April 2007

# Monthly Research Progress

**Institute for Clean Energy Technology**
**(formerly Diagnostic Instrumentation & Analysis Laboratory)**
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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*

The month's work was directed towards the goal of getting the Stage II image "stitching" experiments underway and further development of the capabilities of the FTP image simulator.

As mentioned during the March report, a "pan" and "tilt" apparatus had been constructed with the goal of achieving angle measurements on the order of 0.02 degrees. During April, the apparatus was utilized for obtaining multiple adjacent images where only the "pan" angle was varied. These images were subsequently analyzed and it has been demonstrated that the reconstruction of these images can be achieved with sufficient accuracy to meet our data quality objective. The manipulation of the "tilt" angle did not proceed at this point due to an unanticipated coupling of the

"pan" and "tilt" adjustment means. A correction procedure was developed to enable us to calculate the adjustment settings necessary to manipulate the "pan" and "tilt" at any desired combination. Further testing of the "tilt" manipulation is necessary to verify its precision.

We are in the process of training multiple members of our research team in the use of the FTP image simulator. Previously, only the author/developer of the simulator was adequately knowledgeable in its use and simulations of the FTP system had to be planned and scheduled in advance. This caused delays in the turnaround time between the initial identification of potential problems with the FTP procedure and the analysis/presentation of simulator results.

*Process Chemistry and Operations Planning for Hanford Waste Alternatives*

Pure gibbsite, as confirmed by XRD analysis, was obtained from Alcoa and is now being used to extend the experiments on the gibbsite to boehmite transition to lower temperatures and higher ionic strengths. Difficulties were encountered with the TGA. Instability in the control of the temperature was observed. Evaluation of the system indicated a new furnace was required.

Efforts continue on documenting previous efforts associated with development of the neural network and on publishing data associated with earlier solubility measurements. Further discussions with OLI systems Inc. will proceed in May following the potential re-alignment of workscopes.

## Task 3 Disposition of Idaho HLW Calcine

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

A second run was completed this month with the main difference being that the water was already in the mixer as the powders are added. In addition, the one-inch bottom drain was replaced with a two-inch drain.

The resulting product was much improved over the previous test. Three 2-inch cubes were taken as samples. One of the cubes was tested for compression strength after 7 days and found to be less than 150psi. The other two cubes will be tested after 28 days.

Another run will be scheduled for early May. It is expected that the size of the batch will be increased and that the mixing process will be altered so that as the materials are being added, the mixing will be continuous. We are also looking at extending the mixing blades so that they reach closer to the bottom of the mixing tank.

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

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

The $KNO_3$ / $NaNO_3$ and $CsNO_3$ / $NaNO_3$ systems in water, 1m and 3m NaOH solutions at 50°C are equilibrating. These systems appear to have longer equilibration times before solids form. Work continues on compilation of a topical report for modeling of tall column experiments performed at FIU. Also included in the report will be several Hanford simulant experiments at saturated and unsaturated conditions.

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

Work on testing different fiber optic configurations for delivering the laser pulse to create plasma spark on the sample surface continued. A hollow-core fiber from Polymicro Technologies, LLCA for transmitting high pulse energy laser was tested. Since the transmission of this fiber is optimized for CO2 laser, we only obtained 50% transmission efficiency for 532-nm laser beam. We have requested another hollow fiber (HWEA 7501200) from Polymicro Technologies, LLCA that has optimized transmission for Er:YAG laser. This fiber will be tested for its transmission efficiency for 532-nm laser and long-term operation. To design a die that can be mounted on a rotating plate during LIBS measurement, we have redesigned the die that is used to press plutonium oxide surrogate. We are in the process of testing the different size of the die suitable for the LIBS experiment in glove box at SRNL.

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

A series of crucible-scale melts has been started examining the behavior of mixtures of materials for Sludge Batch 4. These materials include:

Frit 418
Frit 503
SB4 SRAT Product
SB4 SME Product + Frit 418
SB4 SME Product + Frit 503

The SME Product mixtures have been made up with a waste loading of 35%. The materials were dried in an oven at 110C then 20g-sized melts were heated for two hours at 700C, 750C, 800C, 850C, and 900C. The object was to compare the behavior of the two frit compositions.

Some observations:

1. Both frits raise to about the same height in the crucible during the melting process.
2. Frit 503 has fewer/bigger bubbles than Frit 418 at 750C.
3. Frit 503 seems to have consolidated more than Frit 418 at 800C.
4. Frit 418 has more bubbles than Frit 503 all the way up to 900C.
5. Frit 503 has almost no bubbles at 900C.

