August 2007

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

Task 1 Support of Oak Ridge Site Closure

Characterization of Corrosion for Closure of Oak Ridge Research Reactor

Due to 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 August 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. During August, the ICET Fourier Transform Profilometry (FTP) effort continued its efforts to address how FTP handles curved (non-perpendicular) background surfaces (such as a curved waste tank bottom). An algorithm has been developed and the software coded. Preliminary testing of the implementation has been completed using synthetic images; the results are promising, but more extensive testing is needed.

A draft of the Task 2.1 FTP and Stereovision effort for CA08 was composed and discussed with our Hanford collaborators during a bi-weekly conference call. At the recommendation of our Hanford collaborators, ICET will present a conceptional design of the FTP deployment system to Hanford after completion of FTP Performance Evaluation Stage 4 (stitching of results of multiple images of objects on simulated curved tank bottom) and will provide information for a design review of the FTP deployment system after completion of Stage 5 (simulation of full-scale deployment at ICET).

Process Chemistry and Operations Planning for Hanford Waste Alternatives

Studies of the gibbsite to boehmite transition continued. Data from the "dry" experiments were compared with that obtained from the "wet" experiments using the product consistency test vials. Calculated activation energies from the dry and wet experiments were, within error, the same at 98 and 84kL/mol, respectively. Evaluation of pure gibbsite over the forced transition range of from 200-400^{o}C (using the TGA) resulted in an activation energy of 150kJ/mol. This value is in excellent agreement with earlier published work. Additional experiments are in progress to extend the investigation to measurements at 100^{o}C.

Data packets were transmitted to OLI Inc. for the porting of the double salt database (V7DBLSLT) to the mixed solvent electrolyte (MSE) framework. Only a few data sets remain to be worked up and routed.

Task 3 Disposition of Idaho HLW Calcine

Support of the CH2M-WG Idaho Calcine Disposition Project

The fifth pilot-scale run was designed to have about 10% less water in the batch and to mix for 30 minutes (as opposed to 20 minutes previously) in an attempt to increase the compressive strength.

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). Compressive strengths measured at 7 and 25 days (see below) are still lower than desired.

7-days
Sample 1A: 213psi.
Sample 2A: 180psi
Sample 3A: 238psi

25-days
Sample 1B: 318psi.
Sample 2B: 387psi
Sample 3B: 374psi

Task 4 Support of SRS Salt Disposition and Other SRS Alternatives

Modeling and Experimental Support for High-level SRS Waste Disposition

Analysis of the solubility studies of Cs and of K at 50^{o}C was completed. This data will be ported into the MSE database in 2008. Calculations on the leaching of Batch 5 sludge continue. Blending studies of the predicted leachate with predicted DDA fractions from tank 41H and with portions of the DWPF recycle stream are in progress. Of particular relevance is the re-precipitation of solids following mixing. Initial results indicated minimal (<5% by weight) solids re-precipitation provided that the temperature is held at the 40^{o}C value for the waste in 41H. Cooling the temperature to 30^{o}C resulted in significant solids formation when 200 kgal of the DWPF recycle stream was added. Blending calculations with smaller fractions of the DWPF recycle stream are in progress.

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

The problem of broadband spectrometer reported in last monthly report was identified with the helps of the experts at LLA Instruments GmbH. The wavelength shift and low signal strength found in the observed LIBS spectra is due to a small readjustment of the spectrometer and thermal influences. This type of problem can be fixed by reset an adequate Current Pixel Shift in both x and y direction to compensate such a mechanical shift. We have recorded the calibration spectra from Deuterium, Argon and Hg lamps to obtain the adequate Pixel Shift in x and y direction. After this correction, the LIBS spectra of pellet have reasonable signal strength for all analyte lines. We are resuming the work on collecting LIBS data form the new surrogate batch. This batch contains Cl and F, which are not present in the last evaluated batch.

We have collected some data of the new batch under different laser energy, detection window, and lens-to-sample distance. The reported limits of detection for Cl, F are relatively poor with LIBS as compared to other elements. We have found we need a laser energy above 75mJ/pulse to excite Cl and F lines while a laser energy of 5-10 mJ/pulse is sufficient to excite the rest of elements. Further work to improve the sensitivity of Cl and F detection is needed.

