laser, a few parts in the systems had to be fixed or replaced. For example, a replacement for the master power switch on the power supply to the YAG laser was ordered and installed. The experimental work resumed once the laser was operational.

The first C-H overtone spectra of many organic compounds are located in the NIR spectral region. The C-H overtone spectra of different compounds are partially overlapped; however, each compound has its own spectral features and some of which can be resolved in a specific spectral band. Therefore, in order to measure different tank VOCs, a careful selection of the C-H overtone spectral fingerprints must be done. The first C-H overtone of methane has been measured around 1590 (+/-1.5) nm. The multiplexing of the laser diode has been tested by measuring CO<sub>2</sub> and CH<sub>4</sub> at the two wavelengths simultaneously.

*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.

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