QUARTERLY PROGRESS REPORT

October 1 to December 31, 2015

Florida International University's Continued Research Support for the Department of Energy's Office of Environmental Management

Principal Investigator:

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Prepared for:

U.S. Department of Energy
Office of Environmental Management
Under Cooperative Agreement No. DE-EM0000598

FIU | Applied Research Center
FLORIDA INTERNATIONAL UNIVERSITY

Introduction

The Applied Research Center (ARC) at Florida International University (FIU) executed work on four major projects that represent FIU-ARC's continued support to the Department of Energy's Office of Environmental Management (DOE-EM). The projects are important to EM's mission of accelerated risk reduction and cleanup of the environmental legacy of the nation's nuclear weapons program. The information in this document provides a summary of the FIU-ARC's activities under the DOE Cooperative Agreement (Contract # DE-EM0000598) for the period of October 1 to December 31, 2015.

The period of performance for FIU Year 6 under the Cooperative Agreement will be August 29, 2015 to August 28, 2016. The projects have been reorganized for FIU Year 6. Projects 2 and 3 from FIU Year 5 have been combined into a single project (Project 2) focused on soil and groundwater remediation research. The D&D and Workforce Development projects were subsequently renumbered as Projects 3 (D&D) and 4 (Workforce Development).

Highlights during this reporting period include:

Program-wide:

- FIU submitted four (4) Project Technical Plans (PTPs) detailing the scope of work for FIU Year 6 (August 2015 – August 2016) to DOE on October 15, 2015.

Project 1- Chemical Process Alternatives for Radioactive Waste:

- Milestone 2015-P1-M19.2.1, the test loop set up for the evaluation of nonmetallic components in the waste transfer system, was completed on November 20, 2015. In addition, draft papers based on project research were submitted to Waste Management 2016.

- Milestone 2015-P1-M18.3.1, "Completion of a test plan for temperature measurements using IR sensors," was completed on December 18, 2015. The milestone 2015-P1-M18.1.1 and the corresponding test plan deliverable, also due on December 18, has been delayed. Additional time is needed to confer with the client and determine the extent of testing needed.

Project 2- Environmental Remediation Science & Technology:

- Draft papers based on project research were submitted to Waste Management 2016 (milestone 2015-P2-M1).
- The Task 4 deliverable, "Draft sustainable remediation report for the M1 air stripper," was submitted by its due date 12/18/2015. The Task 3 Milestone (2015-P2-M2) "Complete refinement of MIKE SHE model configuration parameters for the simulation of overland flow using revised model domain (Subtask 3.1)," was also completed by its due date 12/30/2015.

Project 3 – Waste and D&D Engineering & Technology Development:

- Draft papers based on project research were submitted to Waste Management 2016 (milestones 2015-P3-M1.1 and 2015-P3-M3.1). The first D&D KM-IT Workshop to DOE EM staff at HQ is being reforecast based on the schedule and availability of DOE EM staff.
- Milestone 2015-P3-M2.1 was met with the completion of the Phase 1 testing of fixatives for the incombustible fixatives task.

Project 4- DOE-FIU Science & Technology Workforce Development Initiative: (previously known from FIU Year 5 as Project 5)

- Development, review, and site approval of the DOE Fellow summer 2015 internship reports were completed. The reports are available and have been posted in the DOE Fellows website http://fellows.fiu.edu under the "DOE Fellows Internship Reports" tab.
- The DOE Fellows Class of 2015 was also identified and recruited, and a list was sent to DOE.
- The DOE Fellow Induction Ceremony (milestone 2015-P4-M3) to welcome the DOE Fellows Class of 2015 was held on November 5, 2015.

The program-wide milestones and deliverables that apply to all projects (Projects 1 through 4) for FIU Year 6 are shown on the following table:

Task	**Milestone/ Deliverable**	**Description**	**Due Date**	**Status**	**OSTI**
Program-wide (All Projects)	Deliverable	Draft Project Technical Plan	10/16/15	Complete	
	Deliverable	Monthly Progress Reports	Monthly	On Target	
	Deliverable	Quarterly Progress Reports	Quarterly	On Target	
	Deliverable	Draft Year End Report	10/14/16	On Target	OSTI
	Deliverable	Presentation overview to DOE HQ/Site POCs of the project progress and accomplishments (Mid-Year Review)	02/29/16*	On Target	
	Deliverable	Presentation overview to DOE HQ/Site POCs of the project progress and accomplishments (Year End Review)	08/31/16*	On Target	

**Completion of this deliverable depends on availability of DOE-HQ official(s).*

Project 1
Chemical Process Alternatives for Radioactive Waste

Project Manager: Dr. Dwayne McDaniel

Project Description

Florida International University has been conducting research on several promising alternative processes and technologies that can be applied to address several technology gaps in the current high-level waste processing retrieval and conditioning strategy. The implementation of advanced technologies to address challenges faced with baseline methods is of great interest to the Hanford Site and can be applied to other sites with similar challenges, such as the Savannah River Site. Specifically, FIU has been involved in: analysis and development of alternative pipeline unplugging technologies to address potential plugging events; modeling and analysis of multiphase flows pertaining to waste feed mixing processes, evaluation of alternative HLW instrumentation for in-tank applications and the development of technologies to assist in the inspection of tank bottoms at Hanford. The use of field or *in situ* technologies, as well as advanced computational methods, can improve several facets of the retrieval and transport processes of HLW. FIU has worked with site personnel to identify technology and process improvement needs that can benefit from FIU's core expertise in HLW. The following tasks are included in FIU Year 6:

Task No	Task
Task 17: Advanced Topics for Mixing Processes	
Subtask 17.1	Computational Fluid Dynamics Modeling of HLW Processes in Waste Tanks
Task 18: Technology Development and Instrumentation Evaluation	
Subtask 18.1	Evaluation of FIU's Solid-Liquid Interface Monitor for Estimating the Onset of Deep Sludge Gas Release Events
Subtask 18.2	Development of Inspection Tools for DST Primary Tanks
Subtask 18.3	Investigation Using an Infrared Temperature Sensor to Determine the Inside Wall Temperature of DSTs
Task 19: Pipeline Integrity and Analysis	
Subtask 19.1	Pipeline Corrosion and Erosion Evaluation
Subtask 19.2	Evaluation of Nonmetalic Components in the Waste Transfer System

Task 17: Advanced Topics for HLW Mixing and Processing

<u>Task 17 Overview</u>

The objective of this task is to investigate advanced topics in HLW processing that could significantly improve nuclear waste handling activities in the coming years. These topics have been identified by the Hanford Site technology development group, or by national labs and academia, as future methods to simulate and/or process waste streams. The task will focus on long-term, high-yield/high-risk technologies and computer codes that show promise in improving the HLW processing mission at the Hanford Site.

More specifically, this task will use the knowledge acquired at FIU on multiphase flow modeling to build a CFD computer program in order to obtain simulations at the engineering-scale with appropriate physics captured for the analysis and optimization of PJM mixing performance. Focus will be given to turbulent fluid flow in nuclear waste tanks that exhibit non-Newtonian fluid characteristics. The results will provide the sites with mathematical modeling, validation, and testing of computer programs to support critical issues related to HLW retrieval and processing.

Task 17 Quarterly Progress – October 1 – December 31, 2015

Subtask 17.1: Computational Fluid Dynamics Modeling of HLW Processes in Waste Tanks

FIU drafted and submitted a paper for the Waste Management Symposia to be held on March 6-10, 2016 in Phoenix, AZ. The paper was accepted for presentation at the conference.

> **Abstract**: 16260 – Improving the Accuracy of Computational Fluid Dynamics Simulations of Nuclear Waste Mixing using Direct Numerical Simulations
> **Session**: 030C - Posters: Tank Waste
> **Date**: Monday, March 07, 2016
> **Time**: 1:30 - 5:00 PM

In this reporting period, 2D simulations of the non-Newtonian fluid for Re = 550, 1650, 10000, 15000, and 20000 were conducted. We used the H-B, alpha, shear rate correction methods for viscosity modeling and k-ε method for turbulence modeling and compared our results to experimental data of Escudier et al. (2005). The purpose of this analysis was to find a threshold Reynolds number beyond which the alpha-inverse overall method could be used instead of the alpha overall method.

Numerical Approach

FIU continued the simulation of the non-Newtonian fluid for two laminar flows, Re = 550 and 1650, for which experimental data is available. Additional simulation cases of turbulent flows were created by extracting data from a second order fit to the experimental data at Re = 10000, 15000, and 20000.. Figure 1-1 shows the predicting profile that passes through available data (square markers) and has a fitness quality of R^2 = 0.9999. The results of these interpolation operations are available in Table 1-1.

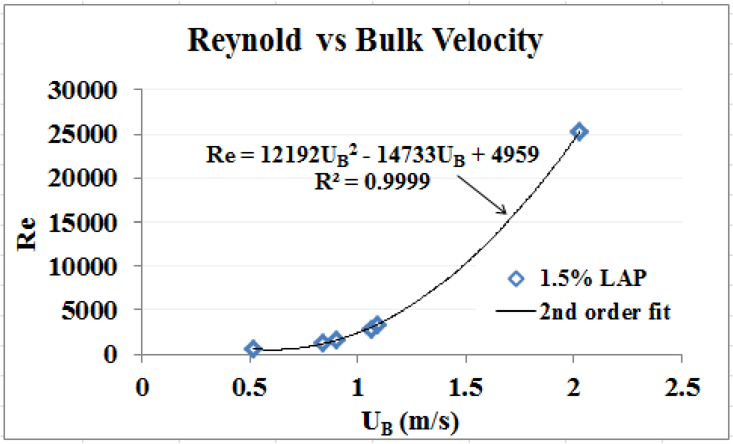


Figure 1-1. Extraction of data from a second order fit to the experimental data.

Table 1-1. Data Interpolation Between Experimental Values

Re	U_B (m/s)	$\dot{m}$ (kg/sec)	μ_w
10000	1.4866	11.7	0.0149
15000	1.6945	13.3	0.0113
20000	1.8686	14.7	0.0093

Simulation Results

The alpha method was accurate for Re = 550 approximately half way through the cross section. For Re = 1650, the alpha method is not accurate. For other turbulent cases, except for Re = 25300, the overall method was closer to the H-B method than the overall inverse method. No transitional Reynolds number could be detected based on these results.

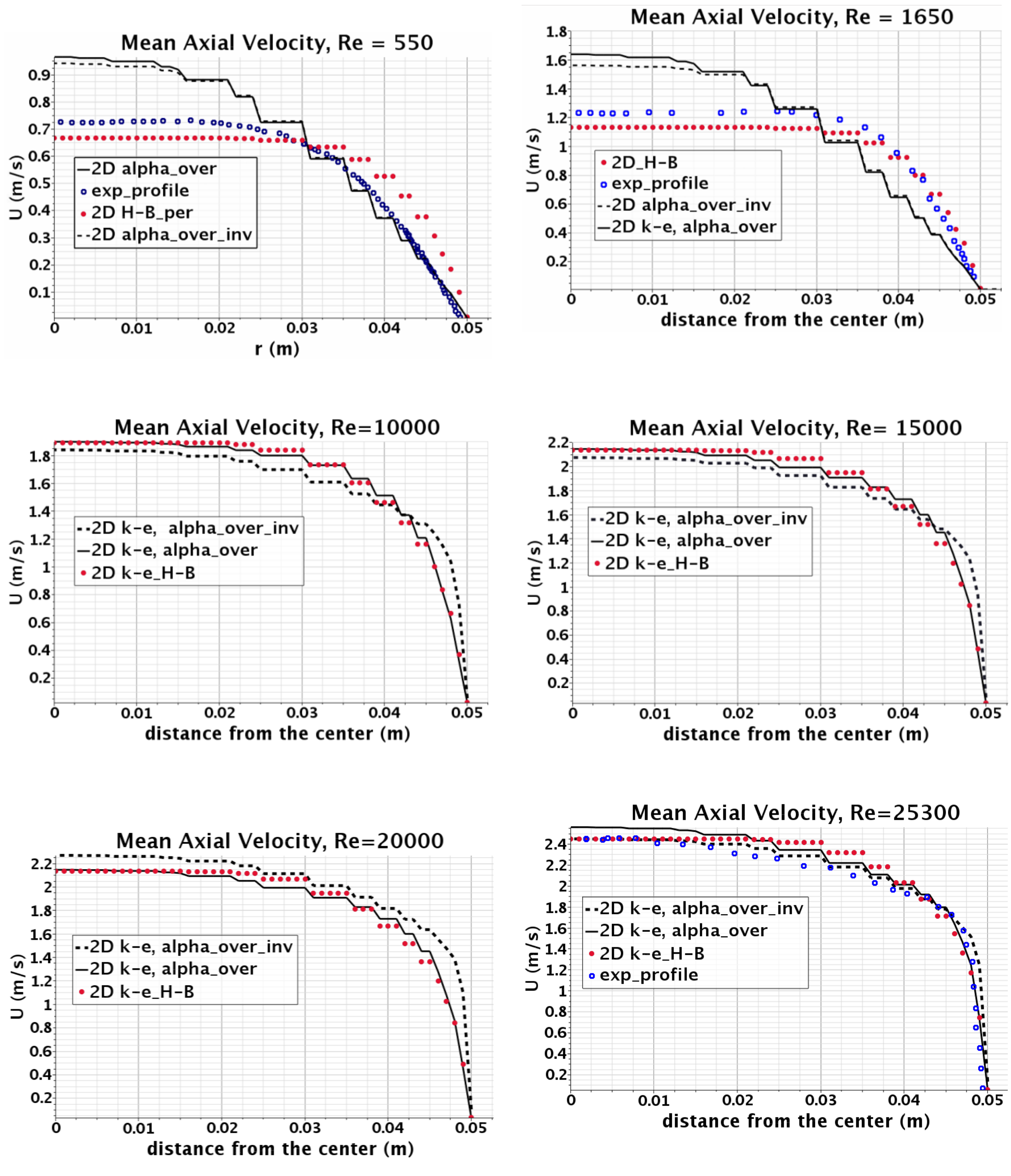


Figure 1-2. Simulation results for various Reynolds numbers.

FIU also conducted a set of 2D RANS simulations with the same non-Newtonian fluid for Re = 550 using both laminar and turbulent solvers in STARCCM+. The purpose of this investigation was to show that both solvers provide extremely similar results except for the proposed Inverse-alpha method. In this case, the results were significantly improved when compared to the experimental data. In addition, results of error calculations for the simulation cases considered thus far are presented.

As discussed above, the use of the alpha method in both inverse and direct schemes (Eqs. 1&2) could not improve the accuracy of the laminar flow with a Reynolds number of 550 (Figure 1-3). In these equations, we set the threshold for the dissipation rate to zero (i.e., ε-THS = 0) in order to maximize the sensitivity of the model.

$$\mu_{sub.} = \begin{cases} \mu_{H-B} = \dfrac{|\tau|}{\Upsilon} = \dfrac{\tau_Y}{\Upsilon} + k\,\Upsilon^{(n-1)} & \varepsilon_{H-B} \leq \varepsilon - THS \\ \alpha \times \mu_{psudo\_Newt.} & \varepsilon_{H-B} > \varepsilon - THS \end{cases} \qquad \text{Eq.(1)}$$

$$\mu_{sub.} = \begin{cases} \mu_{H-B} = \dfrac{|\tau|}{\Upsilon} = \dfrac{\tau_Y}{\Upsilon} + k\,\Upsilon^{(n-1)} & \varepsilon_{H-B} \leq \varepsilon - THS \\ \dfrac{\mu_{psudo\_Newt.}}{\alpha} & \varepsilon_{H-B} > \varepsilon - THS \end{cases} \qquad \text{Eq.(2)}$$

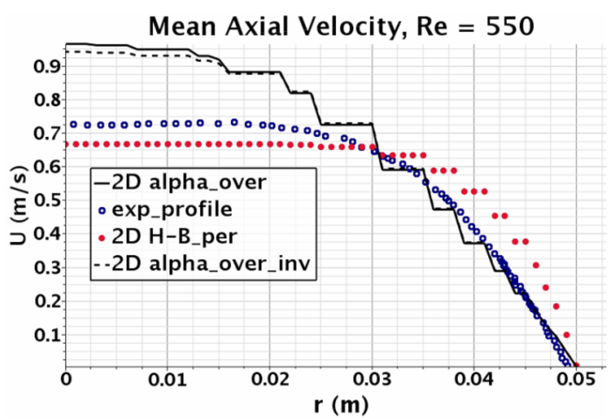


Figure 1-3. Velocity profile for the laminar case, Re = 550 (reported in monthly report for the period of 10-20 to 11-20, 2015).

Results:

During evaluation of the results, it was observed that if the flow was modified using the turbulent solver, almost identical results were obtained for all models (Figure 1-4 a-b), except for the Inverse-alpha method (Figure 1-4-c).

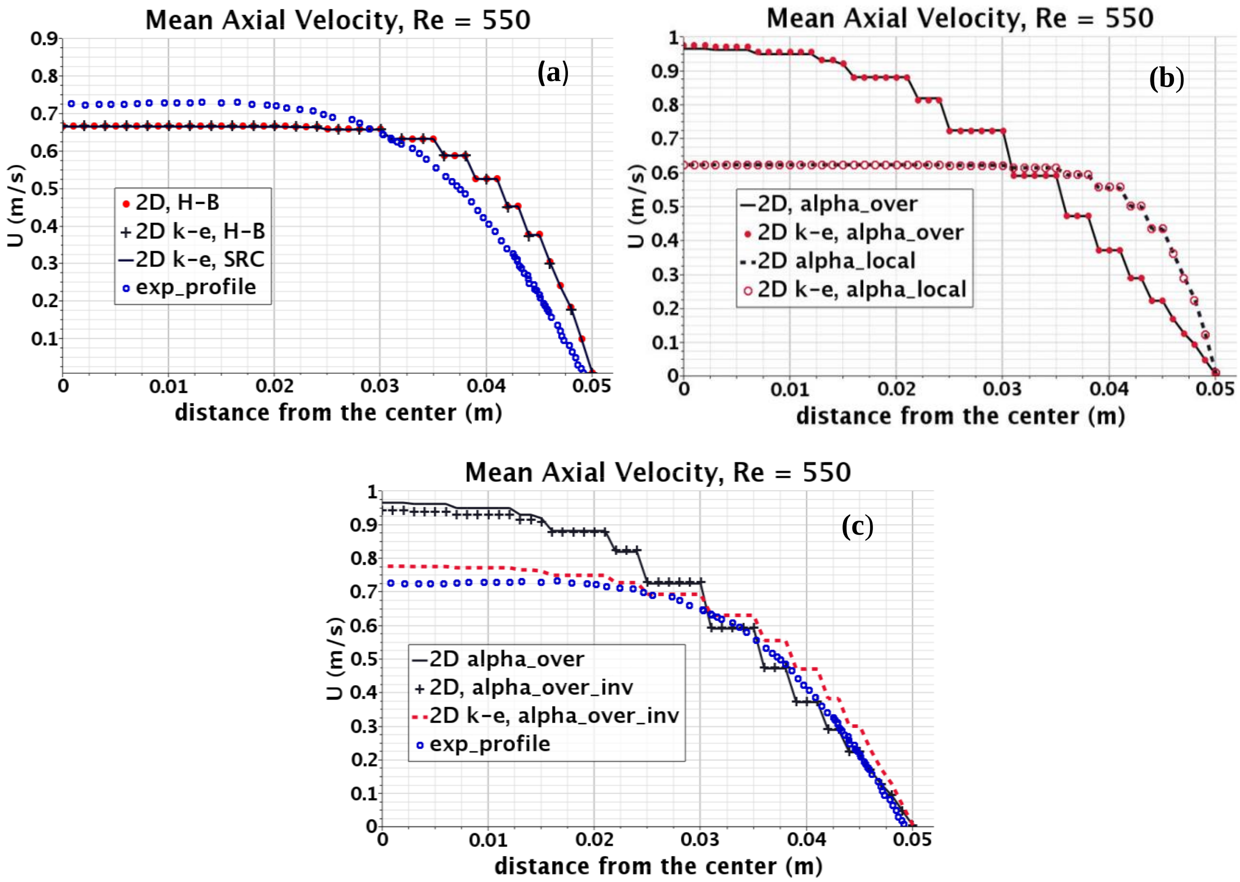


Figure 1-4. Comparison between velocity profiles obtained for the laminar case by laminar and turbulent solvers, Re = 550, (a) H-B and SRC methods (b) Direct α-methods, and (c) Inverse and Direct α-methods.

Error analysis:

The results of error analysis for different Reynolds numbers, i.e., percentage deviation of the velocity profiles from the experimental profiles, , are shown in the following figures. The best profile in each figure is shown with a bold line. The best profiles were selected based on the smallest values of the average relative error, as defined by Eq. 3. Table 1-2 shows the results of average relative error used for determination of the best methods.

$$Err_{avg.}\ (\%) = \frac{100}{N} \times \sum_{\lambda=1}^{N} (|\ f_{exp}(\lambda) - f_{sim}(\lambda)\ |)\ /\ f_{exp}(\lambda). \qquad \text{Eq.(3)}$$

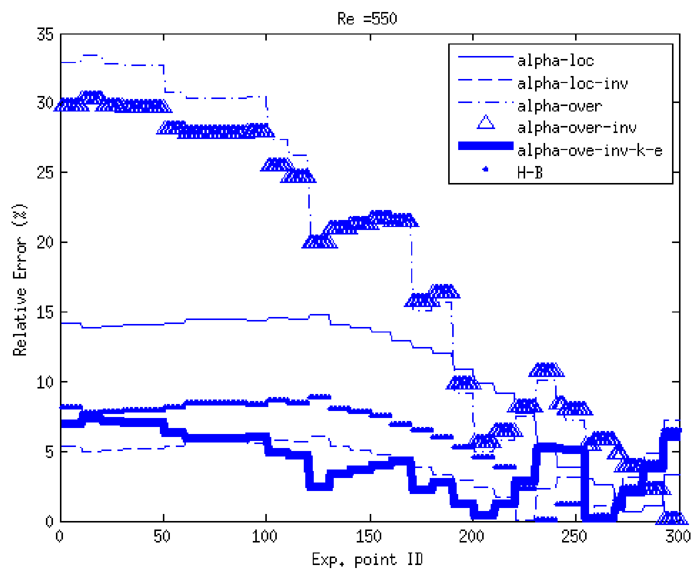


Figure 1-5. Comparison between error profiles obtained from comparing each experimental data point against numerical data point for different models, laminar flow with Re = 550.

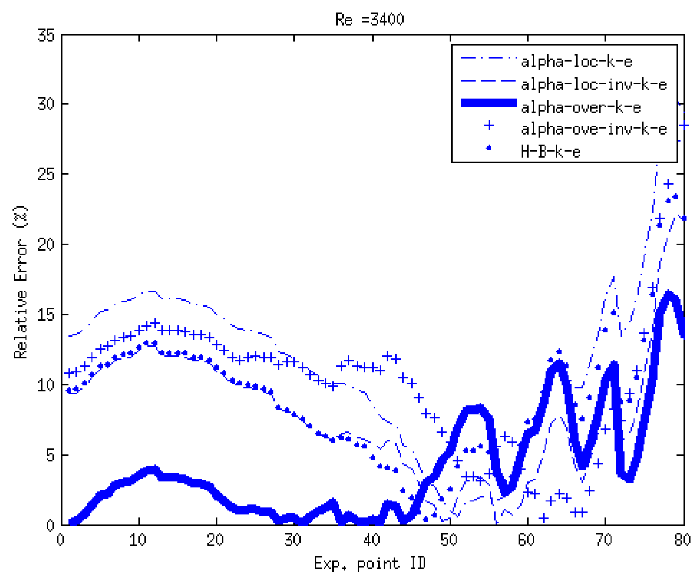


Figure 1-6. Comparison between error profiles obtained from comparing each experimental data point against numerical data point for different models, transitional flow with Re = 3400.

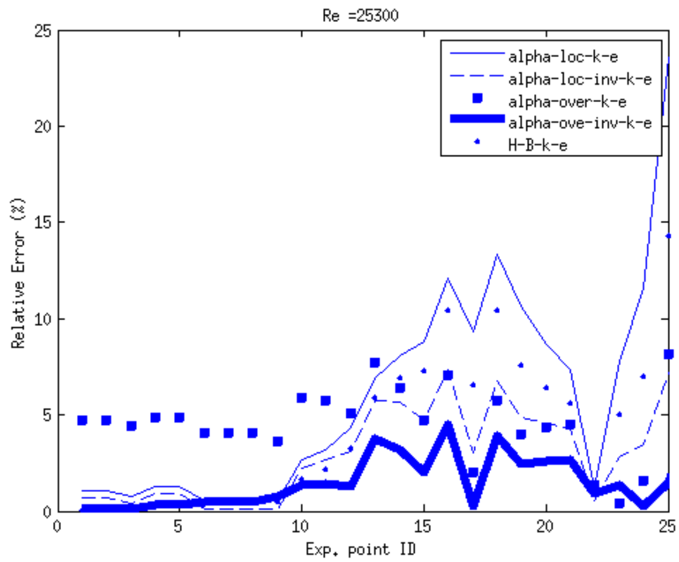


Figure 1-7. Comparison between error profiles obtained from comparing each experimental data point against numerical data point for different models, turbulent flow with Re = 25300.