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

A draft report was issued describing a new series of crucible-scale melts 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.

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.

The next set of experiments will examine the thermal behavior of the materials using Differential Scanning Calorimetry (DSC) and Thermogravimetric and Differential Thermal Analysis (TGA/DTA).

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

The port assembly, adaptor, installation/operation instructions, and system schematics were sent off from ICET to SRNL. Initially, this set of equipment shall provide clear optical access for the optical instrument during the CEF operation. Feedbacks from SRNL are expected after CEF test runs, which may provide parameters for equipment modification if needed. The goals are not only to provide clear optical access but also to have an optimized field of view inside CEF.

Task 5 DOE Headquarters Support

DOE HQ Road Map

Workshop on Heavy Metal Phytoremediation

HEPA and Regenerable Filter Performance Assurance

During August, three sets of tests were completed. A set of tests was completed to see how much potassium chloride (KCl) could be loaded onto the filter over a five day period with no air backpulse until after the fifth day of loading. The second set of tests involved loading the filter in 0.2 inches water column increments. The third set of tests involved loading the filter in 0.5 inches water column increments.

During the first test, it was determined that a total of 39.1 grams of KCl aerosol was loaded onto the filter. The initial pressure drop across the filter was 9.97 inches water column at 14 standard cubic feet per minute (scfm). The final pressure drop across the filter was 24.7 inches water column at 14 scfm. The filter was air backpulsed at 100 psi a total of six times. The pressure drop across the filter after the backpulse was 11.3 inches water column.

The second set of tests completed was loading the filter in 0.2 inches water column increments. The filter was air backpulsed after each 0.2 inches water column of loading. The filter was loaded for a total of 10 cycles. The initial pressure drop across the filter was 10.8 inches water column at 14 scfm. The final pressure drop after 10 cycles was 12.4 inches water column. A total of 0.82 grams of KCl was loaded on the filter.

The third set of tests completed was loading the filter in 0.5 inches water column increments. The filter was air backpulsed after each 0.5 inches water column of loading. The filter was loaded for a total of 10 cycles. The initial pressure drop across the filter was 10.1 inches water column at 14 scfm. The final pressure drop after 10 cycles was 12.55 inches water column. A total of 4.12 grams of KCl was loaded on the filter.

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

In this month, the new experiment on the effect of sulfide concentrations on solubility of HgS in soils was planned. At the same time, we continued to revise the manuscript on “Bioavailability of HgS in Oak Ridge soil”. David L Monts presented our study in the 11th International Conference on Environmental Remediation and Radioactive Waste Manage, 2007, Belgium.

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

During the month of August, we have finished the phytoremediation experiment with our homemade chamber. Boston fern (*N. exaltata*) was grown on clean soil, but the plant shoot was enclosed into a chamber that containing mercury contaminated soil (with no direct contact to the plant). The experiment was designed to investigate the possible leaf uptake of mercury vapor from atmosphere. Plant tissues are harvested for chemical analysis. We expect to process the samples and data in coming months.

Task 6 Technology Development

Development of New Technologies for DOE Site Applications

The UV pulsed wavemeter was sent back for testing and/ or repairs. Upon inspection of the unit, the vendor claimed that the wavemeter could not be further repaired due to the aging of several key optical components. The wavemeter will most likely be shipped back without any action. In order to proceed with this project during the absence of the wavemeter, the experimental system is going to utilize an optical parametric oscillator laser (OPO). This broadly tunable laser system, recently obtained by the Institute for Clean Energy Technology, is capable of high-resolution scans. The OPO laser has been configured to efficiently scan the wavelength region associated with the 254 nm peak of mercury, as well as the forbidden transitions in Hertzberg band I $A^3\Sigma_u^+ \leftarrow X^3\Sigma_g^-$ of O_2. Preliminary background scans clearly display the O_2 transitions as well as the baseline readings for the mercury measurements.

A portable ringdown system has been successfully packaged and the initial tests have been conducted. The system is designed to operate in to NIR spectral regions for detections of multiple compounds. The system can be remotely controlled through a wireless network and requires a minimum maintenance once the system is tuned. This system will be extensively tested with several selected site-concerned VOCs or gases in the following months.

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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