The presence of bigger bubbles and more rapid consolidation seems to indicate that Frit 503 would be less likely to foam. That could translate into a faster melting rate.

Subsequently, mixtures of the SRAT Product and each frit have been made up with waste loadings of 40% and 45%. Results of these experiments will be reported next month.

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

The fabrication of a mock-up of the Cold Cap Evaluation Furnace (CEF) has been started. The initial thrust is to redesign the top with appropriate window locations in order to provide access for diagnostic instrumentation. A proper field of view for the stereovision cameras is most critical.

Discussions have been started on a task involving using ICET diagnostic instrumentation to assist with melting studies using the Cold Cap Evaluation Furnace (CEF) that has been built at SRNL. A conference call was held to discuss the pros and cons of various methods of monitoring methods, concentrating on the problem of minimizing the fouling of windows by gases generated within the furnace.

This task will be a large part of our effort in the coming year.

## Task 5 DOE Headquarters Support

*DOE HQ Road Map*

*Workshop on Heavy Metal Phytoremediation*

*HEPA and Regenerable Filter Performance Assurance*

Work completed in April 2007 included 7 load and backpulse cycles utilizing Large CeraMem Filter #1. The filter was weighed before loading, after loading, and after backpulse. This will provide information relating the filter differential pressure drop ($\Delta P$) increase to the mass captured by the filter.

The ΔP of Filter #3 was noted prior to a loading cycle, and then the filter was washed with water and dried until the original ΔP was recovered. This took 3 cycles of washing and drying. It was determined that the filters must be dried overnight to completely drive off all the water from washing. This procedure established the procedure in which a salt laden filter can be restored to a "like new" condition.

A series of tests that will determine the optimum backpulse pressure needed to regenerate, i.e., clean, the Large CeraMem Filters was initiated. This testing will be done on a set of 3 filters. Each filter will be loaded and backpulsed at 25 psi, 50 psi, 75 psi, and 100 psi. Each filter will be loaded and backpulsed 4 times. This testing will continue into the next month as well as additional testing on the large CeraMem filters.

The manuscript "Load-Wash Testing of Regenerable Sintered Metal and Ceramic Membrane Filter Media" was accepted for publication in *Environmental Engineering Science.*

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

During this month, we continued experiments on the effects of naturally occurred minerals on mercury release from Oak Ridge soil contaminated with HgS. $Fe_3O_4$, $Fe_2O_3$, and $MnCO_3$. The preliminary results showed that iron oxides triggered the release of Hg, while $MnCO_3$ did not affect the release of Hg from HgS minerals in Oak Ridge soils. The concentration of Hg released from HgS mineral in contaminated soils was linearly correlated to that of dissolved Fe in extracts.

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

Status Assessment and Forecast (present analysis of program/project status, proposed solutions to problems, and future expectations regarding the project).

The experiment is expected to be continued in the coming two months. More variables of the reactions and pathways such as release/exchange of sulfur, iron, and pH as well as various Mn oxide minerals will be examined.

*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 this month, we finished a phytoremediation experiment using our homemade chamber. The experiment was started in February. Chinese brake fern was grown on clean soil, but the plant shoot was enclosed into a chamber that containing mercury contaminated soil (from different sources such as $HgCl_2$, and HgS). Our objective was to investigate the possible leaf uptake of mercury vapor from atmosphere above the contaminated soil. Plant samples were collected and will be analyzed in following months.

## Task 6 Technology Development

*Development of New Technologies for DOE Site Applications*

Uranium detection with an ICP-CRDS system using a blue diode laser was explored. Further characterization of the triggering mechanism as well as the corresponding ringdown stabilities were examined. Various laser parameters, such as current, temperature, and trigger level, were monitored and optimized for CRDS experiments.

Isotopes are powerful spectral identifications. Many tank wastes and contaminant sources can be characterized through the measurement of trace isotopes, including isotopes of elements, such as mercury and uranium, etc., as well as isotopes of the elements contained in compounds. A standard $^{12}CO_2$ and $^{13}CO_2$ mixture with C-13 isotope abundance of +9.8 ‰ was tested in the ringdown system. The results show that the system can measure C-13 in $CO_2$ gas that has a concentration range of 0.1 – 10%.

*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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