Table 1-2. Average Relative Errors

Re = 550

model	error (%)
alpha_loc	27.5
alpha_loc_inv	15.6
alpha_ove	18.8
alpha_ove_inv	17.9
alpha_ove_inv	11.1
H_B	19.1

Re = 3400

model	error (%)
alpha_loc	36.8
alpha_loc_inv	38.4
alpha_ove	17.1
alpha_ove_inv	54.9
H_B	29.9

Re = 25300

model	error (%)
alpha_loc	5.8
alpha_loc_inv	2.9
alpha_ove	4.5
alpha_ove_inv	1.4
H_B	4.1

In addition, 3D periodic pipe flow simulations of the non-Newtonian fluid for Re = 25300 with different grid sizes were conducted. The H-B method (for viscosity modeling) and QDNS and RANS methods (for turbulence modeling) were used and the results were compared to the experimental data of Escudier et al., (2005). The purpose of this analysis was to compare the results of the QDNS and RANS methods.

Simulation Results

Figure 1-8 shows that similar results were obtained by the RANS method (k-ε model) when 250,000 and 750,000 cells were used. In the case of the QDNS, results with the 600,000-cell and 1.3m-cell grids were slightly different. The gird-independence analysis, sh shown in Figure 1-8, suggested that no better results could be obtained by further reduction of the grid size with both the RANS and QDNS methods. However, the RANS method performed better in comparison with the QDNS method in terms of agreement with the experimental data. To get a better understanding of the errors seen in the QDNS simulations, FIU will look at how reducing the time step size affects the results. It is anticipated that reducing the time step size will significantly improve the QDNS results.

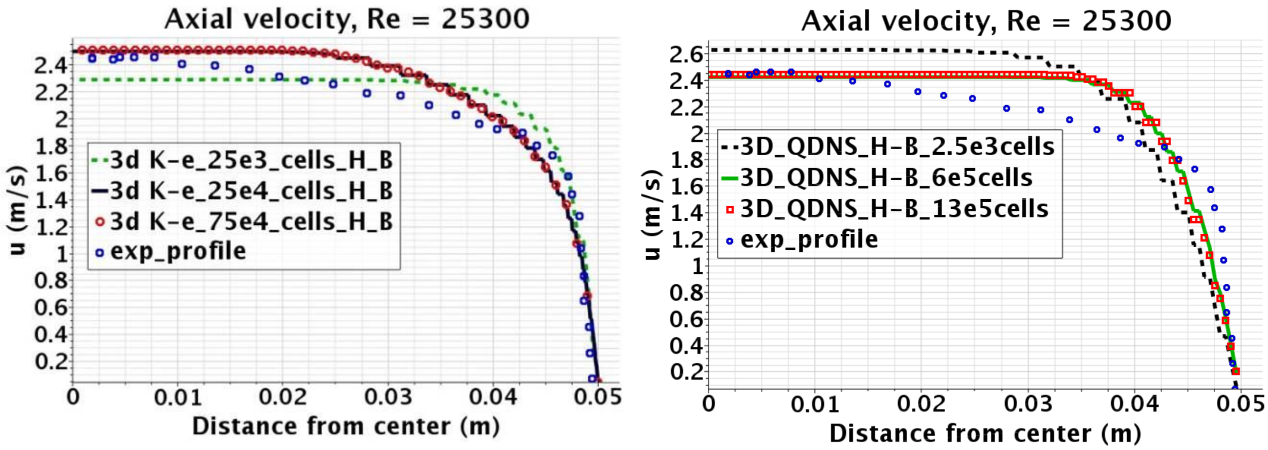


Figure 1-8. Velocity profiles obtained from different methods and grids. RANS method (left) and QDNS (right).

References:

Escudier M.P., Presti F., Pipe flow of a thixotropic liquid, J. Non-Newtonian Fluid Mech., volume 62, PP. 291-306, (1996)

Another effort for this task focuses on simulation validations of jet impingement correlations. During this reporting period the discrepancies observed in the original simulation of Poreh's experiment were investigated. In particular, velocity profiles along the radial wall jet seemed to "stick" to the wall. In other words, the velocity gradients from the wall to the maximum profile velocity were much higher than the experimental data. It is important to understand and correct for this discrepancy in order to confidently state that Poreh's experimental data was simulated successfully.

Investigation Approach

Different parameters of the simulation were altered in order to understand the variables that affect this issue. The mesh was refined extensively. The effects of the outlet pressure boundary condition on the domain were investigated by varying the distance of the boundary from the center of impingement. The Reynolds number was reduced in order to see if a low Reynolds number would lift the velocity profile from the wall. These different parameter studies did not lead to any promising results. The problem was addressed, however, by a change in the turbulent viscosity definition. It appears that the turbulent dissipation has a role in the high velocity gradients near the wall. This modification was accomplished by switching from the K-Epsilon Realizable model to the Standard K-Epsilon turbulence model.

Simulation Results

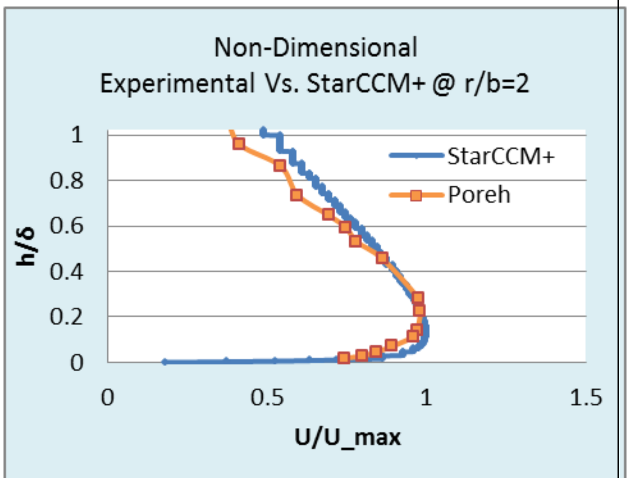
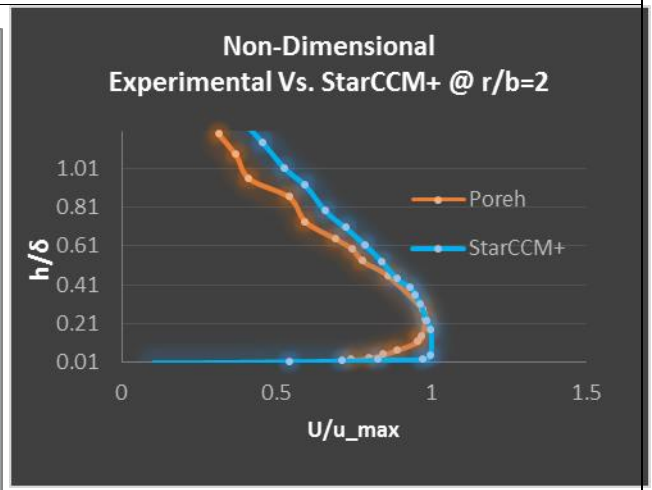
Standard K-epsilon	Realizable K-Epsilon
	

Figure 1-9. Standard vs. Realizable K-epsilon model velocity profiles.

It was observed that the standard K-epsilon model resulted in lower velocity gradients near the wall. These results imply that the turbulent dissipation in the Realizable model is not appropriate for this case. It might also be affected by other aspects of the standard k-Epsilon model. As a path forward, the different k-epsilon turbulence models will be compared in order to investigate why the Standard K-epsilon model produced better results than the Realizable model, contrary to what the literature would predict.

The different model coefficients in the standard K-Epsilon model that effect turbulent dissipation were also altered. In the standard K-Epsilon model, there are three empirical model constants, namely C_μ, $C_{\epsilon 1}$ and $C_{\epsilon 2}$. Because the ultimate goal is to simulate the entire PJM system using a K-Epsilon Realizable model, the intention is to use insights learned from this investigation in order to attain proper velocity profiles in the Realizable model. Three cases in which the different model coefficients were individually increased by 20% of its original value while holding the remaining constant were conducted.

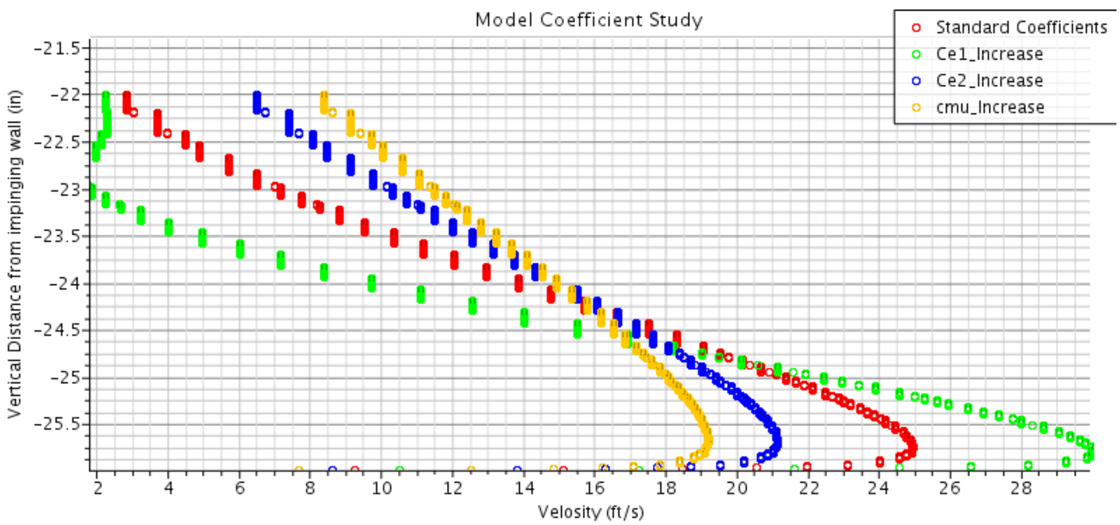


Figure 1-10. Standard K-Epsilon model coefficient study.

The results show that the model coefficients have a significant effect on both the velocity gradients near the wall and the maximum velocity value. Below is a summary of the effects of the parameters on the correlations of interest.

increase C_{1e} → decrease in δ increase in U_{max}
increase C_{2e} → increase in δ decrease in U_{max}
increase C_{μ} → increase in δ decrease in U_{max}

It is clear from this study that altering the model coefficients can lead to the proper near-wall velocity gradients and radial jet thickness predictions as dictated by Poreh's experimental data. The overall goal to use the Realizable K-Epsilon model will be achieved by appropriately altering its model constants. This brings forth an obstacle due to the fact that the C_μ is not a constant in the Realizable turbulence model as is in the standard K-Epsilon model. As a path forward, a literature review on how to alter turbulent dissipation in a K-Epsilon Realizable model will be conducted in order to gain available tools to reach the desired results.

Task 18: Technology Development and Instrumentation Evaluation

Task 18 Overview

The objective of this task is to assist site engineers in developing tools and evaluating existing technologies that can solve challenges associated with the high level waste tanks and transfer systems. Specifically, FIU is assisting in the evaluation of using a sonar (SLIM) developed at FIU for detecting residual waste in HLW tanks during pulse jet mixing (PJM). This effort would provide engineers with valuable information regarding the effectiveness of the mixing processes in the HLW tanks. Additionally, the Hanford Site has identified a need for developing inspection tools that provide feedback on the integrity of the primary tank bottom in DSTs. Recently, waste was found to be leaking from the bottom of the primary tank in AY-102. FIU will assist in the development of a technology to provide visual feedback of the tank bottom after traversing through the refractory pad underneath the primary tank.

Task 18 Quarterly Progress – October 1 – December 31, 2015

Subtask 18.1: Evaluation of FIU's SLIM for Estimating the Onset of Deep Sludge Gas Release Events

The objective of this task is to assist DOE site scientists and engineers in developing tools and evaluating existing technologies that can solve challenges associated with the high-level waste (HLW) tanks and transfer systems. Specifically, FIU is assisting in the evaluation of using a 3D profiling sonar as part of its Solid-Liquid Interface Monitor (SLIM). SLIM was developed at FIU for imaging the settled solids layer in million gallon HLW tanks and for quantifying the residual waste volume on the floor of HLW conditioning tanks during pulse jet mixing (PJM) operations. This effort would provide engineers with valuable information regarding the effectiveness of the mixing processes in the HLW tanks. In Summer 2015 the focus of research was changed to address a new Hanford need to investigate the ability of the 3D sonar to image small increases in HLW volume as an early indication of possible Deep Sludge Gas Release Events.

Additionally, the Hanford Site has identified a need for developing inspection tools that provide feedback on the integrity of the primary tank bottom in DSTs. Recently, waste was found to be leaking from the bottom of the primary tank in AY-102. FIU will assist in the development of a technology to provide visual feedback of the tank bottom after traversing through the refractory pad underneath the primary tank.

Task 18 Quarterly Progress – October 1 – December 31, 2015

Subtask 18.1: Evaluation of FIU's SLIM for Estimating the Onset of Deep Sludge Gas Release Events

FIU drafted and submitted a paper for the Waste Management Symposia to be held on March 6-10, 2016 in Phoenix, AZ. The paper covers research related to the previous focus of rapid imaging while mixing and the new scope of monitoring for the onset of Deep Sludge Gas Release Events (DSGREs). The paper was accepted for poster presention at the symposia.

> **Abstract**: 16386 – Sonar Testing, Imaging and Visualization for Rapid Scan Applications in High-Level Waste Tanks
> **Session**: 030C - Posters: Tank Waste
> **Date**: Monday, March 7, 2016
> **Time**: 1:30 - 5:00 PM

During October, FIU continued its literature review of deep sludge gas release events (DSGREs) as well as sonar imaging of gas bubbles and for monitoring gas buildup in solids in tanks. In addition, a successful preliminary proof-of-concept test was completed, imaging pebbles and then the same pebbles with bubble wrap material underneath the pebbles. This is a precursor to working with WRPS during November and December to develop a test plan for 2016 for sonar imaging of increased heights of settled solids surfaces as an indication of possible gas retention.

The goal is to quantify the minimal increases in HLW volume able to be imaged in order to understand and monitor gas retention that might lead to DSGREs.

The initial literature reviewed was from Washington River Protection Solutions on the DSGRE Tall Column Project which demonstrated that gas is not retained in deep HLW sludge and that waste retrievals that moved sludge to double-shelled tanks could safely continue. FIU has reached out to WRPS to request information on the dmax theory related to gas retention from the Dutch as well as reports on the DSGRE Tall Column Project that include the data and data analysis.

Past research at Ames Lab and FIU has shown that the presence of over 1% gas in the liquid phase can result in the complete attenuation of sonar signals (pings) traversing over 10 feet of liquid. This means that pulsed air mixing does not allow for sonar imaging. Other mixing processes that can entrain over 1% of air in the liquid continuously are likely to not allow for imaging also. It is envisioned that pulsed jet mixing would not entrain sufficient air to attenuate the sonar signal but this should be tested with an envelope of PJM conditions to ensure that this is the case. FIU has reached out to others involved in sonar imaging in tanks to ascertain if such data and testing already exist.

A literature review on direct imaging of bubbles by acoustic imaging was also initiated. Bubbles have been imaged with other acoustic imaging systems. The acoustic signal undergoes a strong reflection when the media it traverses has a large spatial density gradient such as at a solid-liquid interface or a liquid-gas interface. FIU will work with WRPS to identify tests to evaluate the ability of the commercial sonars at FIU to image these bubbles. Care must be taken not to generate over 1% entrained air (micro-bubbles) which might completely attenuate the sonar signal.

<u>Preliminary Testing</u>

In the images below are the experimental configuration as well as some commercial sonar images. Figure 1-11 (left) is a photo of a 4.4 inch diameter dish holding small pebbles and a piece of bubble wrap. In the photo on the right, bubble wrap has been inserted under the pebbles, raising the surface 6 mm. Figure 1-12 is a photo of the dish-bubble wrap-pebbles inserted into the 3-ft diameter test tank with the 3D imaging sonar inserted 4 inches into the water.

Figure 1-13 is the commercial sonar image of the metal plate with the dish and pebbles on it. In this image, the field of view is 60 degrees which allows the entire plate and the bottom of the test tank to be imaged. The commercial sonar imaging software interpolates to fill in points not imaged which results in the loss of edges such as that of the metal plate. FIU's sonar data processing software provides a much improved image of sharp edges, steep rises and falls of the solids layer and has several filters to eliminate double scatters, reflections off entrained particles while mixing and more. FIU will process and image this data in the coming months.

Figure 1-14 (left) is the sonar image of the dish with pebbles without the bubble wrap and on the right is the same setup but with the bubble wrap. Note that the field of view has been returned to 30 degrees.

Figure 1-11. Plate of pebbles for sonar imaging and bubble wrap (left) and bubble wrap inserted under pebbles (right).

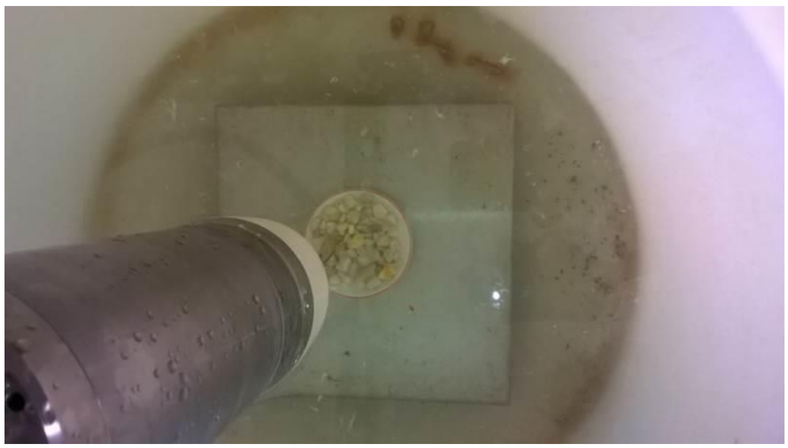


Figure 1-12. 3D Sonar in test tank directly above plate with pebbles.

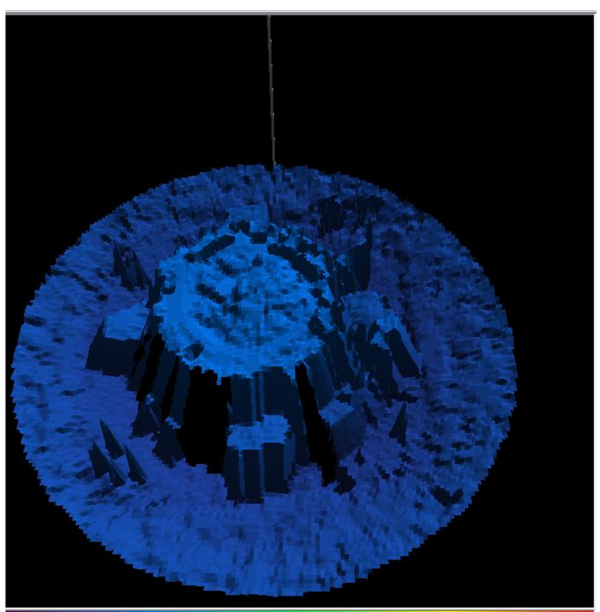


Figure 1-13. Sonar image of large metal plate with small dish of pebbles in the center.

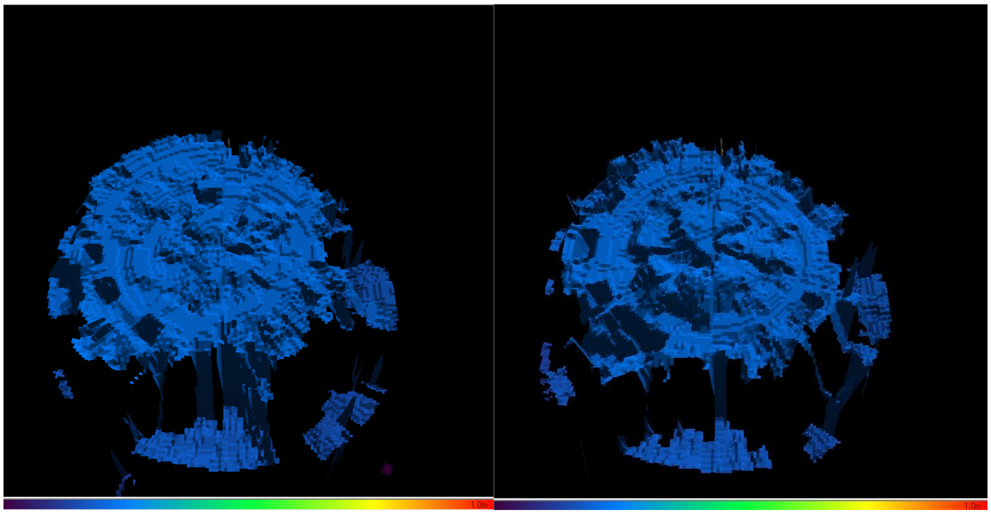


Figure 1-14. Sonar image of dish with pebbles on a large metal plate (left) and bubble wrap added to dish (right).

In Figure 1-14, both images show the flat metal plate and in the foreground is a drop off to the bottom of the tank. In the center of both images, the plate with the pebbles can be discerned although not in the resolution and symmetry possible with post processing. Note the major increase in the height of image of the plate and pebbles that results from adding 6 mm of height from the bubble wrap.

During November, FIU completed testing of the 3D sonar under mixing conditions in order to incorporate research results into the Waste Management paper. A flat, ceramic, rectangular parallelepiped shaped object was used as the object on the floor to be imaged during mixing tests. In Figure 1-15 below is a photograph of this ceramic rectangular parallelepiped (flat plate 13”x7”x1”) object.

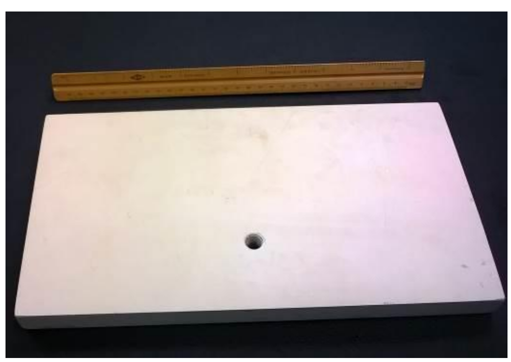


Figure 1-15. Photograph of ceramic object used during initial mixing studies.

In Figure 1-16 that follows, is the commercial sonar’s image of the flat ceramic object. Notice the excellent shape of the object and that the field of view has been optimized to minimize the time for scanning and post-processing.

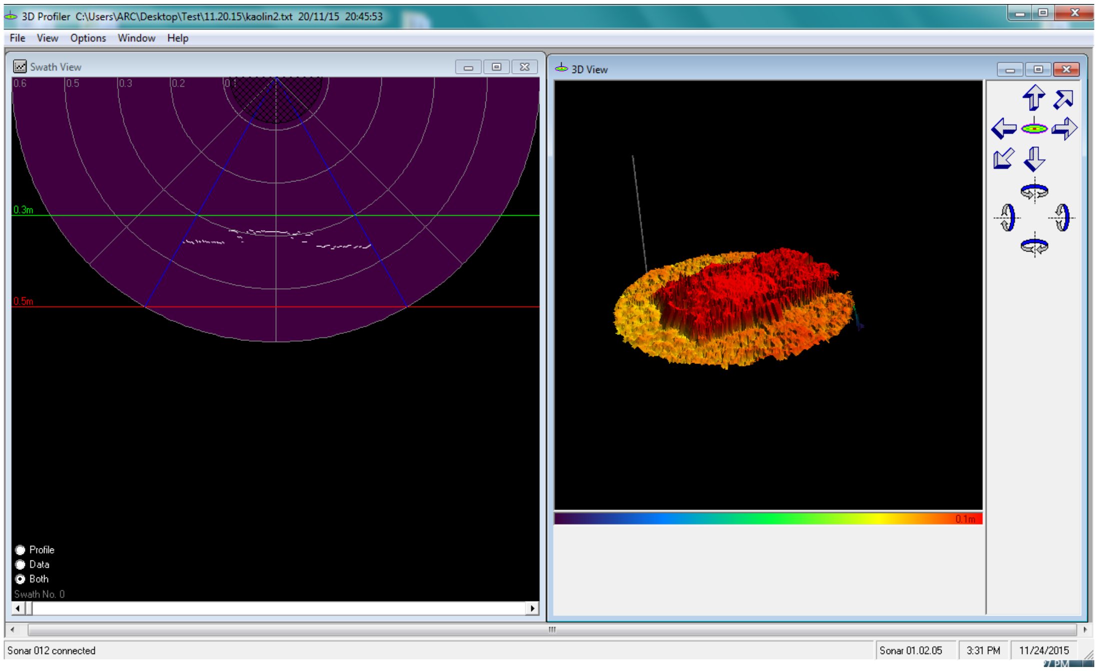


Figure 1-16. Baseline sonar 2D and 3D imaging of the rectangular parallelepiped, ceramic object.

