

Monthly Research Progress

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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 November by the unexpected departure of the graduate student working on this effort. The retroactive rescission of CA07 funds in May have prevented replacement of the graduate student. Lack of manpower significantly limits progress on this and other efforts. The literature review of recent stereovision research has continued.

Fourier Transform Profilometry. In order to explore the feasibility of utilizing a higher-resolution camera for FTP volume determinations in tanks, ICET purchased a Cannon G9 digital camera and began testing of its flexibility and repeatability. The Hanford collaborators are checking with Hanford instrumentation specialists on feasibility of utilizing G9 in Hanford tanks. ICET is nearing completion of its efforts to incorporate corrections that address how FTP handles curved (non-perpendicular) background surfaces (such as a curved waste tank bottom). A 2008 Waste Management Symposium conference paper on FTP evaluator dependence and preliminary radiation testing results for FTP cameras and optics was prepared and submitted to the Hanford collaborators for comments/suggestions. Efforts on procedures for how to best propagate height across stitched image boundaries are ongoing.

Process Chemistry and Operations Planning for Hanford Waste Alternatives

Additional data packets were transferred to OLI Systems Inc. for incorporation in the MSE framework. Most all of the regressions have been completed at OLI Systems Inc. and the information from the ICET V7DBLSLT double salt database will be in the next build of the ESP software scheduled for release in January.

Efforts continued on developing the flowsheet model for C-108 retrievals. A number of questions arose in linking the necessary unit operations and these were sent to Randy Kirkbride at CH2M Hill Hanford Group. A response was received and further model generation is in progress. Changes in the approach for automating the ESP software to permit the development of large data sets for neural network generation are in progress.

Task 3 Disposition of Idaho HLW Calcine

Support of the CH2M-WG Idaho Calcine Disposition Project

Because the proper strength from the 34% formulation could not be reached, a formulation with the lower waste loading was run. Below is the recipe.

20% Waste Loading		
Fly ash	37.7	pounds
Slag	37.7	pounds
Portland Cement	37.7	pounds
Simulant	56.7	pounds
Water	102.0	pounds
Sodium Sulfide	3.3	pounds
Total	275.0	pounds

Nine samples were picked and are expected to be tested for compressive strength this month, after they are cured enough for testing (possibly early December).

Discussions were held with Alan Herbst regarding wrapping up the project which is scheduled to terminate in December. Final entries will be made in appropriate notebooks, chain of custody forms checked for adequacy, and materials will be properly disposed of. A final report, basically an update of our mid-year report issued in July, will also be issued.

Task 4 Support of SRS Salt Disposition and Other SRS Alternatives

Modeling and Experimental Support for High-level SRS Waste Disposition

Further mixing calculations with the aluminum-rich leachate from processing of sludge batch 5 with DDA fractions from tank 41H and a fraction of the DWPF recycle stream were started. Direct mixing of the sludge batch 5 leachate with the salt solution did not present any modeling difficulties. Further attempts to mix the silicon-rich DWPF recycle stream with the resulting compositions created model conversion errors when using the zeolite database. This compilation was originally developed at SRS/SRNL in 2002 for use in ESP version 6.5 and has previously predicted the behavior of sodium aluminosilicate systems examined in ICET laboratories. The current ESP version (7.0) contains updated thermodynamic information in the default Public database. The problems in using the zeolite database have been traced to changes in the representation of the aluminum chemistry in going from version 6.5 to 7.0. One option under consideration is to re-fit the zeolite database in the MSE framework during FY'2008.

A conference call was held with workers at SRS and at Parsons regarding the re-precipitation of solids in the simulant used for testing the CSSX process. Initial calculations have indicated that the solution is saturated in aluminum and oxalate and fluoride or phosphate. It is also anticipated that there is excess silicon in the simulant and use of the corrosion database predicted the formation of natrolite ($\text{Na}_2\text{Al}_2\text{Si}_3\text{O}_{10}\cdot 2\text{H}_2\text{O}$). Chemicals needed to make the simulant have been ordered and development of a test plan is underway.

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

Previous data show that it requires a laser energy above 60 mJ/pulse to detect Cl and F lines from the simulated batch powder while other elements of interest can be detected with laser energy of 5-10 mJ/pulse. The use of high laser energy (>25 mJ/pulse) for pellet measurement is not desirable because it might cause some resonance lines to saturate the detector or have self-absorption problem. Multivariate data analysis techniques are being investigated for extracting hidden Cl and F concentration information from LIBS spectra obtained from low laser pulse energy.