Data from the above baseline object imaging was imported into MATLAB and the resulting image is shown below in Figure 1-17. The sonar and processed images are the same. The importance of the post-processing is more important for short scans and during mixing when the sonar's imaging software cannot create images.

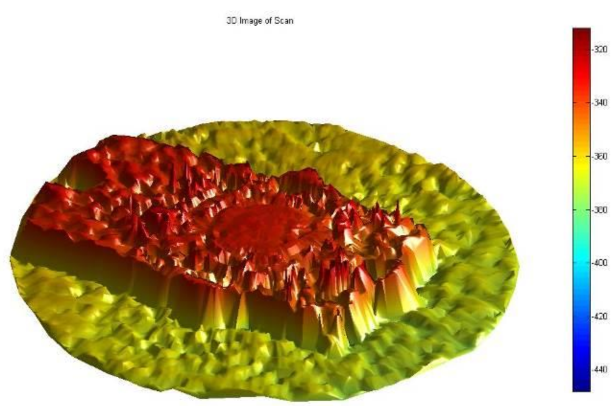


Figure 1-17. Sonar data processed in MATLAB for visualization.

With baseline testing without mixing solids completed, kaolin clay was added to the tank for use in mixing studies. Kaolin clay with ~1 micron diameter was added to yield 5% by volume solids in the liquid with complete suspension of the particles. In Figure 1-18 (upper image) the 2D profile clearly shows the tank floor with the flat ceramic object directly under the sonar. Reflections off particles can be seen above the plate and can be removed in the post-processing of the images. Note that the commercial sonar does not filter the data and so all reflections off suspended particles render the direct sonar's 3D image (lower right) useless. Also note that the

2D profile (lower left) clearly shows the tank floor and the flat ceramic object.

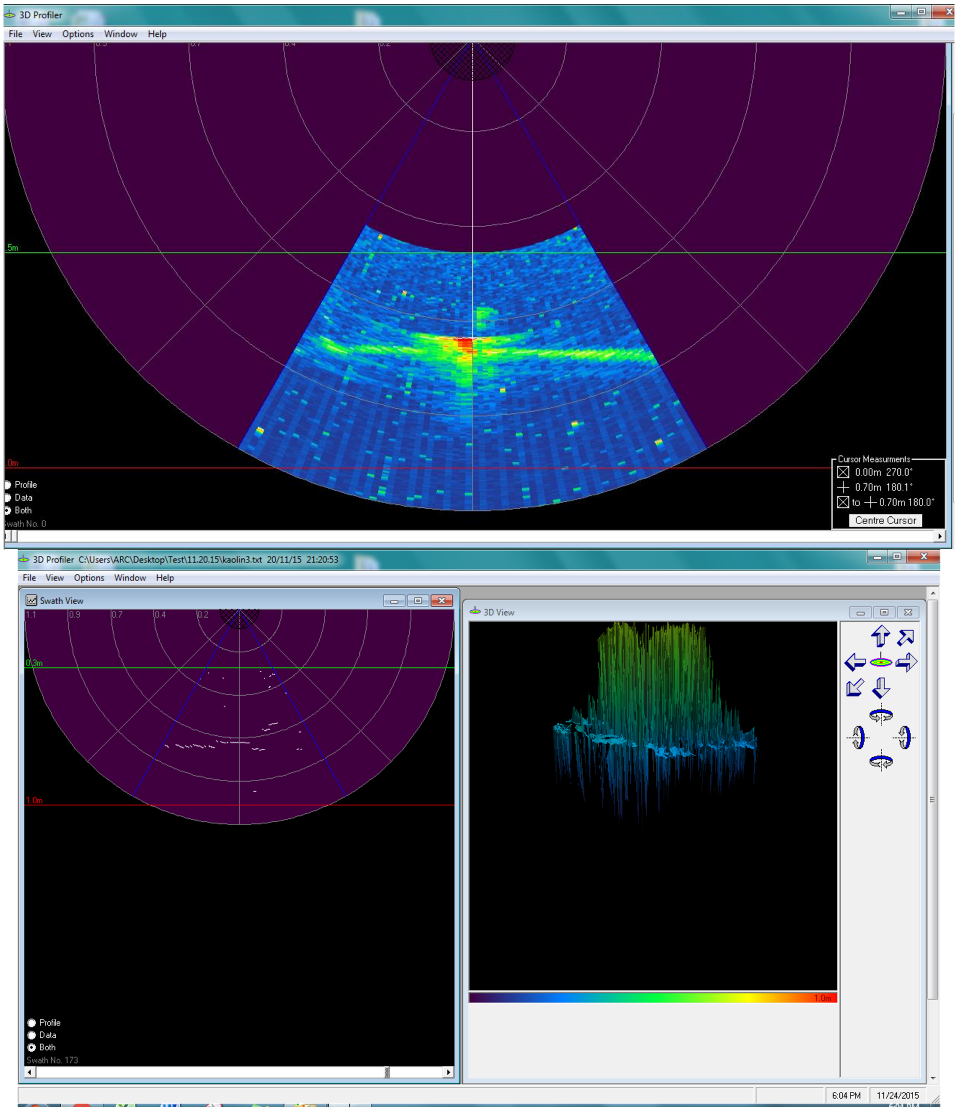


Figure 1-18. Commercial sonar image windows for test involving suspension of 5% kaolin clay in the liquid.

During December, FIU initiated testing of the 3D sonar to measure small changes in the height of settled solids. FIU located a flat aluminum, circular plate that has been inserted into a 24-inch diameter test tank in order to have a flat surface for the floor. The floor of the tank is concave, which presented problems in creating accurate measurements of increases in heights for upcoming experiments.

In Figure 1-19, a plastic lid filled with sand can be seen on the metal plate floor in the 28-inch high, 24-inch diameter test tank. Wires were connected equidistant along the outer circumference of the metal plate to allow for it to be lifted 1 cm at a time.

Figure 1-19. Photograph of plastic lid filled with sand on an aluminum plate on the tank bottom. The 3D sonar can be seen in the center top of the photograph.

In Figure 1-20, the 2D profile image of the 3D commercial sonar of the lid filled with sand is shown. Note that the distance to the sand in the center is 51 cm from the tip of the sonar.

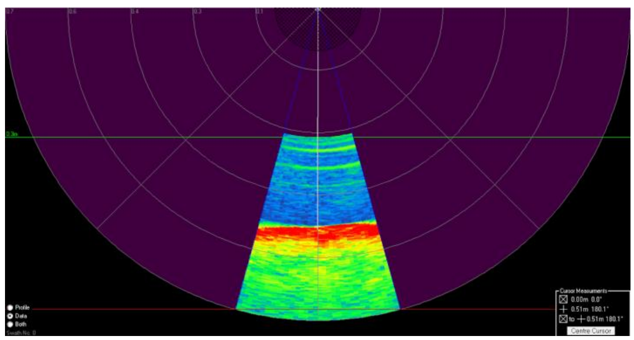


Figure 1-20. Baseline 3D sonar image of the lid filled with sand on the aluminum plate with 4 wires to raise the plate and the solids uniformly 1 mm at a time.

In Figure 1-21, the 2D profile image of the 3D commercial sonar of the lid filled with sand after it has been raised 1 cm is shown. Note that the distance to the sand in the center is 50 cm from the tip of the sonar. This matches exactly with the 1 cm that the entire lid of sand was raised. The accuracy of the cursor in this 2D image is +/- 1 cm. Post-processing of the sonar's 3D data will greatly improve the surface height measurements (mm resolution) and allow for an estimation of the increase in height.

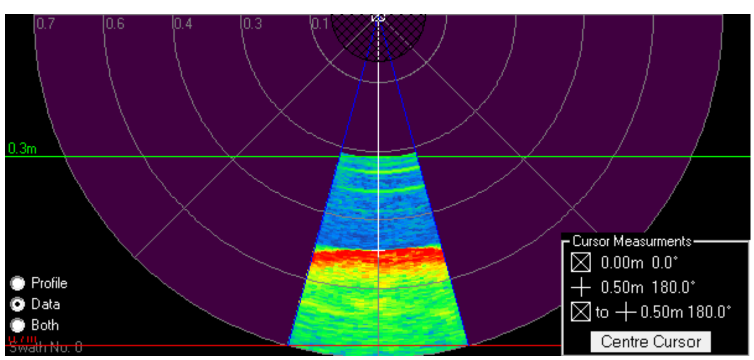


Figure 1-21. 3D sonar image of the lid filled with sand on the aluminum plate with 4 wires used to raise the plate and the solids uniformly 1 cm from the baseline position.

Subtask 18.2: Development of Inspection Tools for DST Primary Tanks

FIU drafted and submitted a paper for the Waste Management Symposia to be held on March 6-10, 2016 in Phoenix, AZ. The paper was accepted for presentation at the conference.

> **Abstract**: 16383 – Development of Inspection Tools for the AY-102 Double-Shell Tank at the Hanford Site
> **Session**: 095 – Novel Inspection – Tools and Equipment to Support Tank Storage
> **Date**: Wednesday, March 09, 2016
> **Time**: 3:15 - 5:00 PM

Miniature Motorized Inspection Tool

For the miniature rover inspection subtask, the refractory mock-up channel was moved to a new lab. The channel allows the operator to observe the response of the device through the clear plastic wall as it navigates inside the mock up channel. Simultaneously, the user can view the path directly in front of the unit which provides a better understanding of the challenges in front of the tool and adopt strategies for navigation and path correction. Figure 1-22 and the red arrow shows the set up and the view from inside the channel (on the screen).

Figure 1-22. Inspection tool laboratory scale mock up test set-up.

Additional magnets were received and allowed FIU to assemble and test the inspection tool with the completed system. The tests to determine the maximum pull force were repeated using a new scale with a maximum capacity of 20 lbs. Figure 1-23 shows the experimental set-up used to measure the maximum pull-force.

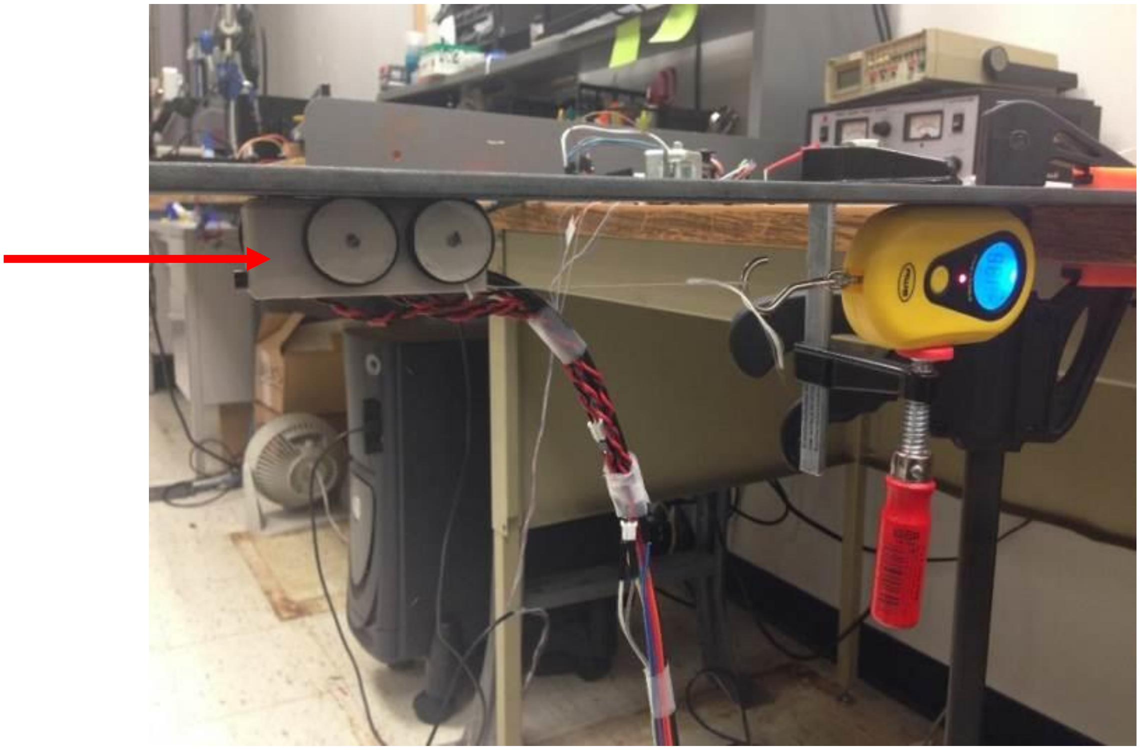


Figure 1-23. Maximum pull force measurement set up, arrow points to the Rover.

Fifteen trials were conducted using two power sources: 1) internal source from the micro controller, and 2) external power supply system. Measurements indicated that the device has an average 4.75 lb pull force with a 5V external power supply. Considering the 0.18 lb weight of inspection tool, its power to weight ratio was determined to be 26. Since the motors are rated for performing between 3 and 9 V, more torque is available from the motors, if needed. However, in order to convert the torque into available pull force, stronger magnets will be needed.

During the month of November, a new camera module controlled by a Raspberry Pi board was introduced into the design of the inspection tool. The primary objective was to improve the camera quality for clear visibility during inspection. The previous camera utilized provided VGA quality (1.3 MP). The module has a five megapixel fixed-focus camera that supports video streaming as well as still captures.

In order to communicate with the camera, the Raspberry Pi 2 system was used, which is the second generation of a single board Linux-based educational computer. The primary components in the Raspberry Pi 2 unit include:

- A 900MHz quad-core ARM Cortex-A7 CPU
- 4 USB ports
- 40 GPIO pins
- Full HDMI port
- Ethernet port
- Combined 3.5mm audio jack and composite video
- Camera interface (CSI)

- Display interface (DSI)
- Micro SD card slot
- Video Core IV 3D graphics core

Figure 1-24 shows the Raspberry Pi camera module incorporated into the inspection tool. Note that the camera module easily fits on the front of the inspection tool and was assembled using the existing fasteners.

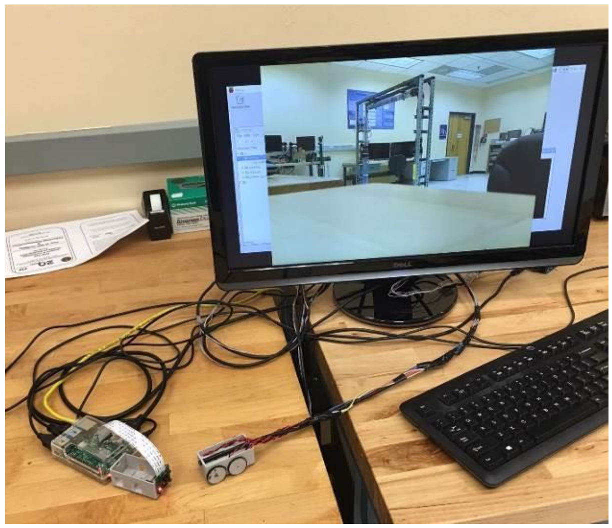


Figure 1-24. Image from the 5 Mega pixel HDMI camera.

The camera module is connected via a 15cm flat ribbon cable to the CSI port on the Raspberry Pi. This system will retain signal integrity but is not suitable for our particular application in this task which requires long distance communication within a tether.

An international vendor was identified that has developed an extension module for the camera. The extension module includes CSI to HDMI adaptors shown in Figure 1-25 on both the camera and Raspberry Pi ends. Both the camera module connector and the HDMI connector have four data buses, which means the small converter only connects the proper pins to each other on a PCB board with no extra electronics on the board. Out of 19 HDMI pins only 15 are used and there are 4 free pins available for future sensor and instrumentation use.

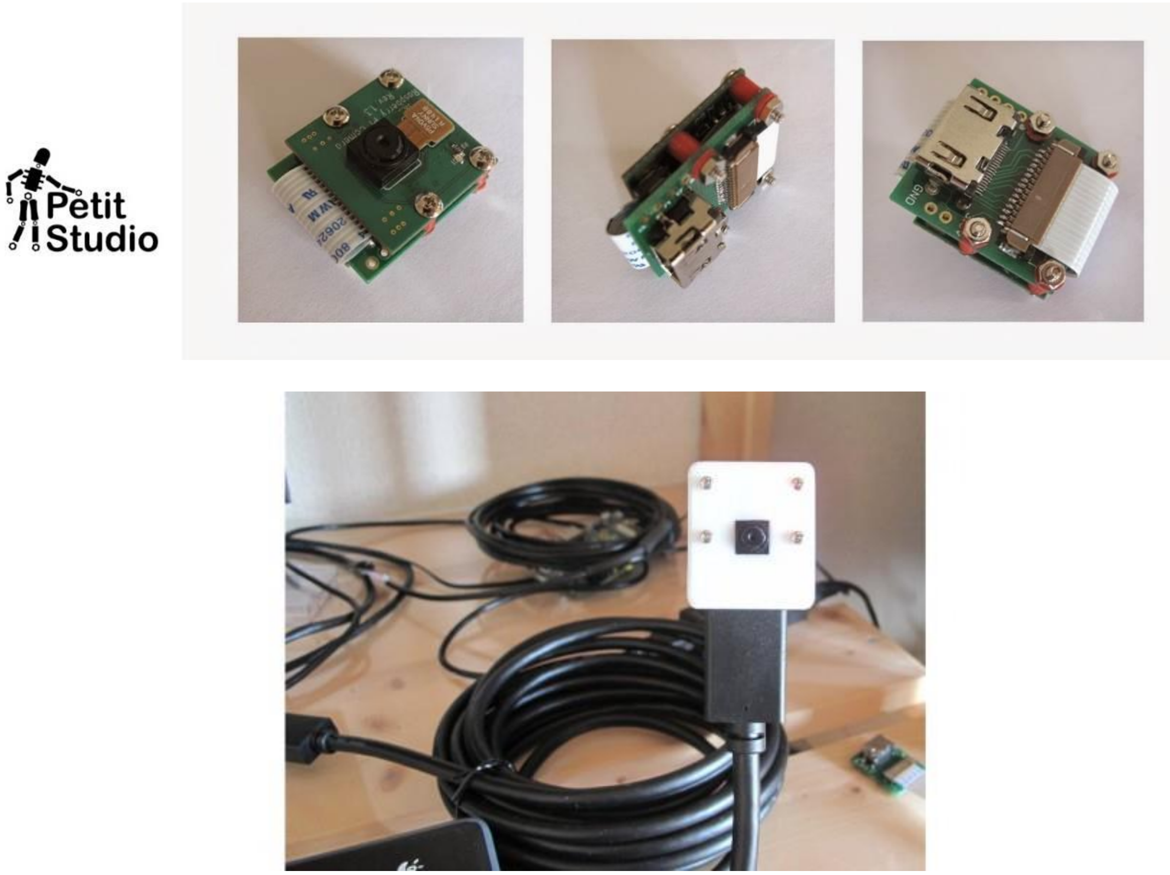


Figure 1-25. The Raspberry Pi Camera Extension adapter and cable developed by Petit Studio.

A full size HDMI cable uses 28 AGW wires that normally requires the cable to be as thick as 10 millimeters. In order to keep the weight and drag force from the HDMI cables minimal, the ultra-thin HDMI based on the RedMere technology was identified and ordered. RedMere cables are designed due to minimal American Wire Gauge and maximum lengths. RedMere cables can be much thinner than normal HDMI cables and can reliably handle high speed (10.2 Gbps) signals over long distances. Additionally, RedMere HDMI cables have considerably smaller outer diameters.

During the month of December, efforts were focused on finalizing the tether design.The goal is to optimize the control system of the inspection tool to keep the number of wires inside the tether to a minimum. In the last monthly report, the integration of a Raspberry Pi camera board and YsImda Ultra Slim HDMI cable to the inspection tool was discussed. The new camera presents various advantages such as computational abilities, improved response time and improved video resolution. The Raspberry Pi camera communicates with the main board via a CSI port that utilizes a 15-cm flat ribbon cable. In order to use an extended cable, the CSI port interfaces with an HDMI cable through the Raspberry Pi camera extension adapter. The HDMI port has 4 extra pins that are not used.

In addition to the camera line, the control circuit of inspection tool uses 8 wires to power and control polarity of the four DC gear motors. The 5 V power source of this configuration provides up to 3 Amps of current while all motors run in parallel. An onboard power source is not desired in this application, and so its size is not an issue. This, in combination with the 4 available pins in the Ysimda Ultra Slim HDMI cable, initiated the idea to include the power cables in this HDMI cable to unify the entire tether in a single cable. This would require a reduction of the power wires supplied to the motors onboard from 8 to 4. As shown in Figure 1-26, a reduction was achieved by configuring the power cables such that 2 motors (each side) ran in parallel, with another set of parallel motors on the other side. As discussed, this reduced the current available to the motors, but depending on the navigation performance of the device inside the mock up channel, alternate power supplies might be evaluated for suitability.

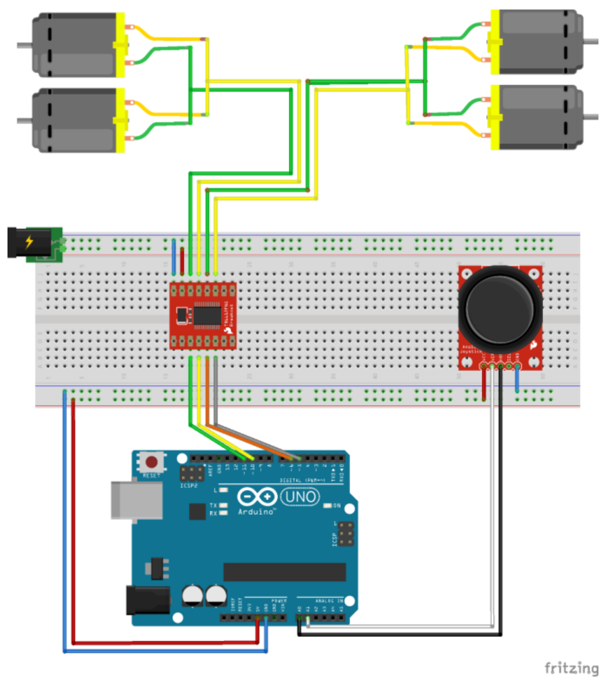


Figure 1-26. Simplified schematic diagram of modified control circuit.

The amount time that the microcontroller takes to process the code affects the response of the system and may give the user a feeling of "lag" if the processing time is too high. For this reason, the code was reviewed for possible length reduction and was successfully reduced from 173 lines to 124 lines of code.

<u>Peristaltic Crawler Inspection Tool</u>

An additional effort under this task is associated with developing an inspection tool that can navigate and provide visual feedback through the 4" air supply pipe that leads to the tank central plenum of AY-102. One of the activities during this period was the construction of a bench scale testbed, shown in Figure 1-27. The modular testbed is currently being used to enhance the crawler design which is in its final stages.

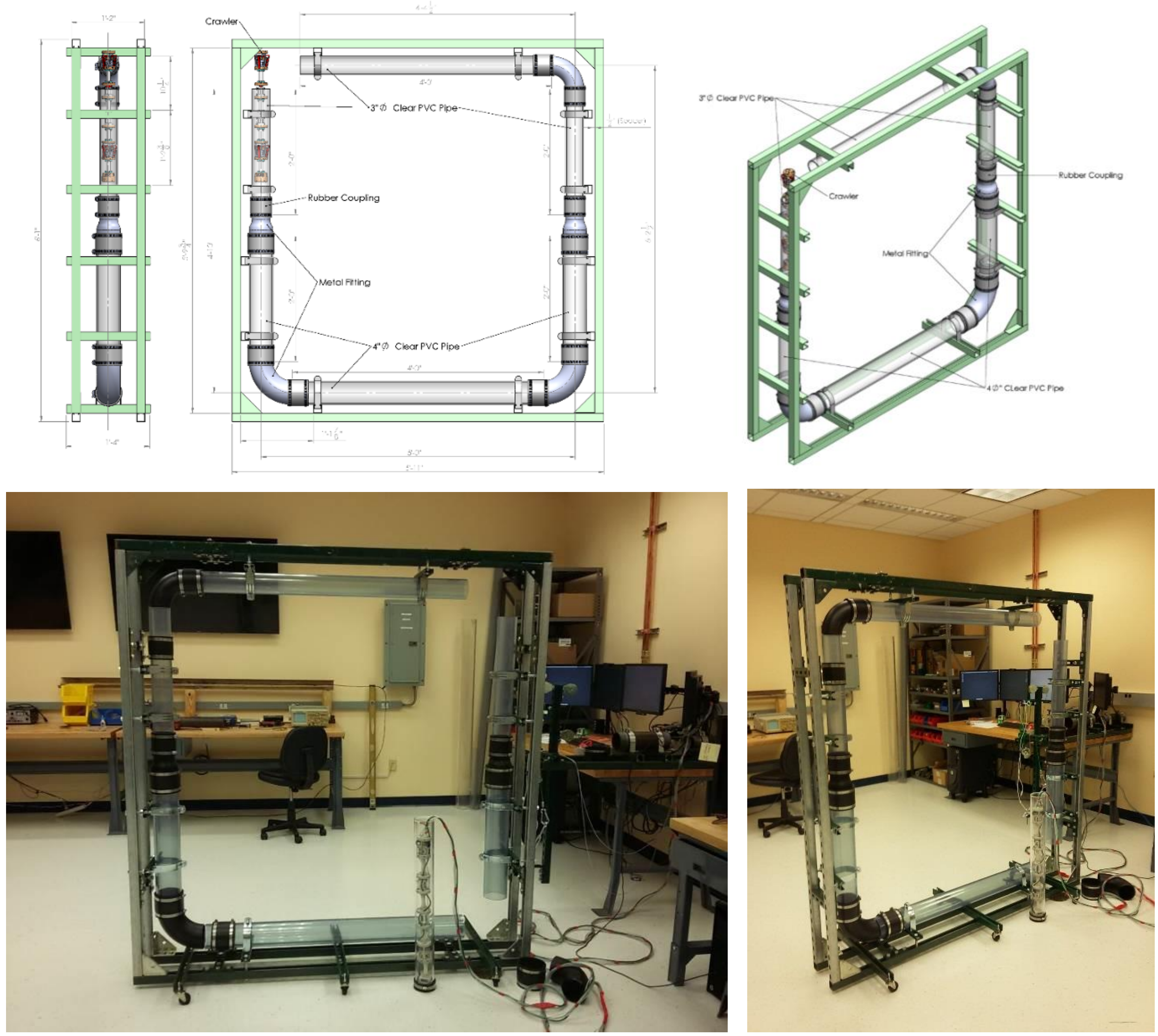


Figure 1-27. Modular bench scale testbed.

One of the design enhancements newly incorporated into the design is the addition of ribs to the case of the front camera, shown in Figure 1-28.

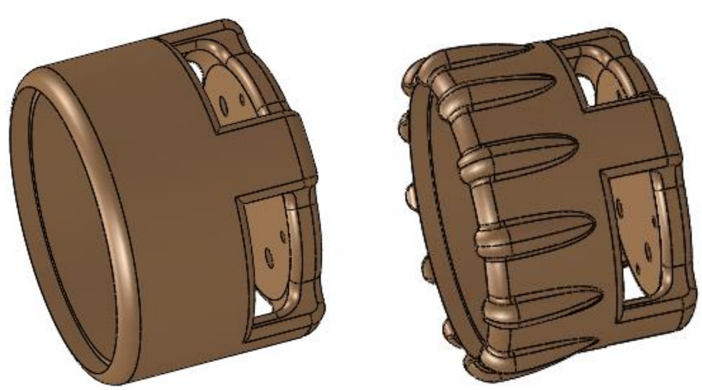


Figure 1-28. Original and redesigned front camera case.

As illustrated in Figure 1-29, the ribs help the camera overcome misalignments in the pipe connections during turning maneuvers.

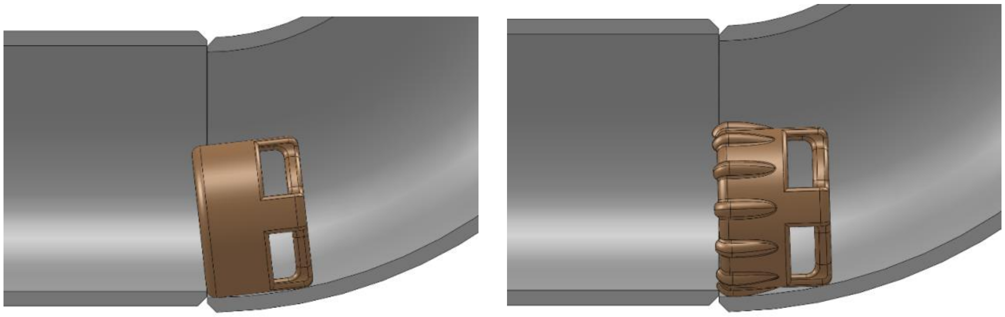


Figure 1-29. Piping misalignment and front camera overcoming.

Figure 1-30 shows the crawler successfully going through the 3" elbow in the pipe loop, where several levels of misalignments were tested.

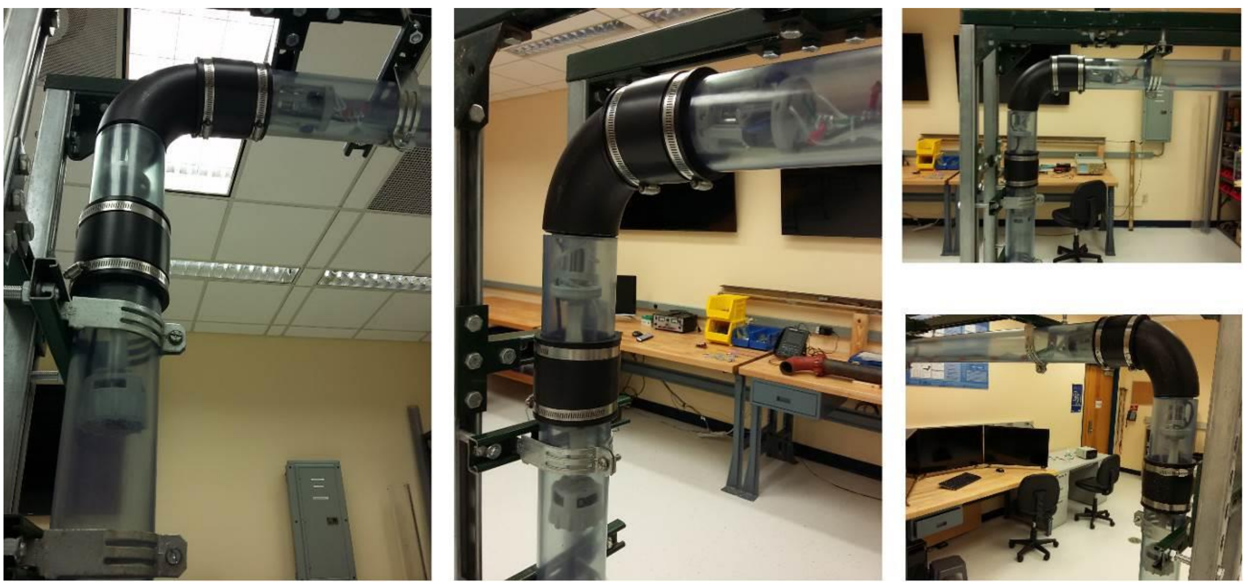


Figure 1-30. Crawler going thru a 3" elbow.

In addition, a prototype of the previously designed load cell was built during the same period. The load cell, shown in Figure 1-31, will be attached to the tether. The load cell in conjunction with the bench scale testbed will be used to estimate the tether dragging forces associated with the proposed inspection.

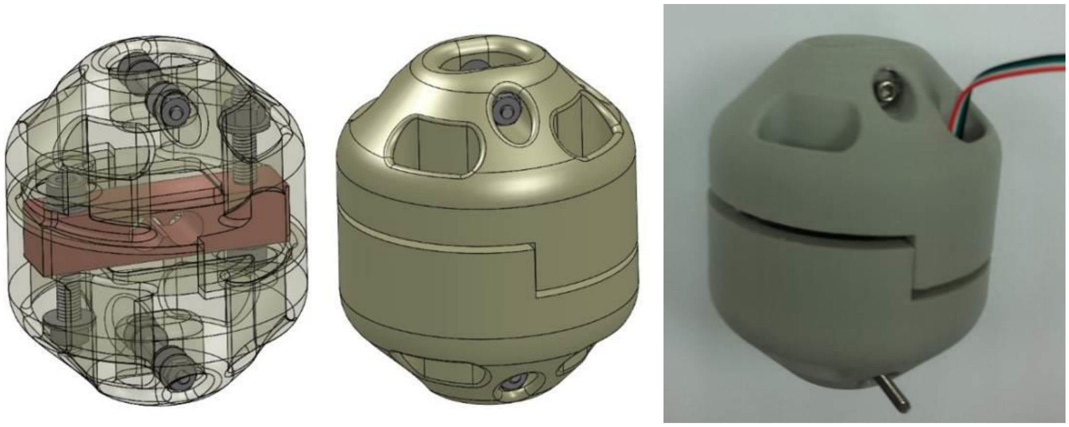


Figure 1-31. 100 lbs tether load cell.

In an effort to finalize the tether design, several abrasive resistant sleeves were also evaluated with the objective of reducing the tether drag.

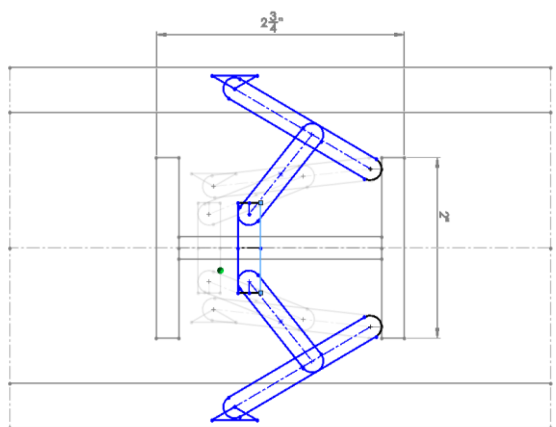


Figure 1-32. Schematic of an electric gripper.

A prototype of the gripper with five claws was built as shown in Figure 1-33. A stronger grip is expected by the increased contact area of the redesigned claws using a hinged flat pad. A stronger grip would allow for longer inspections, and allow the crawler to carry additional instruments and payload. Perusing this goal is critical in using the crawler for future inspections, other than inspections of AY-102 tank.

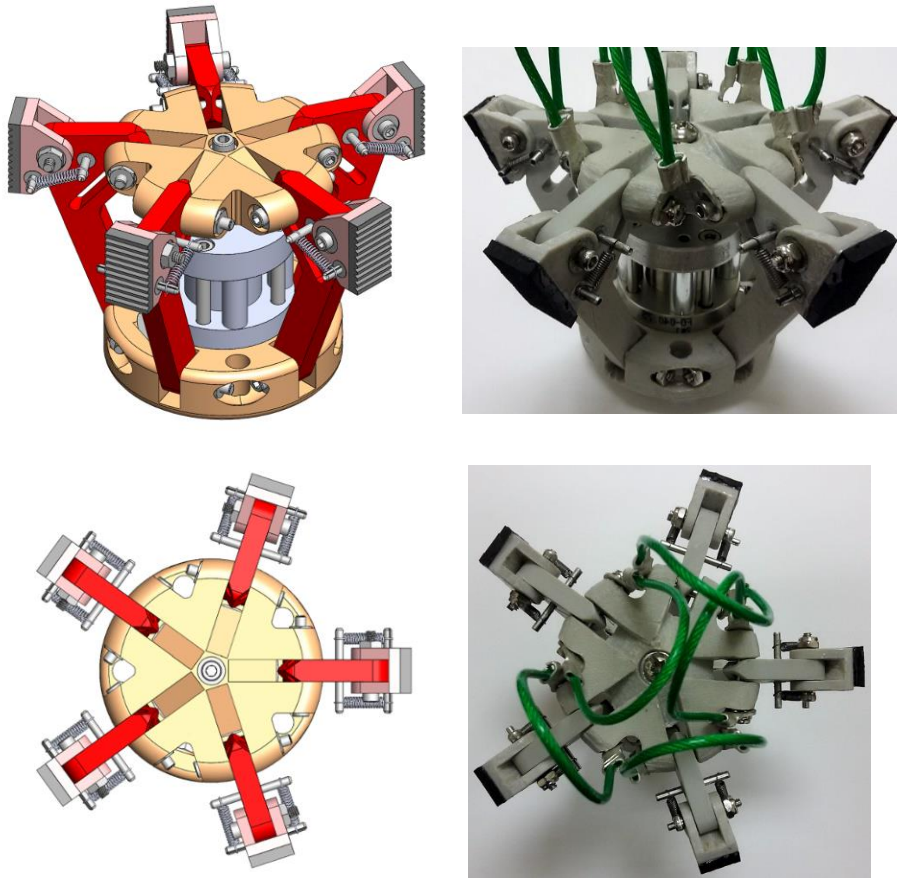


Figure 1-33. Five claw gripper design (left) and prototype (right).

In addition, a shorter gripper was also designed. As shown in Figure 1-34, the module length was reduced by almost 5/8”, without relevant changes to the original locking mechanism. A compact gripper will significantly improve the navigation ability of the crawler through 3” bends. Future modules should not be designed with dimensions greater than 3” in length and 2.5” in diameter, due to the restrictive dimensions of pipefittings with a 3” diameter.

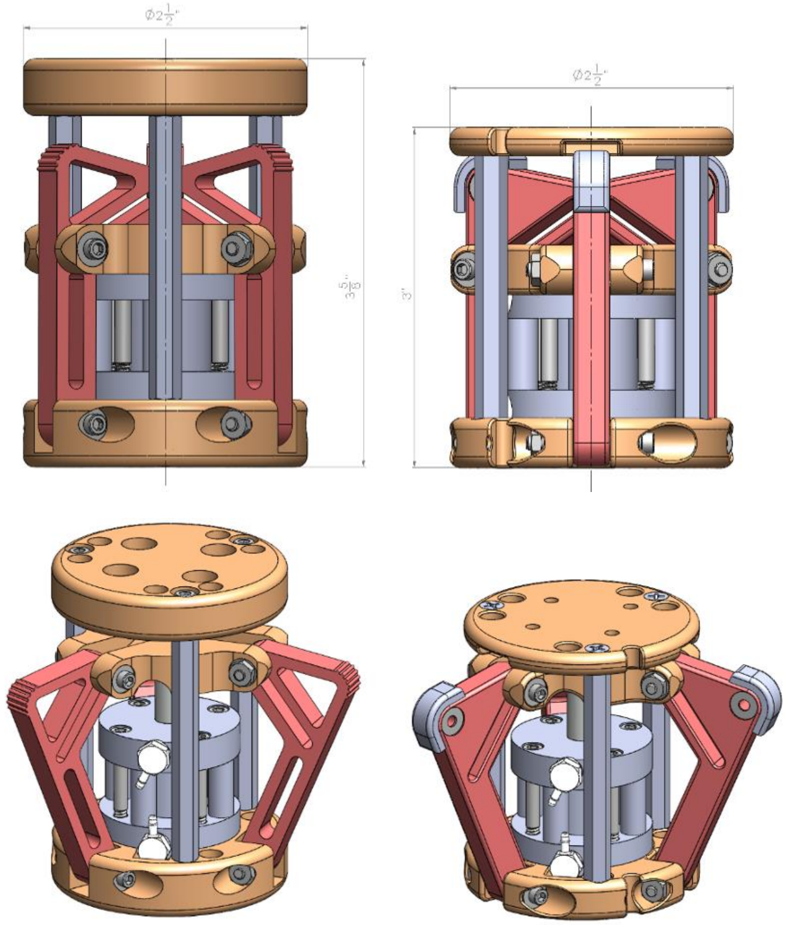


Figure 1-34. Original gripper (right) and redesign (left).

The gripper's previous design has been improved. The primary improvement consisted of increasing the contact area of the gripper with the wall which should increase the grip strength. Figure 1-35 shows the evolution of the gripper design throughout the year.

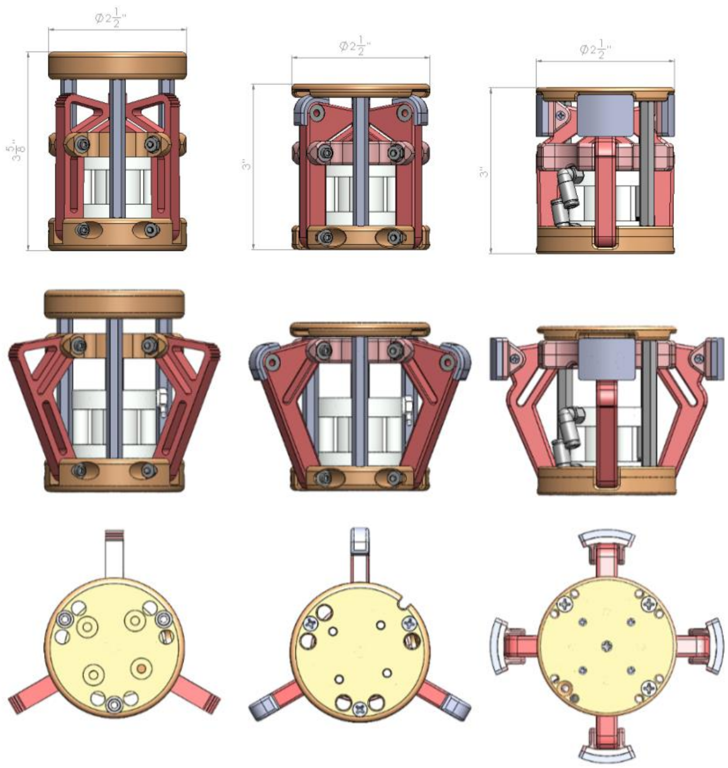


Figure 1-35. Original gripper (left), previous (center) and improved (right) design.

As shown in Figure 1-36, the new gripper has four claws with cylindrical pads attached to the tip of each claw. The cylindrical pads have dimensions of approximately 1" by 3/4". The improved design also uses push-to-connect tube fittings to supply air to the pneumatically cylinder. These fittings are more reliable than the barbed fittings used previously. The structure of the gripping mechanism was strengthened as well, and the pin inserts were embedded in the mechanism hinges in a slimmer design.

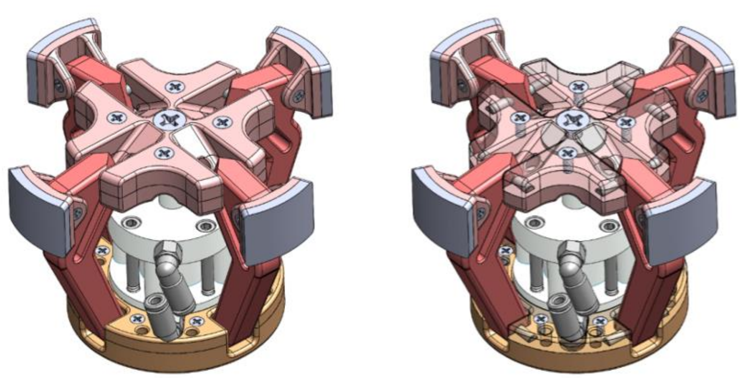


Figure 1-36. Gripper's current improved design.

Also in this period, another relevant task was to study the strengthening techniques for 3D printed parts. The use of thermoplastic parts has significantly expedited the design process of the crawler; however there are multiple concerns related to their strength and durability. Weak points, especially the interlayer adhesion, lead to premature fatigue and eventually delamination. Typical wear and tear during repeated friction raises concern about their long-term use. Most popular strengthening techniques utilize coatings after manufacturing.

One method involves coating the 3D printed part with epoxy resin. These resins have remarkable adhesion properties, which results in a significant increase in the interlayer strength of the printed material. This technique works well with ABS plastic material, such as the ones used in the crawler. After mixture, the epoxy resin is heated to lower the viscosity, which promotes better spreading. The part is then soaked in the epoxy and left to dry in an oven.

Another alternative is to use an acetone vapor bath. In this process, the vapor penetrates the interlayer spaces of the 3D printed material, leaving the solvent bonded afterwards. The acetone works especially well with ABS type plastics. Another benefit of the vapor bath is that the part will be left with a smooth print surface with a high gloss sheen.

There are a few other methods that are also being considered. This includes a rubberized dip protective coating that will leave the part with a waterproof seal and should increase the durability and strength of the piece. Fiberglass coatings and resins are also being investigated.

The 3D parts can also be reinforced with injected structural adhesive, by printing pieces with additional recesses and sparser infill printing pattern. Afterwards, the patterns are filled with the adhesive, which has superior mechanical properties than the printed part. Bonding inserted metal parts, such as bolts, nuts, washers and plates are also promising, especially when threading is involved. These methods are particularly promising, especially combined with the others described above.

A suspension mechanism was also designed during this period. Figure 1-37 shows the mechanism, which uses small arms and torsion springs to keep the crawler at center of the pipes while crawling. The suspension mechanism will be installed in all modules of the crawler, with the objective of minimizing bouncing and dragging. It will also prevent the collection of debris (bulldozer effect) in the front camera.

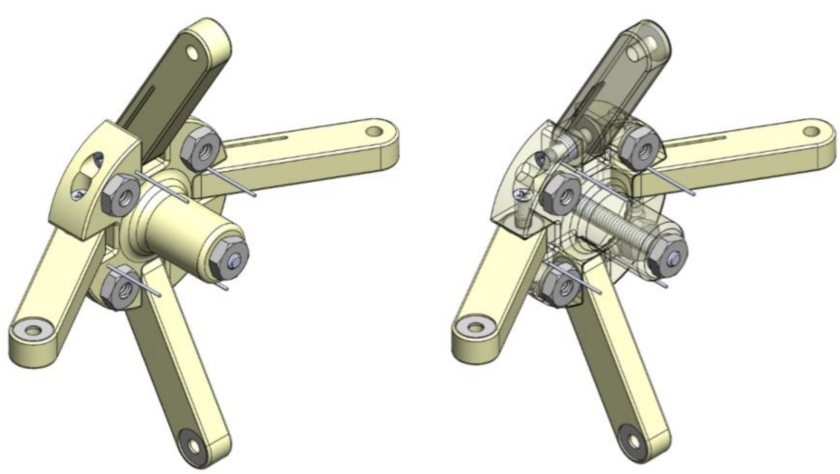


Figure 1-37. Suspension module.

Another component designed during the period was a tether quick connector, shown in Figure 1-38. The connector is a coupling fitting that uses eight push-to-connect tube connectors to straight-connect the pneumatic lines. The connector will allow for quick disconnects and tether extension. The coupling also provides central access for passing other cables.

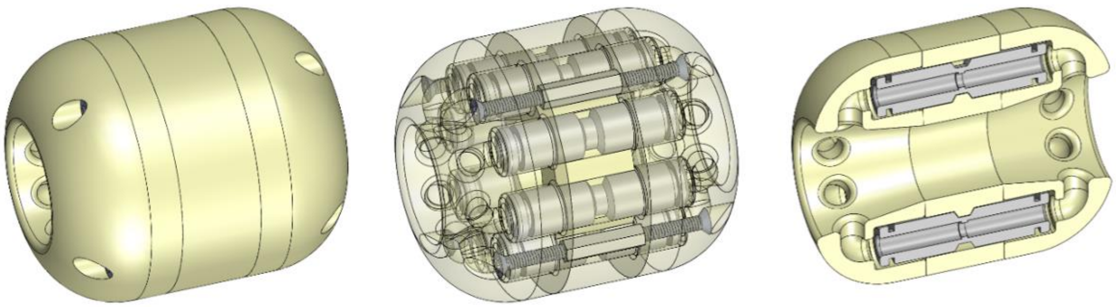


Figure 1-38. Tether couple fitting.

Figure 1-39 provides a rendering of the updated crawler design. The rendering, however, does not include the improved grippers, but shows the suspension mechanism attached to crawler modules. It also shows the load cell and the quick connector placed at end of the unit.

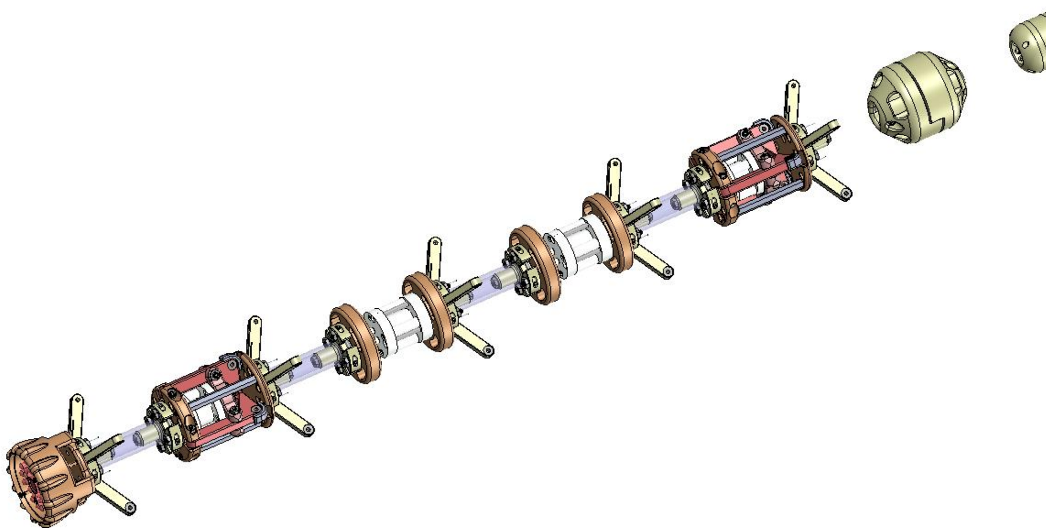


Figure 1-39. Crawler, load cell and quick connector.