Since a large number of samples is required for multivariate analysis, 8 different composition pellet samples have been prepared from the prepared batch in the test. LIBS data of these samples were taken and the analysis results show that CeO₂ can be accurately measured. However, these samples have collinear constituent concentrations in the batch powder, which is not desirable for elements in the batch. Some more samples need to be prepared without using the batch powder to avoid collinear constituent concentrations in the samples. A compact and rugged broadband spectrometer from Andor Technology was ordered. This spectrometer will be used to design a dual-pulse broadband LIBS system which will be used in the demonstrated test at SRNL.

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

Experiments examining the thermal behavior of the various frit/waste simulant combinations using Differential Scanning Calorimetry (DSC) and Thermogravimetric and Differential Thermal Analysis (TGA/DTA) have been inconclusive and have been discontinued.

Discussions were held with Kevin Fox concerning the end of this work which terminates in December. A report providing the various thermal analysis results and a wrap up of the work done over CY2007 will be issued as a final report next month.

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

Awaiting feedback from SRNL on the performance of delivered port adaptor and port window assembly during the CEF test runs.

Task 5 DOE Headquarters Support

DOE HQ Road Map

No reported activities.

Workshop on Heavy Metal Phytoremediation

This workshop has been rescheduled tentatively for early summer 2008, we will report progress next year.

HEPA and Regenerable Filter Performance Assurance

In November preparations for the autopsy study of filters loaded with KCl during the ICET media velocity study were continued. A cyanoacrylate (super glue) fuming chamber was developed and tested. Pieces of loaded filter media are placed in the chamber and exposed to cyanoacrylate vapors, thus resulting HEPA media suitable for SEM analysis. SEM analysis will allow the determination of filter cake thickness and

possibly the relative degree of aerosol surface vs. depth loading. Over the course of the month, parameters such as the necessary mass of cyanoacrylate and media residence time needed to fully preserve the media were determined. Media velocity dependent loading curves (derived from KCl loading of Flanders and Camfil Farr filters) were fit using a third order polynomial expression.

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

During the month, findings entitled “effects of iron/manganese oxides on stability of mercury sulfide in U.S. Department of Energy’s Oak Ridge Site” were presented at the International Conference of Soil Science Society of America, New Orleans. The presentation was well received by an international audience.

The findings will be submitted as a manuscript to a refereed journal.

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

During the month of November, the phytoremediation experiment started in October was concluded. Plant tissues from the two fern species (Boston fern and Chinese brake fern) were collected together with soil samples. Mercury concentration and speciation will be analyzed in the following months.

Task 6 Technology Development

Development of New Technologies for DOE Site Applications

The OPO laser has become dysfunctional at the current time. General maintenance has been conducted on the other laser systems. In the meantime, efforts have focused on obtaining information about other wavemeters which can efficiently function in the deep UV. To date, the majority of wavemeters on the market cannot operate in this wavelength region. The unrepaired wavemeter was returned from Burleigh. While searching for technical support for this system, a former employee of Burleigh presented a couple diagnostic tests, which will be implemented to ensure whether specific components are functional or not. Burleigh does not have the capability to test wavemeters in the deep UV, so these diagnostics would be easier to test once the system is returned.

Chemical properties of VOCs are different from that of atmospheric molecules such as methane and carbon dioxide; a system that works for small atmospheric molecules without an issue of mirror contamination may not work well for VOCs. For instance, small atmospheric molecules in a ringdown cavity can be easily purged and pumped out; and ringdown mirrors can operate for a long period of time, e.g., a few months, without noticeable degradation of the mirror reflectivity. However, published research results show that some of VOCs can readily contaminate the ringdown mirrors after

intensive use in a short period of time, e.g., a few weeks. Therefore, efforts in this month were focused on the evaluation of the cavity contamination. Isoprene is a typical VOC and has C-H overtone absorption in the near-infrared spectral region. A gas mixture of isoprene in nitrogen with a mix ratio of 5 ppm was procured. A laboratory cw-ringdown system has been used to test the extent of the mirror contamination. The test is being conducted. The result will help address the general issue of the chemical contamination of ringdown mirrors in the development of the tank vapor characterization system.

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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