In addition, a bench scale testbed (Figure 1-40) was constructed with the objective of evaluating the effect of the gripper contact area on the gripping force. The testbed included simultaneous testing capabilities of 3" and 4" pipes, a pulley system, and a turnbuckle used to deliver gradual load increases. In order to record the maximum sustainable force of the grip module, a hand scale with a capacity of 100 lbs was used.

Figure 1-40. Testing platform.

During the tests, the base of the platform was hooked to a steel turnbuckle, which was attached to a scale on the opposite side. As shown in Figure 1-41, the scale was held by a steel wire which fed throughout the pulley system, to the end of the gripper module. The turnbuckle shortened as it turned, which provided increasing pull on the scale and the gripper. The process continued until there was any slip noticed in the grip.

Figure 1-41. Turnbuckle and scale.

The platform has been used to record the effects of the improvements in the design that includes changes in the number of claws and pipe grip contact area. Examples of previous designs tested are shown in Figure 1-42. The recent design modifications have improved the gripping force from 18 to 41 lbs.

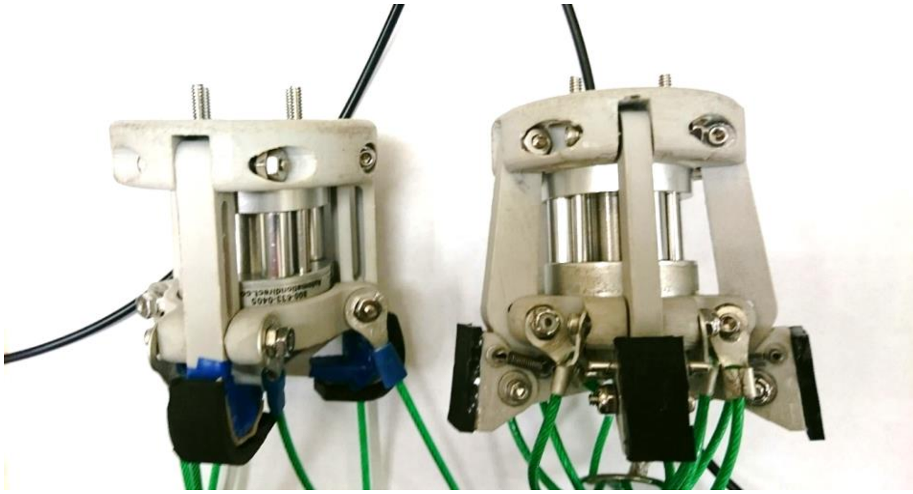


Figure 1-421. Three (left) and five (right) claws grippers.

Subtask 18.3: Investigation Using an Infrared Temperature Sensor to Determine the Inside Wall Temperature of DSTs

This is a new task to determine the temperature measurements of the inside wall of DSTs at Hanford. The basis for this study is to ensure that the temperature inside the DSTs is as per the standards stated in OSD-T-151-00007. Benefits include avoiding corrosion, improving existing models and accurately estimating the elevation of solid waste levels at the wall.

During this performance period, further information regarding the IR sensor and its benefits has been acquired from the site engineers.

The IR sensor requirements are specified as below:

1. Must be a non-contact pyrometer
2. Must be mounted and remote controlled
3. Should be wired (long wire must be available 50' – 75')
4. Must be able to get temperature from dull/rusty carbon steel
5. Must be able to get accurate reading from a distance of 1-3 inches
6. Must have software compatible with windows machine (Windows 7)
7. Software must support data logging
8. Temperature measurements will be 0°F to 250°F
9. Able to operate in an environment of 40°F to 150°F
10. Equipment must have adjustable emissivity

Further, the dimensions of the IR sensor should be suitable to fit within a 6 cubic inch volume. The preferable size would be 6" x 6" x 6" but an alternative size could be 8" x 4" x 4". The spectral range of the sensor should be 8 μm to 14 μm; response time should be less than 1 second and the aperture must be less than 1".

Additionally, the benefits of temperature measurements using the IR sensor will serve multiple purposes as listed below:

1. Real data will be available for ensuring limits are in fact met and if they are not met, immediate mitigation steps could be taken.

2. Physical properties such as thermal heat transfer coefficients could be empirically calculated.
3. Current modeling techniques can be either validated or disproved, improving our overall thermal modeling capability.
4. Solid waste levels could be estimated at the tank wall by looking at temperature gradients.
5. Temperature dependent testing equipment can be more accurately calibrated which in turn could contribute to the UT crawler producing better results.
6. Also, as an added benefit, costs incurred from expensive and time consuming thermal modeling could be reduced or eliminated in situations where temperature data are needed reactively.

The scope of work for the present task has been defined based on the input from the site engineers and a test plan for the bench scale testing of an IR sensor to detect tank temperatures was developed. The test plan was delivered to Hanford engineers and representatives from DOE for their review on 12/18/15 (milestone 2015-P1-M18.3.1). The focus for the test plan is to evaluate an IR sensors ability to measure temperatures within the primary tank from the annulus. The test matrix includes varying the tank temperature, vertical location of the measurement, distance from the tank and thickness of the tank.

The scope of the present test plan, in brief, includes the following:

1. Designing and constructing a bench scale test set up.
2. Formulation of the test matrix.
3. Conducting the tests.
4. Theoretical calculations based on the principles of heat transfer.

Figure 1-43 shows a schematic of the experimental set up. The test set up will consist of a rectangular tank (approximate dimensions 3ft x 3ft) of which one of the sides will be made of the test material (carbon steel). The tank will be filled with water and maintained at a particular temperature for a specified time interval. The non-contact IR sensor will be used to scan from the top to the bottom of the tank recording the outer tank wall temperatures at different points. The important parameters being considered to develop the test matrix include thickness of the plate (tank wall), distance of the sensor to the tank wall, temperature of the water inside the tank, and points (height) of measurement. Also, thermocouples will be attached at various points on the tank wall to verify the actual values. Temperature data sets obtained from the experiments will be used further for heat transfer calculations in the estimation of inside wall temperatures.

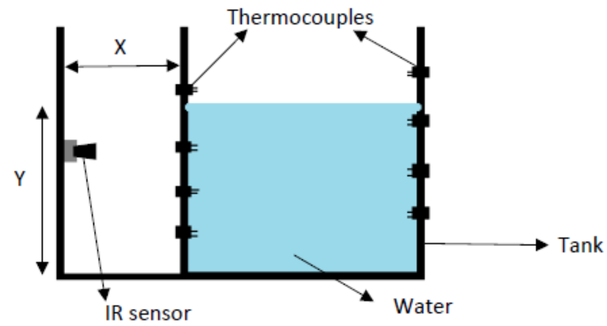


Figure 1-43. Experimental set up.

Additionally, the equipment recommendations made by the site engineers included: The OS-MINI Series from OMEGA® [1], The MI3 from Raytek® [2] and the ThermoMETER CT by Micro- Epsilon® [3]. Based on the information gathered, the Raytek system was selected since it is most similar to the sensors used on-site. A brief description of the sensor is given below:

The Raytek MI3 is a pyrometer (non-contact sensor) that includes a digital screen for temperature display. It mainly consists of two parts: the sensing head and the digital communication box. Based on the specifications of the present task, the product has variable (adjustable) emissivity. The spectral range of the MI3 series is 8-14 µm with a response time of 130 ms and an accuracy of 1° Celsius. It is 0.55 inch in diameter and 1.1 inches in length. Also, a 98 feet cable is available for the sensing head which is one of the major requirements for it to be integrated with the tether of the inspection tool on which the sensor is supposed to "piggy back".

Currently, FIU is in the process of acquiring the IR sensor from Raytek (Model MI3) to start building the test set up. Information was also gathered and a quote was obtained for an IR sensor recommended by the site engineers. The quote for the Raytek IR sensor had the following specifications:

1. Temperature measurement range: 0-250 ºF
2. Spectral range: 8-14 micro-meter
3. Optical resolution: 20:1-30:1
4. Ambient temperature: 40-150 ºF
5. Cable length: 100 ft
6. Adjustable emissivity
7. Cost under $3000

References:

[1] www.omega.com

[2] www.raytek.com

[3] http://www.micro-epsilon.com/index.html

Task 19: Pipeline Integrity and Analysis

Task 19 Overview

The objective of this task is to support the DOE and site contractors at Hanford in their effort to evaluate the integrity of waste transfer system components. This includes primary piping, encasements, and jumpers. It has been recommended that at least 5% of the buried carbon steel DSTs waste transfer line encasements be inspected. Data has been collected for a number of these system components and analyzed. Currently, different ultrasonic transducer systems are being investigated for thickness data measurement to determine the actual erosion/corrosion rates so that a reliable life expectancy of these components can be obtained. An additional objective of this task is to provide the Hanford Site with data obtained from experimental testing of the hose-in-hose transfer lines, Teflon® gaskets, EPDM O-rings, and other nonmetallic components used in their tank farm waste transfer system under simultaneous stressor exposures.

Task 19 Quarterly Progress – October 1 – December 31, 2015

Subtask 19.1: Pipeline Corrosion and Erosion Evaluation

One effort on this subtask was focused on acquiring the flexible smart layer sensors and performing initial laboratory tests on them. In addition, another UT sensor option (which required no couplant) was investigated.

The smart layer single sensor (Acellent technologies) was ordered and received. The sensor as shown in Figure 1-44 consists of a single piezo-element and circuit embedded in the Kapton tape. On one end, it has a connector to connect a sensor cable for capturing the signal. Initial oscilloscope tests were conducted using the single sensor to ensure that the sensors were functioning. The setup is as shown in Figure 1-45. The sensor was manually vibrated to see if the signal was being captured by the oscilloscope. The sensor placed on a 3 inch Victaulic pipe bend is as shown in Figure 1-45. Based on the experiments, it was concluded that the smart sensor was able to transmit the signals.

Figure 1-44. Smart sensor placed on a pipe.

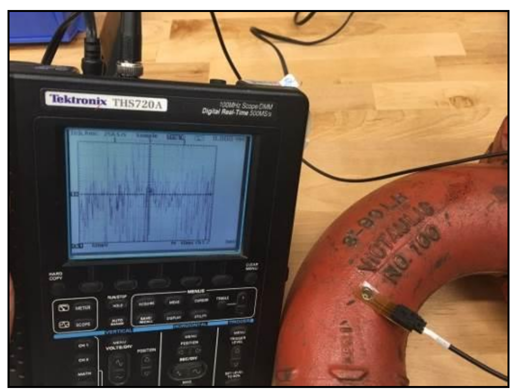


Figure 1-45. Oscilloscope tests using the smart sensor.

We also began setting up the data acquisition (DAQ) system for the sensor. To be cost effective, we are looking into available in-house DAQ systems. If needed, however, we will also investigate purchasing the system.

As part of this study, additional UT sensors were investigated and included a metallic UT sensor similar to the traditional sensors but requires no couplant. It is a dry contact sensor developed by Ultran Group [2] and works by applying a suitable torque to the sensor when mounting. It comes with a polystyrene base and is available in 2MHz and 5MHz frequencies for our requirements. A typical picture of the Ultran sensors (WD 25-2) is as shown in Figure 1-46.

Figure 1-46. **Ultran couplant free contact sensors.**

The Ultran UT sensors are couplant free and are available in smaller dimensions (6.4 mm active diameter) for mechanically mounting four of them in a 2-inch diameter pipe. Currently, we are in the process of acquiring units for testing and developing the data acquisition systems.

Another effort focused on performing the time and frequency domain analysis for the smart sensors and ordering the Ultran couplant free UT sensors. The current lead time for the arrival of these sensors is 4-6 weeks.

The Accelent smart layer single sensor was used to conduct simple thickness measurements. An experimental set up to record pulse-echo sound waves from the smart sensor is shown in Figure 1-47. Input was provided using a function generator, which was used to generate pulses to be transmitted into the pipe through the sensor. Burst mode operation was used to generate pulses since a default pulser-receiver system was unavailable. An echo sent back by the sensor was captured by the oscilloscope (Tektronics THS 720A) as an output. A Fast Fourier Transforms (FFT) spectrum was obtained. By picking up the changes in electrical signal over time, the oscilloscope was able to continuously graph the echoes that were sent back by the UT sensor.

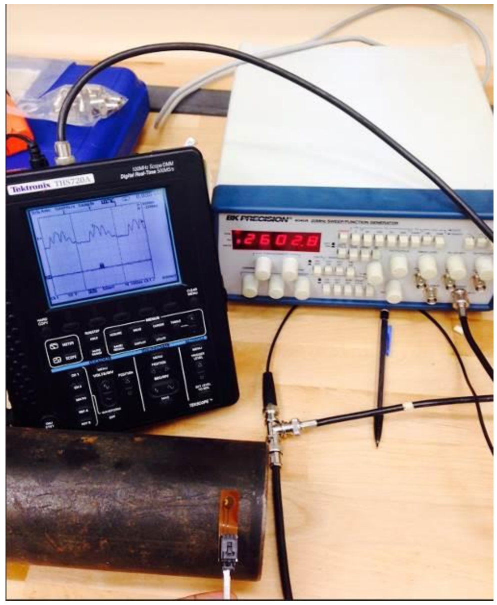


Figure 1-47. **Smart sensor placed on a pipe.**

In order to calculate the thickness of the pipe wall, the time period of oscillations was recorded. The velocity of sound in the carbon steel test piece was obtained from literature, 5920 m/s. The distance traveled is simply determined by multiplying the velocity of sound in the pipe by the time of travel. Certain anomalies, however, were observed while recording the oscillograms (time-amplitude graphs). Currently, the anomalies are being investigated. FIU is also waiting on the Ultran sensors to arrive to initiate testing.

One of the sensors that is being evaluated to obtain real time thickness measurements is the Accelent smart layer sensor that is similar to the sensors used in the Pipewrap system that was implemented by WRPS and had little success. These sensors are couplant-free and are used in flexible layers that can easily install on any diameter pipe. Initial testing demonstrated that these sensors can be used for measurements; however, additional instrumentation is required to complete the evaluation.

Another sensor that will be evaluated is the Ultran UT sensor which is also couplant-free and is currently being shipped to FIU. These sensors will also be capable of being installed on two- and three-inch diameter piping; however, a data acquisition system will be required. We are awaiting a quote for the system to determine if it will be beneficial to continue investigating this system.

References:

[1] http://www.acellent.com

[2] http://www.ultrangroup.com

[3] http://host.uniroma3.it/laboratori/escher/res/ESP_III/Data%20Sheet/TEK_THS730A_Manual.pdf

[4] http://people.ece.cornell.edu/land/courses/ece4760/equipment/BK4040a.pdf

Subtask 19.2: Evaluation of Nonmetallic Components in the Waste Transfer System

FIU drafted and submitted a paper for the Waste Management Symposia to be held on March 6-10, 2016 in Phoenix, AZ. The paper was accepted for presentation at the conference.

> **Abstract**: 16302 – Evaluation of Non-Metallic Materials in the Waste Transfer System
> **Session**: 030 - Posters: Facility Structural Integrity
> **Date**: Monday, March 07, 2016
> **Time**: 1:30 - 5:00 PM

One effort during this performance period focused on obtaining the components needed to finish assembling the test loop and perform blowout tests on the HIHTL coupons from River Bend. For the test loop, we decided to purchase 2" PVC SCH40 Unions for the in-configuration testing of the O-ring samples because of the low cost and the similar O-ring exposure area compared to the Chem Joints. In addition, after discussions with engineers from WRPS, we are going to proceed with the in-configuration testing of Garlock Blue-Gard 3700 Series Gaskets. We are currently in the process of procuring all Garlock Blue-Gard 3700 Series gaskets at 1/8" thick to match the exact gaskets that are used in the Hanford HIHTLs.

In preparation for the blow out tests of the HIHTL coupons, all parts/fittings necessary for the tests were ordered and most have been received. We are currently waiting on the fittings ordered by a local vendor to arrive to begin development and preliminary testing. In addition, we also ordered a pressure transducer from Barksdale rated to 7,500 psi (Model 425H3-17) to acquire accurate max pressure readings for the purpose of developing degradation graphs between experimental results.

FIU also focused effort on completing the assembly of the test loop. Test loop assembly was completed and the milestone was met, see Figure 1-48. The test loop consists of schedule 40 PVC piping attached to the HIHTL coupons. There are three separate loops that will each have a different temperature bath (70, 130, 180°F). Each loop will have 6 coupons, 3 that will be exposed for 180 days and 3 that will be exposed for 1 year. Nonmetallic gaskets and O-rings have also been placed in line so they may be evaluated as well.

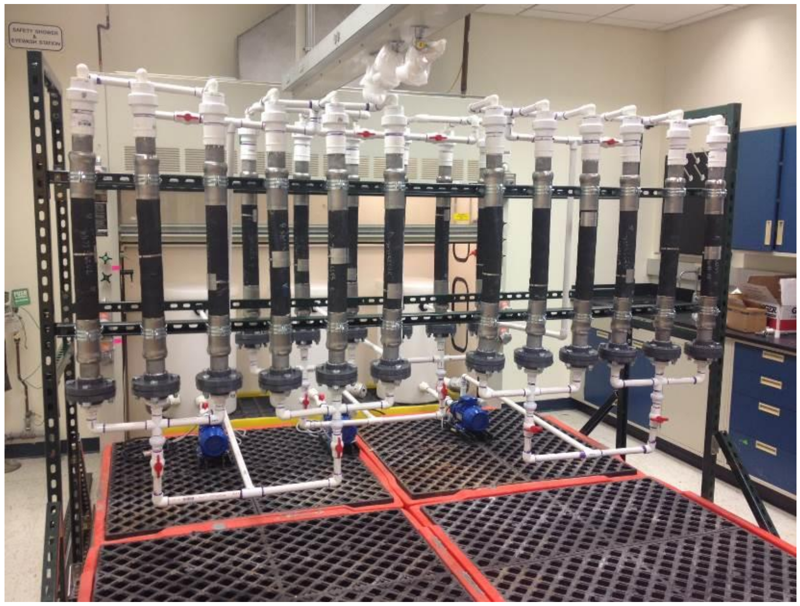


Figure 1-48. Test loop set up.

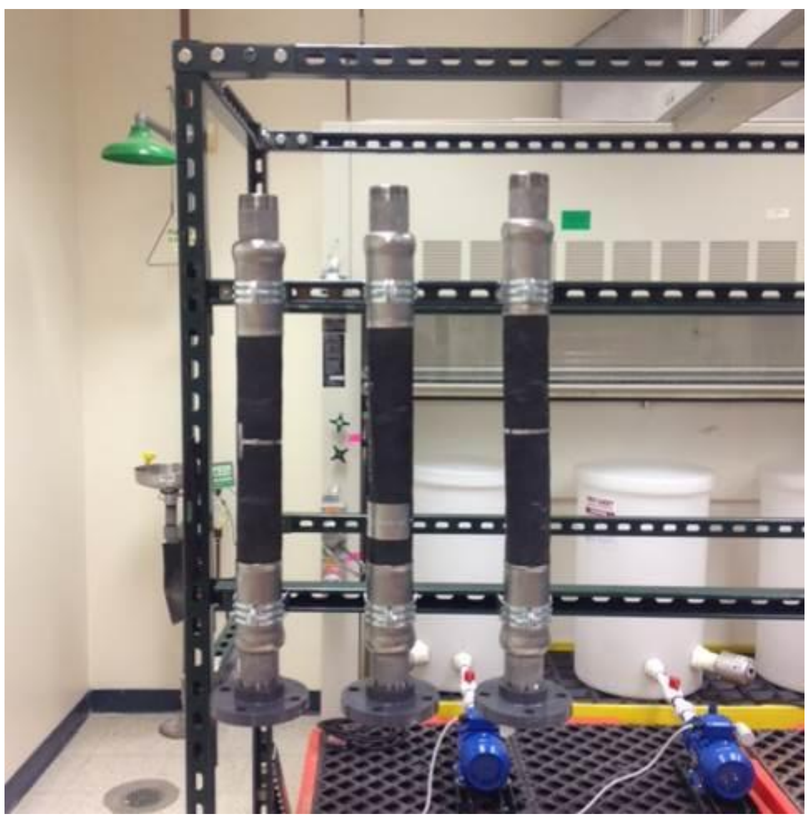


Figure 1-49. Test coupon length variation.

As can be seen from Figure 1-49 the test coupons had length variations of up to 1.5”. The variation is due to manufacturing discrepancies but is not expected to affect the experimental results. The coupon lengths will be noted in the event that anomalies in the burst pressure tests are observed.

Finally, a die has been obtained for manufacturing EPDM and Garlok tension specimens. These specimens will be placed in the three tanks and aged. After the aging, material property tests will be conducted to determine the effects of aging on various properties.

FIU also conducted system shakedown tests on the experimental test loop. During the shakedown, some leaks were discovered from the pipeline and the flange gaskets. The pipeline leaks were due to fittings not being tightened enough. The pipeline leaks were addressed by retightening the fittings. However, the leaks from the flange gaskets were due to the Garlock® gaskets. Garlok® is a hard polymer that does not easily conform to the shape of the PVC flanges. Additional torqueing of the flange bolts was required to eliminate these leaks. As a result of the increased torque, some of the flanges cracked. The flanges that cracked were then replaced with more durably designed PVC flanges.

FIU ordered and received sheets of both EPDM and Garlock® material as well as an ASTM standard die to cut the sheets into standard ASTM coupon shaped samples. These samples will be used to evaluate changes in the material properties due to the aging. Lastly, engineers from Riverbend and WRPS provided blowout test procedures they have used for their HIHTLs. We have begun the review process and will develop our blowout test procedure based on the Riverbend document and ASTM standards.

Milestones and Deliverables

The milestones and deliverables for Project 1 for FIU Year 6 are shown on the following table. Milestone 2015-P1-M19.2.1, the test loop set up for the evaluation of nonmetallic components in the waste transfer system, was completed on November 20, 2015. In addition, draft papers based on project research were submitted to Waste Management 2016. Milestone 2015-P1-M18.3.1, completion of a test plan for temperature measurements using IR sensors was completed on December 18, 2015. The milestone 2015-P1-M18.1.1 and the corresponding deliverable, also due on December 18, has been delayed. Additional time is needed to confer with the client and determine the extent of testing needed.

FIU Year 6 Milestones and Deliverables for Project 1

Task	Milestone/ Deliverable	Description	Due Date	Status	OSTI
Task 17: Advanced Topics for Mixing Processes	2015-P1-M17.1.2	Complete validation of impingement correlations	05/6/2016	On Target	
	Deliverable	Draft Summary Report for Subtask 17.1.1	08/28/2016	On Target	OSTI
	Deliverable	Draft Summary Report for Subtask 17.1.2	05/6/2016	On Target	OSTI
Task 18: Technology	2015-P1-M18.1.1	Complete test plan for evaluating SLIM's ability to detect a precursor of DSGREs	12/18/2015	Reforecasted TBD	

Development and Instrumentation Evaluation	Deliverable	Draft Test Plan for Subtask 18.1.1	12/18/2015	Reforecasted TBD	OSTI
	2015-P1-M18.3.1	Complete test plan for temperature measurements using IR sensors	12/18/2015	Complete	
	2015-P1-M18.2.1	Finalize the design and construction of the refractory pad inspection tool	02/26/2016	On Target	
	2015-P1-M18.2.2	Complete engineering scale mock-up testing	08/28/2016	On Target	
	Deliverable	Draft Summary Report for Subtask 18.2.1 and 18.2.2	08/28/2016	On Target	OSTI
	2015-P1-M18.2.3	Finalize the design and construction of the air supply line inspection tool	02/26/2016	On Target	
	Deliverable	Draft Summary Report for Subtask 18.2.3	02/26/2016	On Target	OSTI
	Deliverable	Draft Summary Report for Subtask 18.3.1	07/29/2016	On Target	OSTI
Task 19: Pipeline Integrity and Analysis	2015-P1-M19.2.1	Complete test loop set up	11/20/2015	Complete	
	2015-P1-M19.1.1	Evaluate and down select alternative UT systems for bench scale testing	03/11/2016	On Target	
	Deliverable	Draft Summary document for Subtask 19.1.1	03/11/2016	On Target	OSTI
	2015-P1-M19.2.2	Complete baseline experimental testing	03/25/2016	On Target	
	Deliverable	Draft Summary Report for Subtask 19.2.2	04/8/2016	On Target	OSTI

Work Plan for Next Quarter

- Task 17:
  - FIU will continue to conduct QDNS simulations in three dimensional periodic domains with reduced time step size and Courant number in order to obtain improved results in terms of velocity profiles and Q-criterion for different regimes of the flow. Once completed, implementation of the Shear Rate Correction (SRC) method from Gavrilov et al. (2011) in the QDNS method will be examined.
  - FIU plans to move forward by obtaining geometrical agreement as compared to the PJM within the simulation by implementing a curved impingement surface which accurately depicts the curvature seen in the PJMs. The results will be evaluated and compared to Poreh's correlation in order to address if and when these correlations are valid within the PJM process.
- Task 18:
  - The test plan for the 3D sonar will be submitted to WRPS in January and FIU will incorporate any suggestions and comments and execute the test plan in the next

quarter. The testing involves imaging and quantifying the volume of waste as an indicator of gas retention in sludge. FIU will work with WRPS during the next quarter to identify additional tests they would like FIU to perform and to develop another test plan for the April – June period. The testing of the sonar for measuring settling rates of surrogate HLW and the direct imaging of bubbles are areas in which WRPS has shown great interest.

- For the rover, FIU will finalize the tether design and incorporate the new control system. Additional efforts will focus on development of a model to navigate to the center of the tank and secondary systems to reduce contact of the tether with the refractory corners during turns. FIU will also work with WRPS engineers to design and develop an engineering scale mock-up of the refractory air channels for further testing..

- For the pneumatic pipe crawler, FIU will continue the development of the crawler and perform various tests to ensure that the unit is robust. Durability tests of the 3D printed parts will be conducted with the objective of evaluating the effectiveness of the abs strengthening techniques. Additionally, we will initiate the development of a full-scale mock up test to ensure the system can meet the objectives of the task.

- An initial test plan has been developed for the temperature measurement on the outside of the inner wall of DSTs. Based on the test plan, the Raytek IR sensor will be procured, a testing tank will be fabricated and the experimental test bed will be set up. Additionally, initial tests will be conducted on the carbon steel material (tank) at various set points. Thermocouples will also be installed for accuracy.

Task 19:

- FIU will continue to evaluate the potential ultrasonic sensor systems for 2” and 3” pipelines and down select a couple of them. The integrated system would include the UT sensors and the corresponding data acquisition system for thickness measurements. Additionally, budget based choices will be made for leasing some of the UT integrated sensor systems available in the market for testing. Semi-permanent mounting system designs for the UT sensors will also be investigated.
- For the non-metallic materials task, efforts during the next quarter will include finalizing the shakedown tests on the test loop as well as the initial baseline tests. These tests will include baseline burst tests of the HIHTL coupons as well as baseline material properties tests on both the Garlock and the EPDM samples. Aging of the specimens will also commence.

Project 2
Environmental Remediation Science and Technology

Project Description

This project will be conducted in close collaboration between FIU, Hanford Site, SRS, and LANL scientists in order to plan and execute research that supports the resolution of critical science and engineering needs, leading to a better understanding of the long-term behavior of contaminants in the subsurface. Research involves novel analytical methods and microscopy techniques for characterization of various mineral and microbial samples. Tasks include studies which predict the behavior and fate of radionuclides that can potentially contaminate the groundwater system in the Hanford Site 200 Area; laboratory batch and column experiments, which provide relevant data for modeling of the migration and distribution of natural organic matter injected into subsurface systems in the SRS F/H Area; laboratory experiments investigating the behavior of the actinide elements in high ionic strength systems relevant to the Waste Isolation Pilot Plant; surface water modeling of Tims Branch at SRS supported by the application of GIS technology for storage and geoprocessing of spatial and temporal data; and support for the DOE EM student challenge.

The following tasks are included in FIU Year 6:

Task No	Task
Task 1: Remediation Research and Technical Support for the Hanford Site	
Subtask 1.1	Sequestering uranium at the Hanford 200 Area vadose zone by in situ subsurface pH manipulation using NH_3 gas
Subtask 1.2	Investigation of microbial-meta-autunite interactions - effect of bicarbonate and calcium ions
Subtask 1.3	Evaluation of ammonia fate and biological contributions during and after ammonia injection for uranium treatment
Task 2: Remediation Research and Technical Support for Savannah River Site	
Subtask 2.1	FIU’s support for groundwater remediation at SRS F/H Area
Subtask 2.2	Monitoring of U(VI) bioreduction after ARCADIS demonstration at the SRS F-Area
Subtask 2.3	Humic acid batch sorption experiments into the SRS soil
Subtask 2.4	The synergetic effect of HA and Si on the removal of U(VI)
Subtask 2.5	Investigation of the migration and distribution of natural organic matter injected into subsurface systems
Task 3: Surface Water Modeling of Tims Branch	
Subtask.3.1	Modeling of surface water and sediment transport in the Tims Branch ecosystem
Subtask 3.2	Application of GIS technologies for hydrological modeling support
Subtask 3.3	Biota, biofilm, water and sediment sampling in Tims Branch

Task 4: Sustainability Plan for the A/M Area Groundwater Remediation System	
Subtask 4.1	Sustainable Remediation Analysis of the M1 Air Stripper
Subtask 4.2	Sustainable Remediation Support to DOE EM Student Challenge
Task 5: Remediation Research and Technical Support for WIPP	

Task 1: Remediation Research and Technical Support for the Hanford Site

Task 1 Overview

The radioactive contamination at the Hanford Site created plumes that threaten groundwater quality due to potential downward migration through the unsaturated vadose zone. FIU is supporting basic research into the sequestration of radionuclides such as uranium in the vadose zone, which is more cost effective than groundwater remediation. One technology under consideration to control U(VI) mobility in the Hanford vadose zone is a manipulation of sediment pH via ammonia gas injection to create alkaline conditions in the uranium-contaminated sediment. Another technology need for the ammonia remediation method is to investigate the potential biological and physical mechanisms associated with the fate of ammonia after injection into the unsaturated subsurface.

Task 1 Quarterly Progress – October 1 – December 31, 2015

Subtask 1.1. Sequestering Uranium at the Hanford 200 Area Vadose Zone by In Situ Subsurface pH Manipulation Using NH_3 Gas

FIU drafted and submitted a paper for the Waste Management Symposia to be held on March 6-10, 2016 in Phoenix, AZ. The paper was accepted for presentation at the conference.

> **Abstract**: 16600 – Characterization of U(VI)-Bearing Precipitates Produced by Ammonia Gas Injection Technology
> **Session**: 081 - Complex Site Characterization and Remediation Technologies
> **Date**: Wednesday, March 09, 2016
> **Time**: 8:30 AM - 12:00 PM

During this performance period, FIU continued isopiestic measurements of U-bearing samples. Two standards (sodium chloride and calcium chloride) are being using to evaluate the water activity and osmotic coefficients of the samples. The last calculations, based on sample weight once the system reached equilibrium, were based on the parameters of $CaCl_2$. The molality of the sodium chloride standard was reduced, which helped to determine its osmotic coefficient values from the literature. The comparison between water activity values using $CaCl_2$ and NaCl showed that they differed by approximately 17.9%. The difference was attributed to the rusty spots on the bottom of the nickel crucible. This crucible was removed from the isopiestic system and the standard was re-prepared in another crucible for the next set of measurements once the system reached equilibrium. During October, the isopiestic chamber was opened two times to weigh the samples when the system reached equilibrium and to calculate the values for osmotic coefficient and water activity. The percent of water lost during the weighing process stayed in the same range of 3.0-3.8%, which is similar to what was observed previously.

The last several calculations of molality of the sodium silicate samples prepared in duplicate showed big discrepancies between samples. These duplicate samples will be re-prepared, dried and exchanged with samples that are currently in the isopiestic chamber to check if molality values are similar.

Having completed the submission of the Environmental Molecular Sciences Laboratory (EMSL) proposal, sample preparation for the proposed experiments was started. This began with the modification of the calculations for sample solution preparation to meet the parameters of the experiment. Those changes include a wider range of test concentrations being used for optimization purposes during preliminary analysis. The samples are also being prepared in duplicate in order to evaluate the effect of a DI-water rinse step being considered for the procedure. Once sample preparation is complete, initial analysis will continue with preliminary SEM-EDS analysis of the dried precipitates.

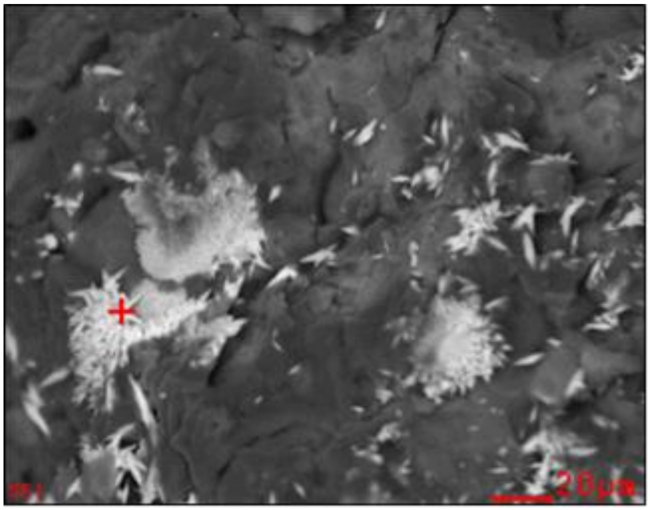
In November, a fresh sodium chloride standard was prepared in a new crucible due to the rusty spot found on the crucible wall. This was done to avoid errors in isopiestic measurements. Currently, the equilibration period is about 12-14 days before the isopiestic chamber is opened to weigh the samples. The discrepancies between molalities of the duplicate sodium silicate samples suggest that the system might need more time for equilibration. Measurements showed that humidity in the chamber is on the level of 67%, which is low to observe any deliquescence of samples.

FIU conducted XRD analysis of several samples composed of 500ppmU_ 50mMSi_5mMCa_5mMAl_50mMHCO$_3^-$ ("high bicarbonate") and 500ppm_50mMSi_5mM Ca_5mMAl_3mMHCO$_3^-$ ("low bicarbonate") missed in previous assessments. In a sample amended with high bicarbonate concentration, uranium solid phases were identified as cejkaite, agricolaite, and grimselite. In a sample amended with low bicarbonate concentration, the diffraction pattern produced a sufficient match with grimselite and soddyide. A near perfect match was found for nitratine ($NaNO_3$) and calcite for both samples.

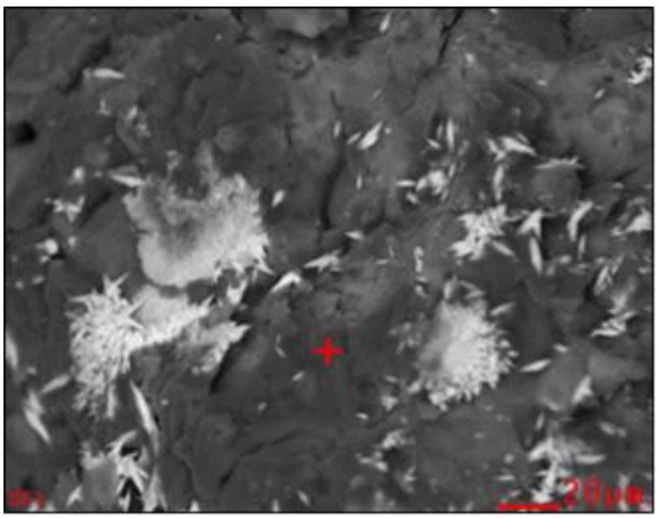
Samples prepared with "high" and "low" bicarbonate concentrations were also evaluated via SEM/EDS analysis (Figure 2-1). Atomic ratio calculations found good correlation between U:Na as 1:4 (correlates with XRD observations that U phases match cejkaite $Na_4(UO_2)(CO_3)_3$).

In addition, FIU was working on the speciation diagrams and updated GWB database with thermodynamic parameters for cejkaite. Work for the month of December included EDS mapping to see overlay of elements and to continue with speciation modeling.

The preparation for the latest batch of samples has continued with the regular monitoring of the solution pH after the injection of ammonia gas. Despite adding an opening to sample containers to allow exposure to air, the re-establishment of pH conditions was stagnant until the samples were gently agitated on the temperature controlled shaker. As the samples re-established equilibrium, the pH dropped from the post-treatment range of 11-12 towards a pre-treatment range of 8-9. From this point the samples will be vacuum-filtered and dried for scheduled SEM-EDS analysis. Additionally, the draft paper for the 2015 Waste Management Symposium oral presentation was completed and FIU is currently awaiting input from the reviewers.



Element	Wt%	At%
CK	08.37	15.71
NK	11.26	18.13
OK	33.43	47.11
NaK	14.54	14.26
AlK	00.27	00.22
SiK	00.73	00.59
ClK	00.55	00.35
KK	01.28	00.74
CaK	00.21	00.12
UL	29.38	02.78
Matrix	Correction	ZAF



Element	Wt%	At%
CK	06.65	10.25
NK	16.72	22.08
OK	36.20	41.84
NaK	20.18	16.23
AlK	00.34	00.24
SiK	05.17	03.41
ClK	08.24	04.30
KK	02.82	01.33
CaK	00.10	00.04
UL	03.57	00.28
Matrix	Correction	ZAF

Figure 2-1. SEM image of sample composed of 50mL Si, 5mMAl, 15mM Ca,50mM HCO_3

In December, measurements taken based on the calcium chloride standard indicated that the humidity level in the isopiestic chamber reached 77%. A change in slope appears indicative of the transition from a solid to a solid+ liquid system.

FIU also conducted geochemical equilibrium modeling via Geochemist's Workbench (GWB) 10.0 (Bethke, University of Illinois) software to predict the formation of uranium aqueous species and solid phases likely to be present as a result of NH_3 gas injections in the synthetic porewater solutions. Examples of speciation modeling for the Ca-free samples are presented in Figure 2-2 and Figure 2-3.

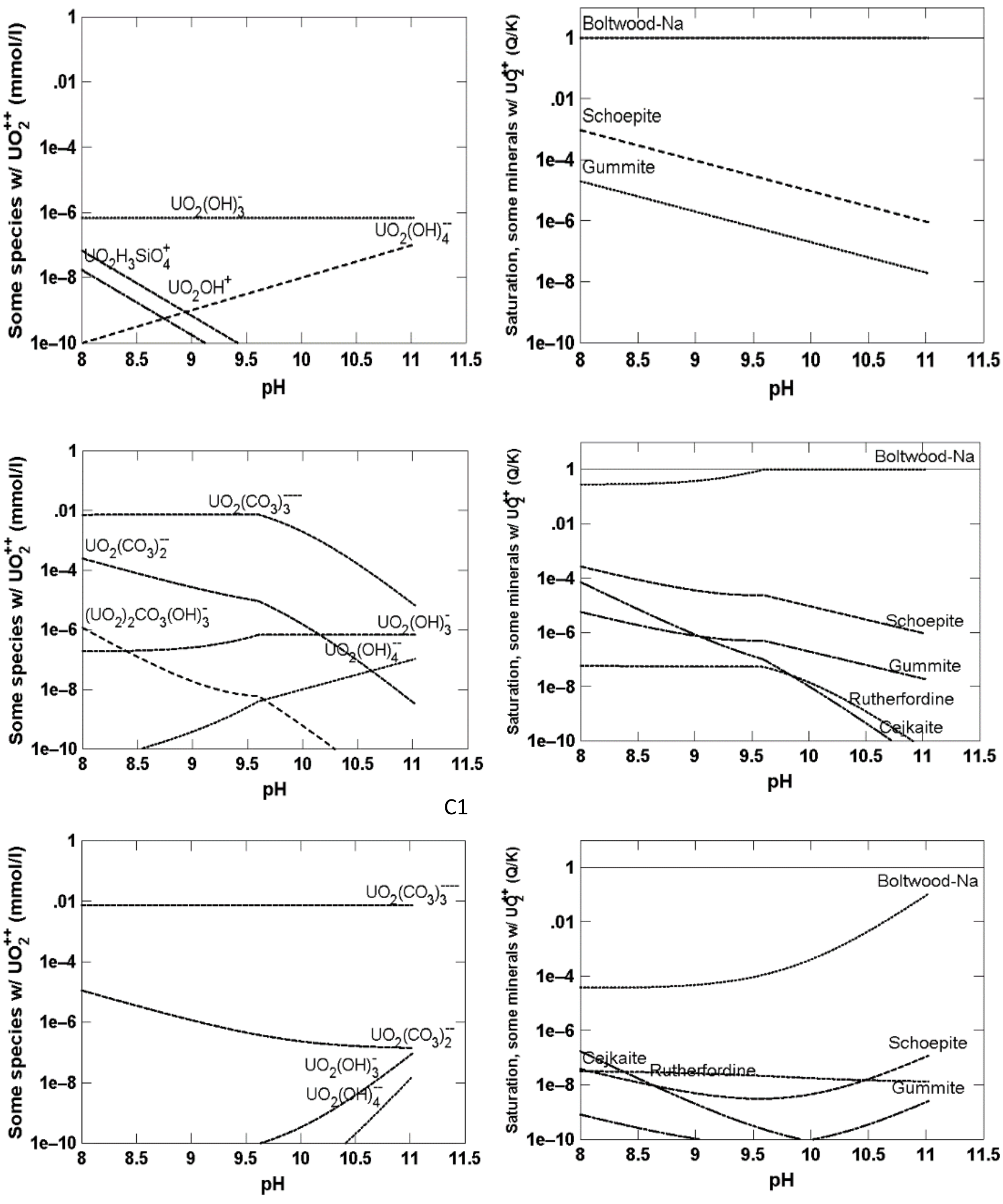


Figure 2-2. Diagrams of uranium aqueous species and saturation indices of some of uranium-bearing mineral phases plotted as a function of pH for 0.1% of NH_3 (0.063mol/L NH_3(aq)). Sample composition includes 50 mM of Si and varied HCO_3- concentrations. The first row shows diagrams for HCO_3—free samples (A1, A2), the 2nd and 3rd row show the diagrams for 2.9 mM (B1, B2) and 50 mM of HCO_3^- (C1, C2).

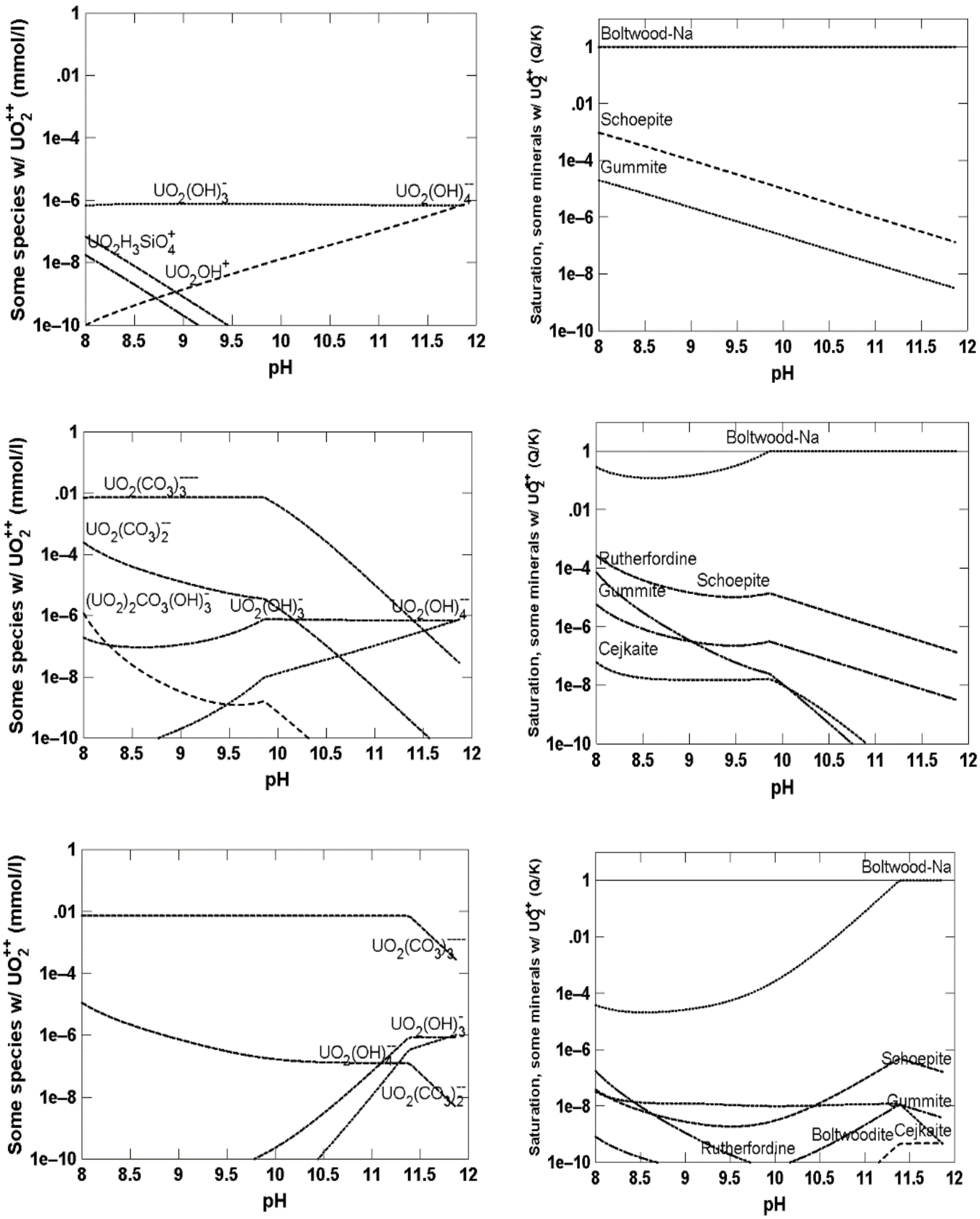


Figure 2-3. Diagrams of uranium aqueous species and saturation indices of some of uranium-bearing **mineral phases plotted as a function of pH for 5% of NH_3 (3.1 mol/L NH3(aq)). Sample composition includes 50 mM of Si and varied HCO_3- concentrations. The first row shows diagrams for HCO_3-free samples (A1, A2), the 2nd and 3rd row show the diagrams for 2.9 mM (B1, B2) and 50 mM of HCO3- (C1, C2).**

According to the speciation modeling, in bicarbonate–free synthetic solutions, $UO_2(OH)_3^-$ and $UO_2(OH)_4^{2-}$ were the predominant aqueous uranium species. In the presence of bicarbonate, aqueous uranium carbonates species, $UO_2(CO_3)_3^{4-}$ and $UO_2(CO_3)_2^{2-}$, dominated uranium speciation at both 0.1% (0.063 mol/L $NH_3(aq)$ and 5% (3.1 mol/L $NH_3(aq)$ of NH_3. However, the concentration of uranyl carbonates species was noted to decrease above pH 9.5; the decrease of $UO_2(CO_3)_3^{4-}$ and $UO_2(CO_3)_2^{2-}$was more pronounced at 2.9 mM HCO_3^- when using 5% of NH_3 (Figure 2-3-B1) compared to 0.1% of NH_3 (Figure 2-2-B1). At higher bicarbonate concentrations, the concentration of $UO_2(CO_3)_3^{4-}$ species was almost unchanged over a pH range from 8 to 11 in both ammonia gas concentrations but, at pH above 11.5, their concentrations were slightly decreased with treatment of 5% NH_3 (Figure 2-2-C1, Figure 2-3-C1). The modeling also predicted the formation of uranyl silicate Na-boltwoodite $[(Na)(UO_2)(HSiO_4) \cdot 0.5H_2O]$ and uranyl carbonate solid phases such as cejkaite $[Na_4 (UO_2)(CO_3)_3$ and rutherfordine $[UO_2(CO_3)]$ in addition to gummite $[(UO_2)_8O_2(OH)_{12}(H_2O)_{12}]$ and schoepite $[UO_2(OH)_3$ (beta)]. Na-boltwoodite, cejkaite, gummite, schoepite, and $[UO_2(OH)_3$ (beta)] were present in all of the conditions tested while rutherfordine was seen only in the presence of bicarbonate in the solution. The formation of uranyl hydroxide minerals, schoepite and $[UO_2(OH)_3$ (beta)] were favored under bicarbonate-free and low 2.9 mM bicarbonate concentrations; however, their saturation indices decreased as the concentration of bicarbonate ions increased for both 0.1% and 5% of NH_3. Overall, saturation indices values for Na-boltwoodite were found the highest for all of the conditions tested.

Sample preparation continued with the isolating of aqueous and solid sample phases for analysis after having seen the pH of treated solutions fall to the near pre-treatment range of 8-9. This was completed using a glass vacuum filtration setup paired with 0.22 µm nitrocellulose filters. All samples were filtered to collect the settled precipitates, though only duplicate samples were rinsed with 5 mL of deionized water. Both filtrate and rinse were collected independently for study. The filter paper and isolated solids were placed in containers and allowed to dry at 30 C° over 3 days.

Small specimens from the dried solid samples were isolated for scanning electron microscope analysis. They were mounted on aluminum studs using carbon tape and sputter coated with a thin layer of gold to minimize charging. SEM analysis of the dried solid samples was planned as a pre-screening method for selecting the samples that receive additional analysis. Samples showing areas of concentrated uranium content would be preferentially selected for analysis because of the improved chances of successful sample characterization. Preliminary evaluation of the results suggested that the major areas of uranium content were predominantly present in the high bicarbonate (50 mM) samples (Figure 2-4). Unlike previous samples, there were no obviously crystalline ornate structures that were high in uranium content. No samples which were rinsed showed areas of significant uranium content. Though additional information is required to form any conclusion, the likely reason for this difference is the dissolution of uranium forms with the rinsing step. The results of aqueous phase analysis of the rinse solutions will be integral in either supporting or countering this theory.

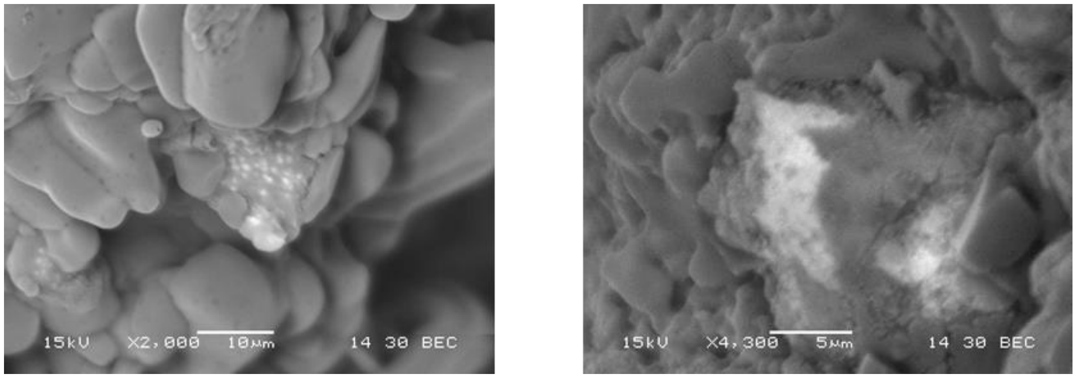


Figure 2-4. SEM imaging of high bicarbonate (50 mM) precipitate samples prepared with 0 mM (left) and 5 mM (right) of calcium.

The analysis of the 18 filtrates and 9 rinse solutions is currently under way. A kinetic phosphorescence analyzer will be used in order to determine the aqueous uranium content and evaluate how the variable constituents affected uranium retention in the aqueous phase. This will involve the digestion, resuspension, and dilution of the samples in nitric acid.

Subtask 1.2. Investigation on Microbial-Meta-Autunite Interactions - Effect of Bicarbonate and Calcium Ions

FIU drafted and submitted a paper for the Waste Management Symposia to be held on March 6-10, 2016 in Phoenix, AZ. The paper was accepted for presentation at the conference.

> **Abstract**: 16429 – The Effect of Bicarbonate on Autunite Dissolution in the Presence of Shewanella Oneidensis Under Oxygen Restricted Conditions
> **Session**: 010 – Groundwater Remediation Projects
> **Date**: Monday, March 07, 2016
> **Time**: 10:00 AM - 12:00 PM

For this reporting period, analysis for the samples with bicarbonate (HCO_3) amended media was completed. Bicarbonate free media results showed almost no change in the concentration of U(VI) between abiotic and biotic samples. For the samples with 3 and 10 mM HCO_3, the results are shown below in Figures 2-5 and 2-6.

In Figure 2-5, it is shown that controls for 3 mM HCO_3 had a slight decrease of U (VI) over time but, after inoculation with facultative bacteria, there was an increase in the released U (VI) measured in the aqueous phase. For abiotic samples in 10 mM HCO_3 (shown in Figure 2-6), U (VI) released in the initial days was high, close to 3000 pbb, but 10 days after beginning the experiments, dropped to below 1000 ppb and remained there until the end of the experiment. For biotic samples in 10 mM HCO_3, there was no reduction of U(VI) after the inoculation with facultative bacteria; instead, the release of U(VI) was higher than the controls, behaving similar to the pattern of 3 mM.

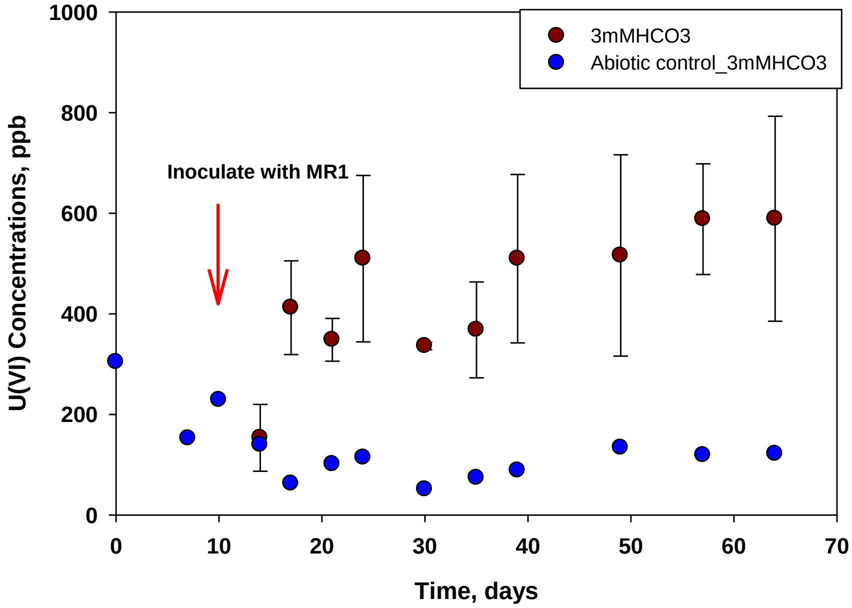


Figure 2-5. U(VI) concentration as a function of time for control (abiotic) samples and biotic samples for 3 mM of bicarbonate media HCO_3.

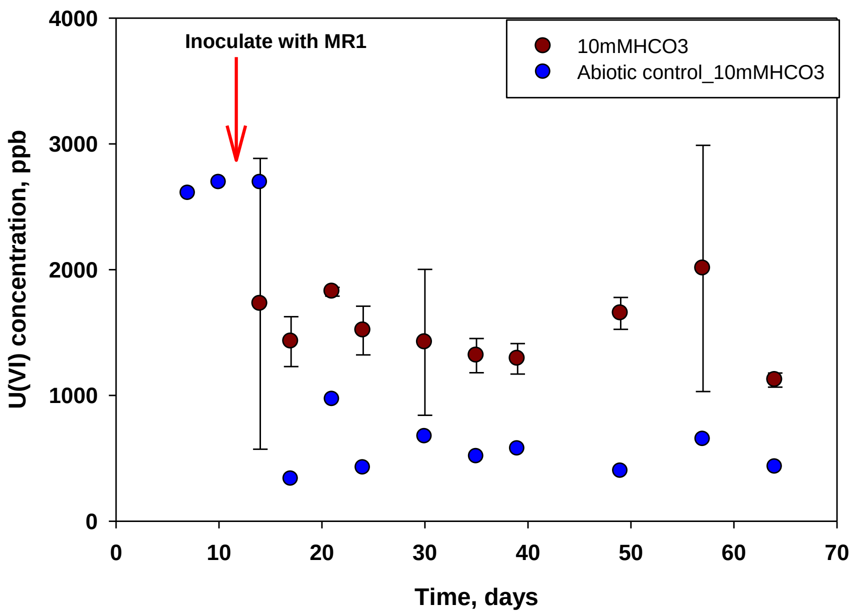


Figure 2-6. U(VI) concentration as a function of time for control (abiotic) samples and biotic samples for 10 mM bicarbonate media.

The next step of the experiment was to process the samples in the ICP instrument to measure calcium and phosphorous concentrations released in the aqueous phase as complimentary data for the chemical analysis.

In November 2015, FIU continued with the analysis of samples for Ca by means of ICP. Figure 2-7 presents the calcium content for the bicarbonate-free samples (controls and biotic). Results revealed a similar trend between biotic and control samples, where the average values for Ca in both cases are very similar. Statistical evaluation via Sigma plot suggested that there is not a statistically significant difference between the input groups (P = 0.466).

Figure 2-8 presents calcium content for samples amended with 3 mM bicarbonate. A similar trend was observed between biotic and abiotic samples that exhibited similar amounts of calcium. Nevertheless, the average Ca concentration for the samples amended with 3 mM bicarbonate was slightly higher (~40 ppm) compared to the one of the bicarbonate- free samples (~30 ppm). Statistical evaluation via Sigma plot suggested that there is not a statistically significant difference between the input groups (P = 0.063). The power of the performed test (0.352) is below the desired power of 0.800.

In the samples amended with 10 mM bicarbonate (Figure 2-9), all samples exhibited a higher concentration of calcium in comparison with bicarbonate-free samples and samples amended with 3 mM of bicarbonate. Statistical evaluation suggested a similar trend as for bicarobnate-free and samples amended with 3 mM HCO_3, showing that there is not a statistically significant difference between the input groups (P = 0.867). The power of the performed test (0.050) is below the desired power of 0.800.

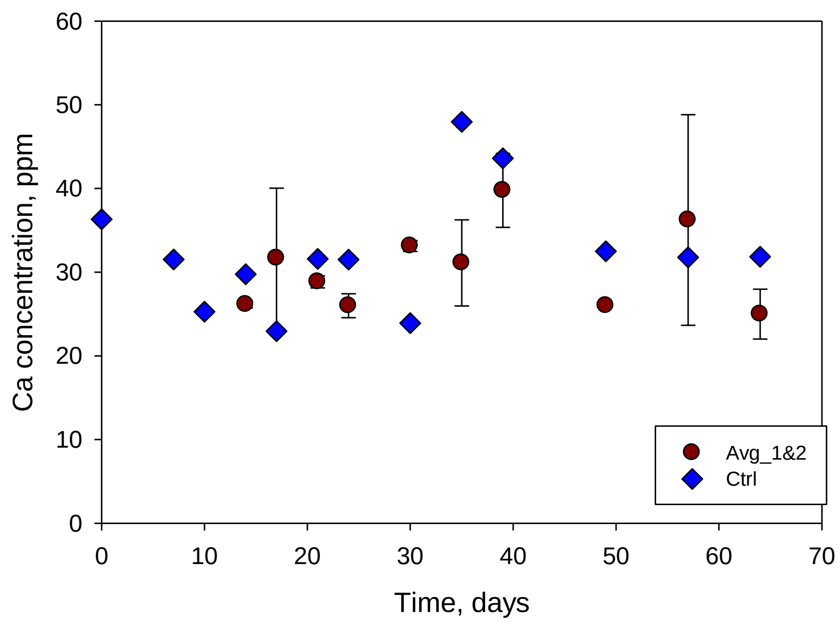


Figure 2-7. Calcium concentration as a function of time for control (abiotic) samples and biotic samples for bicarbonate- free media.

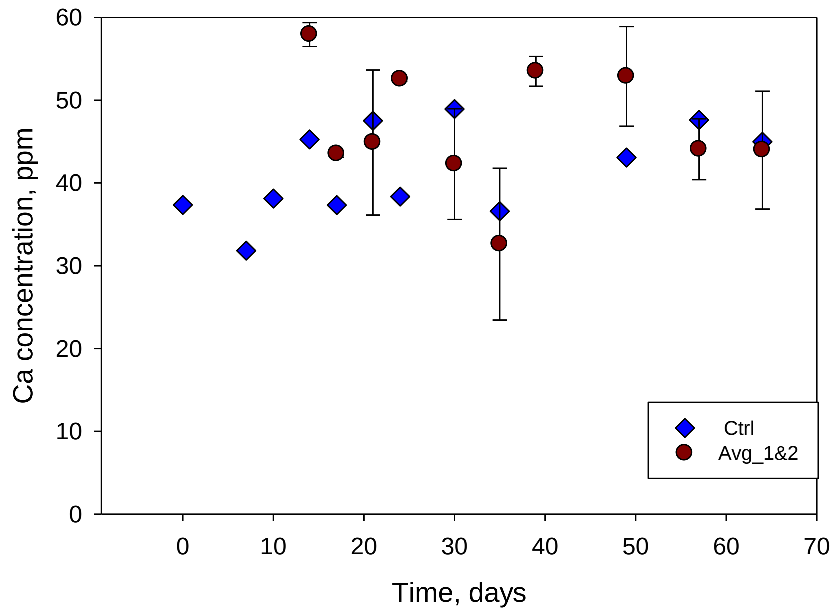


Figure 2-8. Calcium concentration as a function of time for control (abiotic) samples and biotic samples for samples amended with 3 mM bicarbonate.

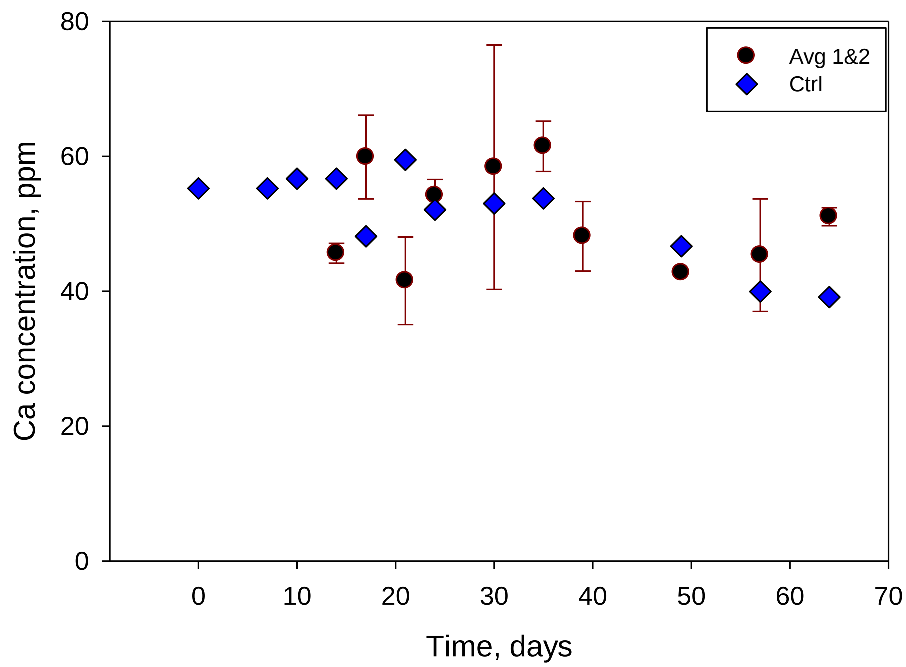


Figure 2-9. Calcium concentration as a function of time for control (abiotic) samples and biotic samples for samples amended with 10 mM of bicarbonate.

Phophorus data are in a progress for evaluation and reprocessing due to discrepancies yielding inconclusive data; sample results will be presented in the next report. In addition, FIU finalized the protein analysis of the microbial cells; this data analysis is in progress.

In December 2015, FIU conducted protein analysis of bacterial cell samples collected during the dissolution experiments. For cell protein determination, a Bicinchoninic Protocol (BCA, Pierce) protein analysis kit was used. The BCA protein assay is based on the highly selective colorimetric detection of the cuprous cation (Cu^{+}) by bicinchoninic acid as a result of the reduction of Cu^{2+} to Cu^{+} by proteins in an alkaline medium. Following the protocol procedures, the cells collected in 1.5 mL centrifuged tubes were lysed by boiling at 100 ^{o}C for 10 min and then cooled on ice. The addition of an alkaline medium followed and the samples were placed in a water bath (60 ^{o}C) for 60 minutes. A calibration curve was prepared by using albumin as a standard (Figure 2-10) and the absorbance was measured at 562 nm spectrophotometrically.

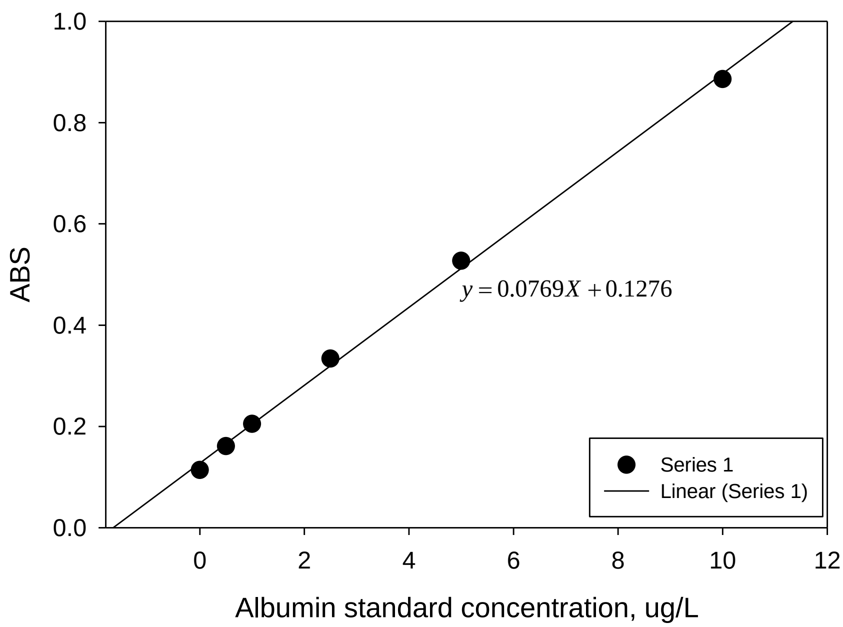


Figure 2-10. Calibration curve for protein analysis.

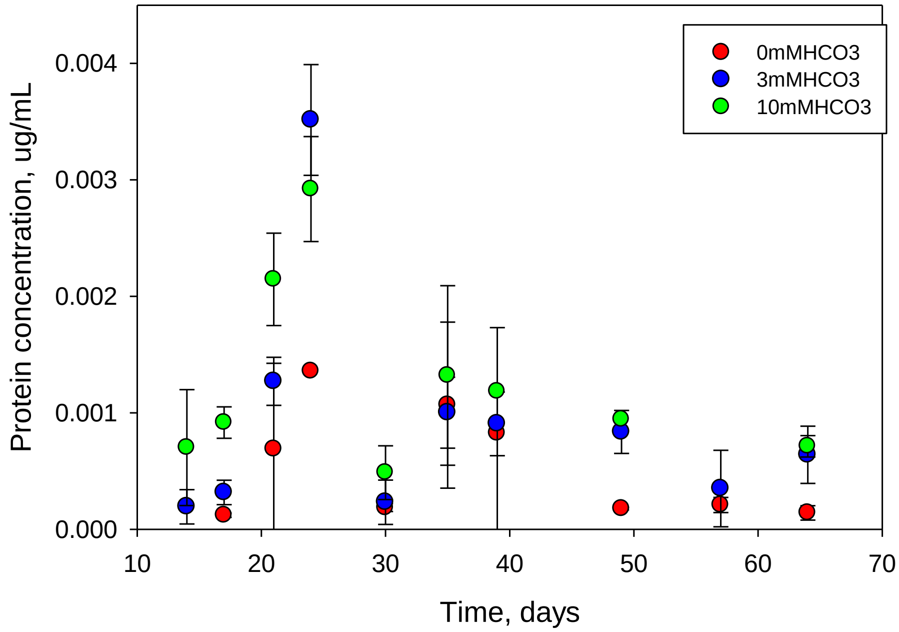
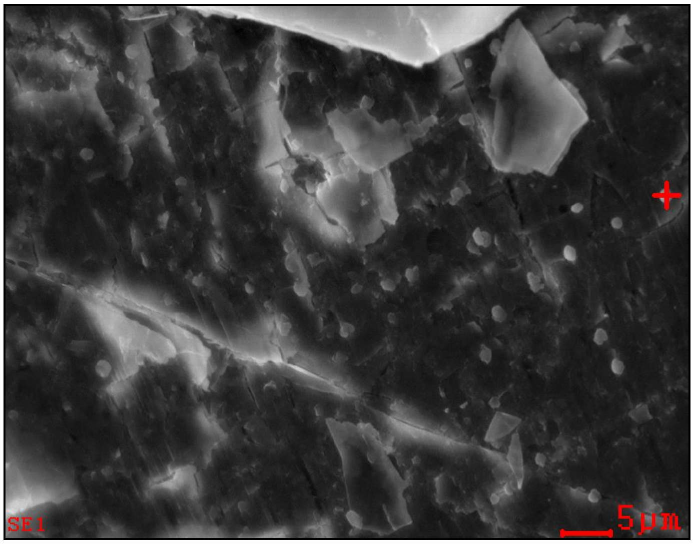


Figure 2-11. Protein content in bacterial cell samples collected from madia solutions amended with bicarbonate.

Cell density calculations and viability assessments are in progress to correlate with the cell protein content results. Preliminary analysis indicates that the cell protein content in *Shewanella oneidensis* MR1 for the samples where the media solution was augumented with 10 mM HCO_3 was the highest among the three bicarbonate concentrations tested (Figure 2-11).

FIU initiated SEM/EDS analysis on autunite particles collected from the sacrifical vials. SEM analysis of post-reacted autunite samples revealed larger fractures and cleavage planes in the biotic reactors. The formation of secondary minerals was identified on the surface of autunite. The atomic ratio calculations based on molar quantities of elements determined via EDS analysis suggested formation of uraniul phosphates and uranyl carbonate phases precipitated on the surface of autunite (Figure 2-12). However, SEM analysis haven't revealed the formation of biofilm on the autunite surface. SEM/EDS analysis is still in progress.



Element	Wt%	At%
CK	06.22	14.63
NK	09.87	19.91
OK	28.16	49.76
NaK	00.65	00.80
PK	05.92	05.40
UM	42.96	05.10
CaK	06.23	04.39
Matrix	Correction	ZAF

Figure 2-12. Secondary minerals precipitated on the autunite surface.

Subtask 1.3. Evaluation of Ammonia Fate and Biological Contributions During and After Ammonia Injection for Uranium Treatment

<u>Subtask 1.3.1: Investigation of NH_3 partitioning in relevant Hanford minerals and synthetic porewater</u>

Preliminary batch sorption experiments started in the month of September were finalized during the month of October.

Triplicate samples were prepared with Ottawa sand (SiO_2) at ~100 g/L, 500 ppb U and 0.008 M NaCl or synthetic porewater with pH adjustment by either 0.025 M NaOH (with 2.5 M NaCl to maintain constant ionic strength) or 2.5 M NH_4OH on ICP.

Results during October showed that there was some silica dissolution at pH ~11.7 in 0.008 M NaCl as shown in Table 2-1. Negligible dissolution of silica occurred at pH 7.5 based on a detection limit of 705 ppb. Further, silica dissolution appears to be solely an effect of pH as pH adjustment with NaOH versus NH_4OH did not produce statistically significant different results. Table 2-1 contains a summary of the calculated results.

Table 2-1. Si Dissolution for NaOH vs. NH_4OH

	Si Dissolution	pH
NaOH	1.49±0.05%	11.69±0.07
NH_4OH	1.83±0.5%	11.66±0.08

Equilibrium measurements for the aqueous U fraction at pH 7.5 and after adjusting pH to 11.7 by NH_4OH are shown in Figure 2-13 below. Results for uranium were obtained by KPA analysis following acidification in 1% HNO_3. As in Si dissolution ICP results, KPA analysis also followed the expected trend. Figure 2-13 shows desorption with increasing pH. However, the triplicate fraction aqueous (U concentration at equilibrium /U concentration initial) results are scattered. Our hypothesis is that this discrepancy could arise from inaccurate initial start conditions (i.e. inaccurate addition of the uranium stock to batch samples) as discussed in the Lessons Learned for October. Source error is due to either: (a) inaccurate pipetting or (b) weighing error on the analytical scale.

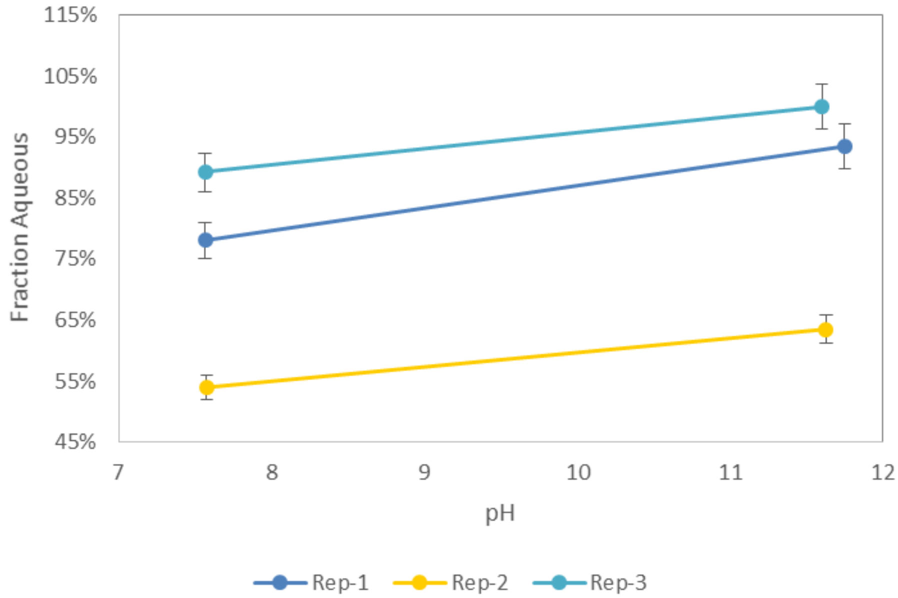


Figure 2-13. Uranium sorption to Ottawa sand (100 g/L) at pH 7.5 with pH adjustment to 11.5 by NH_4OH following equilibration at pH 7.5 and initial ionic strength of 0.008 M NaCl.

Lessons Learned for October:

1. There are fluctuations with the analytical scale at small masses and significant error in micropipettors at small volumes. These are shown by the fluctuations in *triplicate* samples in the figure above. Care will be taken in future experiments to weigh samples during the addition of uranium stock (~ 20 µL) and may be diluted to allow for a more accurate addition by mass and volume.
2. NH_3 electrode readings are sensitive to pH. An ionic strength adjuster (ISA) is added to adjust pH to >11 to transform all NH_3/NH_4^+ species to NH_3 for the electrode to read the NH_3. Further, the ISA adjuster turns the sample blue if sufficient pH is reached. Several samples needed additional ISA to reach appropriate pH levels due to acidifcation for storage. In addition, ammonia off-gassing is a major possible source of inaccurate electrode readings for samples not analyzed immediately (within 15 minutes) after addition of the ISA. Therefore, future samples will be analyzed immediately after the addition of ISA (within 15 minutes).

During the month of November, batch experiments investigating the effects of ammonia on uranium mobility and mineral dissolution were continued. In addition, DOE Fellow Silvina DiPietro submitted an abstract to present in the student poster competition at the Waste Management Conference next year. Data is presented in this report for kaolinite and Ottawa sand with synthetic porewater. However, equivalent experiments with NaCl are ongoing. The following changes were made to batch experiments based on discussion with PNNL collaborators: (1) a synthetic porewater based on Hanford groundwater will be used for some batch experiments (presented in Table 2-2), (2) NaCl at similar ionic strength to synthetic porewater will be used for equivalent experiments for comparison, (3) washing procedures for minerals were included in preparation protocols (summarized in Table 2-4).

Because minor changes were made to experimental protocols, the experiments are summarized here. Prior to use, the minerals were washed following the procedures outlined in Table 2-4. Then, triplicate samples were prepared with Ottawa sand (SiO_2) at ~100 g/L or kaolinite at ~5 g/L, 500 ppb U and NaCl or synthetic porewater. Samples were equlibrated at pH ~7.5 prior to addition of uranium. Following the addition of uranium, the samples were equilibrated for ~3 days. Then, the pH was adjusted to ~11.5 by either 0.025 M NaOH (with 2.5 M $NaClO_4$ or NaCl to maintain constant ionic strength) or 2.5 M NH_4OH (Note: the NaOH concentration was estimated to have a similar base strength to the 2.5 M NH_4OH and the background electrolyte was added to maintain similar ionic strength between the two).

To reduce losses of ammonia to the atmosphere, an aliquot of 2.5 M NH_4OH was added to raise the pH to ~11.5, mixed for 30 minutes, checked for pH, and then capped and covered with parafilm until sampling three days later. The samples that were pH adjusted with NH_4OH were not checked or adjusted for pH during the equilibration period. The samples adjusted by NaOH were then adjusted to the pH measured within the NH_4OH samples. Sampling was completed after three days at pH ~7.5 and then after three days at pH ~11.5. During sampling, an aliquot (well-mixed, assumed homogenous) was removed and centrifuged for 30 minutes at 4500 rpm to remove particles >250 nm (as approximated by Stoke's law). The aqueous phase was then removed for analysis (1% HNO_3 for KPA for U and ICP-OES for Si/Al and 0.2 M H_2SO_4 for ammonia electrode).

Table 2-2. Synthetic Porewater Composition (based on correspondence with Dr. Szecsody) with Total Ionic Strength of ~7.2 mM

Element	(mmol/L)
Na^+	1.1
K^+	0.22
Ca^{2+}	1.4
Mg^{2+}	0.6
HCO_3^-	1.32
Cl^-	3.9

After three days of equilibration at pH 7.57±0.07, the fraction of uranium remaining in the aqueous phase was 49.9±2.7% for quartz (100 g/L) and 89.1±2.7% for kaolinite (5 g/L). This results in a partitioning coefficient of 10.96±1.25 mL/g for quartz and 25.3±6.9 mL/g for kaolinite. These are slightly higher than those measured previously on Hanford sediments at pH ~8 (0.11 – 4 mL/g) (Zachara, Brown et al. 2007, Szecsody, Truex et al. 2013). However, it is likely that there is a greater availabilty of surface area in the batch experiments with pure minerals as compared to the natural sediments.

It must be noted that the ionic strength in these experiments is not constant due to the significant increase in ions during pH adjustment and to dissolution of minerals. The ionic strength for Ottawa sand samples at pH 7.61±0.07 was 7.29±0.28 mM based on the six samples and accounting for the initial ionic strength of the synthetic porewater and additional ionic strength added through pH adjustment. However, the ionic strength increases to 0.53±0.12 M after pH adjustment to 11.68±0.06 (based only on the ions added to the solution for pH adjustment). Similar ionic strength changes were noted in the kaolinite experiments with pH 7.53±0.05 with ionic strength of 8.20±0.15 and increasing to 0.57±0.12 at pH 11.78±0.03. While this is a significant increase in ionic strength, it is likely applicable to the field as Szecsody et al. measured ~0.28M ionic strength 24 hours after treatment of unsaturated columns with 5% NH_3 gas (based on ions reported) (Szecsody, Truex et al. 2012).

ICP-OES analysis shows a significant decrease in aqueous Al in all kaolinite samples at pH ~11.5 with all but one sample below detection limits (LOD 16.7 ppb). However, the aqueous silicon decreases below detection limits for samples treated with NH_4OH (LOD 71 ppb) but increases by approximately an order of magnitude for samples treated with NaOH. It is possible that the Al and Si concentrations reached above solubility and precipitated in the NH_4OH treated samples, but this is still being investigated further with speciation modeling. The aqueous concentration of silicon and percent dissolved from the mineral phase is summarized for samples treated with NaOH in Table 2-3 below. It is notable that greater dissolution of the kaolinite occurred than the quartz (based on aqueous Si) and this is consistent with their respective solubilities.

Throughout these experiments, care was taken to reduce losses of NH_3(g) through volatilization for the samples that were pH adjusted with NH_4OH by minimizing the opening of vials and wrapping with parafilm. Based on ammonia measurements by the electrode and assuming minimal losses to the gas phase, greater sorption of NH_4^+ occurred in the kaolinite clay (23.9±1.7% for kaolinite versus 15.6±1.8% sorbed for quartz resulting in K_d's of 22±3 and 67.7±5 mL/g, respectively). However, during future experiments, a blank sample without solids will be carried throughout the experiments to allow for an estimation of losses to the gas phase.

After equilibration of samples at pH 11.73±0.07, a significant decrease in aqueous uranium is observed for kaolinite (89.1±2.7% at pH 7.53±0.05 decreased to 2.5±0.4% for NH_4OH treatment and 11.8±3.6% for NaOH treatments, respectively) and quartz (49.9±2.7% at pH 7.61±0.07 decreased to 9.1±1.3% for NH_4OH and 11.1±2.6% for NaOH treatments, respectively). Figure 2-14 depicts the aqueous fraction of uranium at each pH for each of the minerals and treatments. It should be noted that aqueous U for the quartz (Ottawa sand) samples are not significantly different with respect to the NaOH or NH_4OH treatment. However, the kaolinite clay is significantly different with respect to treatment likely due to ion exchange of NH_4^+ with kaolinite. Because the NH_4OH appears to lead to a decrease in uranium in the aqueous phase, it is possible that ternary complexes are forming with kaolinite, NH_4^+, and U or co-precipitating with Al and Si. Yet, it may be ruled out that it is a co-precipitation process as quartz samples exhibited a similar decrease in Si with NH_4OH treatment as discussed. Figure 2-15 shows the K_d partitioning coefficients for elevated treatments for both minerals and treatments. Although further investigation is warranted into the interactions occurring in these samples, these results are promising as to ammonia injection as a remediation technology.

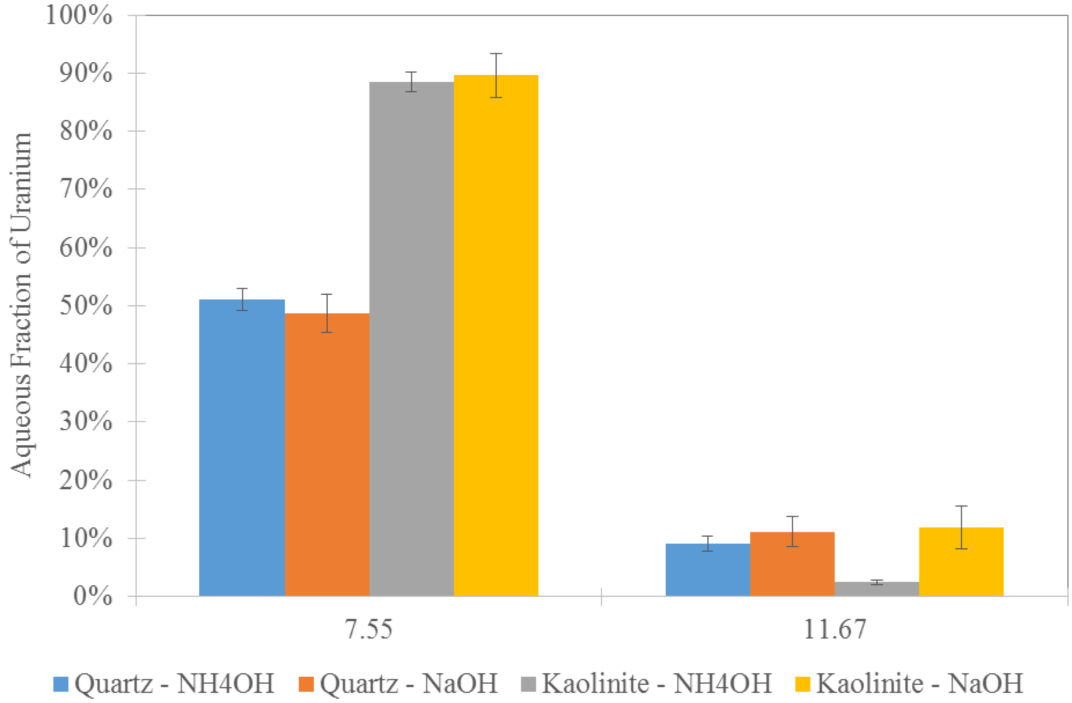


Figure 2-14. Aqueous fraction of U (initially 500 ppb) with respect to pH for 5 g/L kaolinite and 100 g/L quartz (Ottawa sand) suspensions in synthetic porewater with pH adjusted up with either 2.5 M NH_4OH or 0.025 M NaOH – 2.5 M $NaClO_4$ with error bars based on triplicate measurements

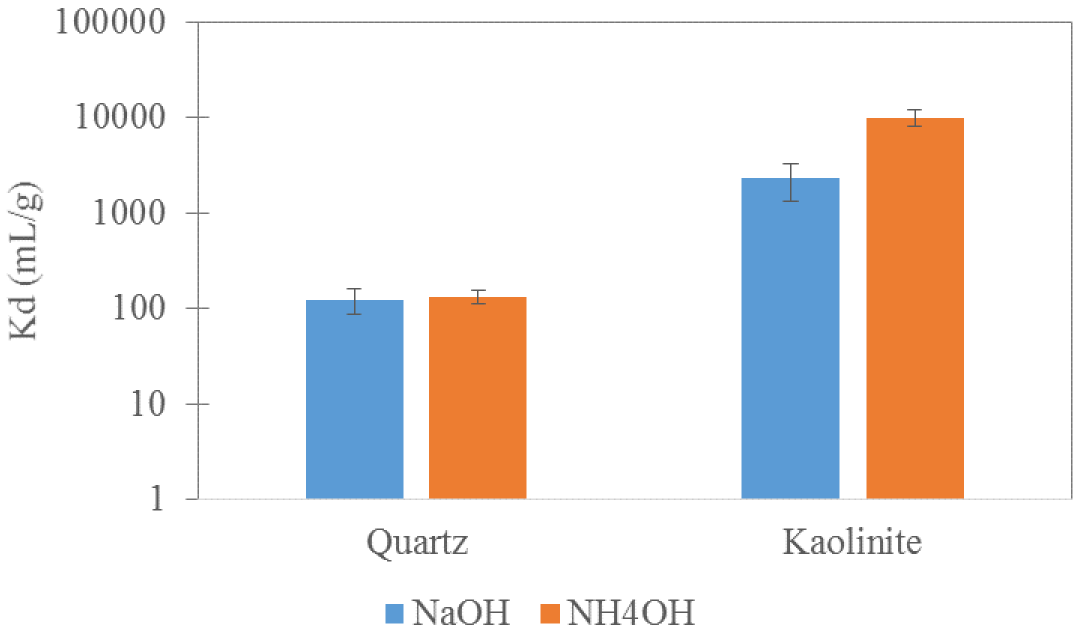


Figure 2-15. Equilibrium partitioning coefficients (K_d, mL/g) for U (initially 500 ppb) with respect to pH for 5 g/L kaolinite and 100 g/L quartz (Ottawa sand) suspensions in synthetic porewater with pH adjusted up with either 2.5 M NH_4OH or 0.025 M NaOH – 2.5 M $NaClO_4$ with error bars based on triplicate measurements.

Table 2-3. Summary of Aqueous Si Measurements in Quartz (100 g/L) and Kaolinite (5 g/L) Batch Experiments Treated with NaOH to Increase pH with Error Based on Triplicate Measurements

	pH		Si (ppb)		pH		Si (ppb)		Si (% diss)	
Quartz	7.61	±0.07	3809	±428	11.68	±0.09	12619	±924	0.039%	±0.003%
Kaolinite	7.53	±0.05	1572	±310	11.78	±0.04	9002	±1421	1.16%	±0.19%

Lessons Learned for November:

1. During the month of November, all filters were replaced for the Barnstead Diamond RO and Barnstead Nanopure water purification units. This has led to a significant change in the conductivity of our source water and led to significant changes to buffering capacity when adjusting pH. All future experiments will utilize deionized water (≥18 MΩ). This will allow for better reproducability of data and likely partially explains the inconsistencies in the data reported in October.
2. In addition, future experiments will include a blank without a mineral phase to carry through to estimate ammonia losses to the gas phase.

Table 2-4. Summary of Mineral Washing Methods

Mineral	Method	Reference
Quartz (Ottawa Sand)	(1) Mix 100 g/L suspension in 0.01 M NaOH for 60 minutes, (2) Centrifuge, decant, replace liquid with 0.01 M HCl, mix 60 minutes, (3) Centrifuge, decant, replace with Nanopure (>18 MΩ) H_2O and mix 3 minutes, (4) Repeat step three two more times, (5) Dry solid at 35°C for ~3 days, (6) Lightly crush with a mortar and pestle to homogenize	(Powell, Kersting et al. 2008, Zavarin, Powell et al. 2012, Boggs, Dai et al. 2015)
Montmorillonite	(1) Mix 100 g/L suspension in 0.001 M HCl for 30 minutes, (2) Add 0.5 mL H_2O_2 and mix an additional 30 minutes, (3) Centrifuge 6 hours at 4500 rpm, decant aqueous and replace with 0.01 M NaCl (or synthetic porewater for synthetic porewater experiments) and mix overnight, (4) Repeat four times, (5) Centrifuge, decant and replace with Nanopure H_2O, (6) Repeat at least four times (until excess ions are removed), (7) Dry solid at 35°C for ~3 days, (8) Lightly crush with a mortar and pestle to homogenize	(Powell, Kersting et al. 2008, Zavarin, Powell et al. 2012, Boggs, Dai et al. 2015)
Kaolinite	(1) Mix 100 g/L suspension in 1 M NaCl (synthetic pore water for synthetic porewater experiments) for 30 minutes, (2) Centrifuge, decant and repeat four more times, (3) Centrifuge, decant and replace with Nanopure H_2O, (4) Repeat four more times, (5) Dry solid at 35°C for ~3 days, (6) Lightly crush with a mortar and pestle to homogenize	(Heidmann, Christl et al. 2005, Heidmann, Christl et al. 2005)
Illite	(1) Mix 100 g/L suspension with 1 M NaCl (or synthetic porewater) for three hours and allow to flocculate overnight, (2) Decant and replace with 1 M NaCl (or synthetic porewater) and mix, (3) Repeat two more times, (4) Decant and replace with Nanopure H_2O, (5) Repeat until excess ions are removed, (6) Dry solid at 35°C for ~3 days, (7) Lightly crush with a mortar and pestle to homogenize	(Baeyens and Bradbury 2004)

During the month of December, batch experiments continued to investigate the effects of pH increase by either NaOH or NH_4OH on uranium sorption and kaolinite mineral dissolution. Data is presented for both synthetic porewater and similar ionic strength in NaCl for samples equilibrated at pH 7.5 to mimic the natural groundwater at Hanford and the increased to pH 11.5 by either NaOH or NH_4OH. These batch samples follow the protocols outlined in the October and November 2015 monthly reports. Additional experiments have begun with synthetic porewater to investigate effects between pH 7.5 and 11.5.

NaOH vs. NH_4OH Treatment

Figures 2-16 and 2-17 show results from batch experiments for 5 g/L kaolinite and 500 ppb U in the presence of a synthetic porewater described in the November monthly report (total ionic strength 7.2 mM, Table 2-5). In the data presented, the pH of the batch samples is raised by either 2.5 M NH_4OH or 2.5 M $NaClO_4$ + 0.025 M NaOH. The $NaClO_4$ + NaCl solution is used to add a similar base power with equivalent ionic strength changes. In the presence of the synthetic porewater, the removal of U from the aqueous phase increases with pH as shown by the increase in K_d partitioning coefficients. Moreover, there is a drop in the K_d values between pH 10.5-11. After pH 11, the K_d values for the two treatments diverge.

The sorption of U is generally expected to decrease with increasing pH for systems open to the atmosphere as carbonate concentrations increase, complex with U in the aqueous phase and decrease sorption. However, a different phenomenon seems to be controlling this system, likely due to the reactions occurring with Na^+, K^+, Ca^{2+}, Mg^{2+}, HCO_3^- and Cl^- in the aqueous phase due to the synthetic porewater. Further, it is possible that additional interactions are occurring with the Al and Si that are dissolving from kaolinite as the pH increases. The current theory is that precipitation processes are occurring that are removing U from the system as the pH increases. However, this will be further investigated through aqueous speciation modeling and sequential extractions described below.

The reason for the drop in K_d values between pH 10.5-11 is not yet known. However, the divergence of the two treatments (NaOH vs. NH_4OH) show that there are some changes occurring in the system, perhaps due to the increasing ionic strength. It should be noted that the NH_4OH system is significantly increasing the ionic strength due to molecular species (NH_3) while it should have a different effect on the solubility of elements versus charged species (by Na^+ and OH^-).

Figure 2-17 shows the dissolution of kaolinite as the pH is increased by either NaOH or NH_4OH. Aluminum dissolution is low and fairly consistent throughout the pH range investigated without significant differences between the two treatments. However, Si dissolution appears to increase much more for the NaOH treatment as the pH is increased. Because Si is likely dissolving as silicic acid (H_4SiO_4), it is possible that the differences are again due to ionic strength dominated by molecular versus charged species. However, this is under further investigation through aqueous speciation modeling.

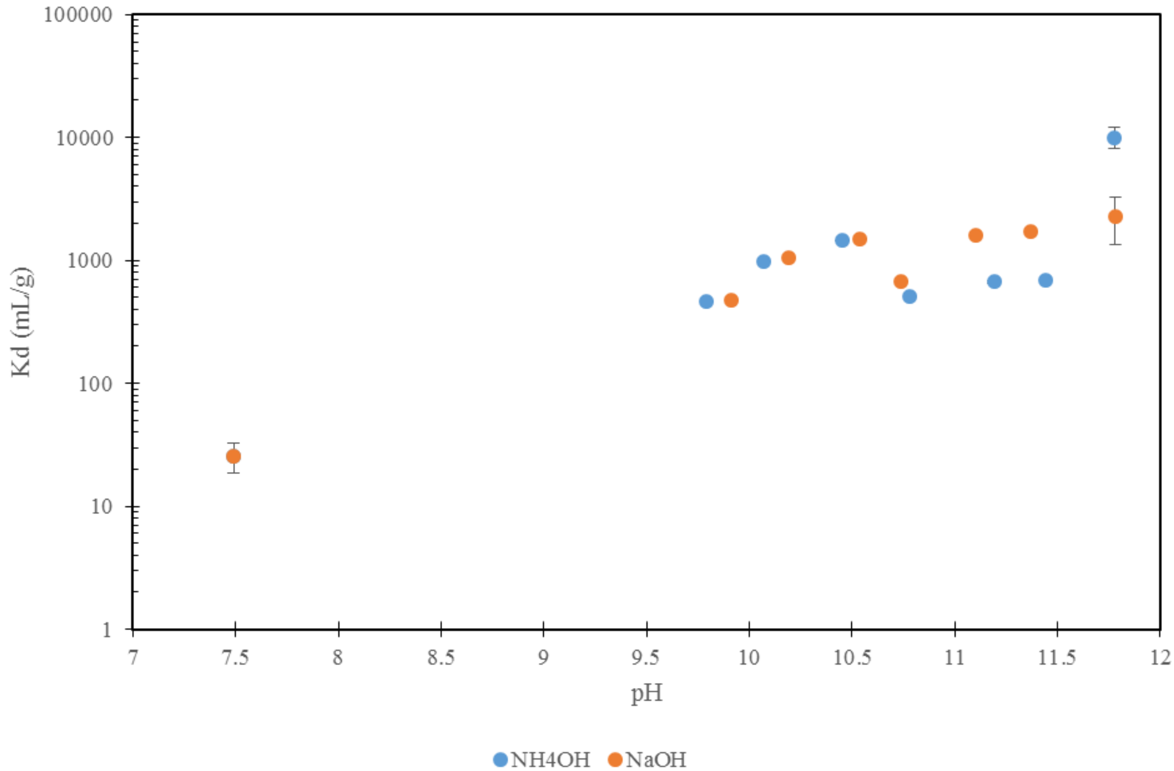


Figure 2-16. K_d (mL/g) for U (500 ppb) sorption to kaolinite (5 g/L) in synthetic porewater at variable pH via adjustment with either NaOH or NH_4OH.

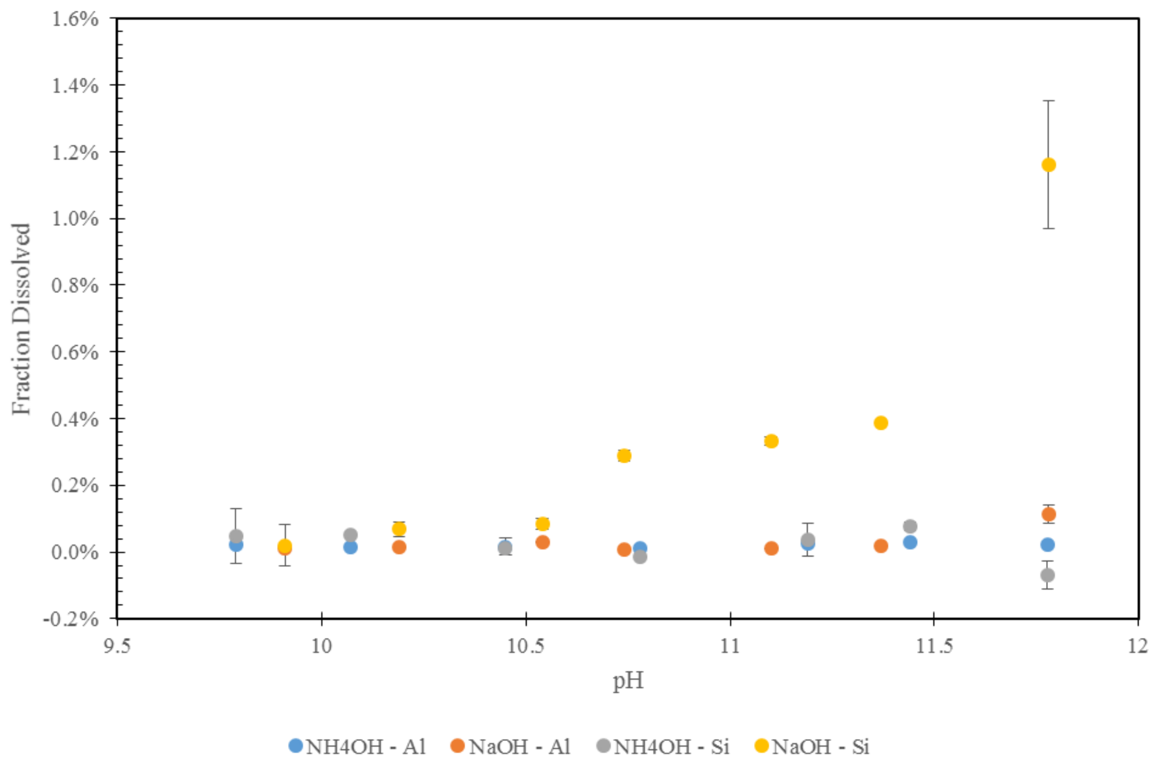


Figure 2-17. Elemental dissolution (Al and Si) of kaolinite (5 g/L) at variable pH with adjustment via NaOH or NH_4OH.

Synthetic Porewater vs. NaCl Electrolyte

Figures 2-18 to 2-20 compare equivalent experiments as described in the November monthly report with either synthetic porewater or NaCl at similar initial ionic strength. As shown by Figures 2-18 and 2-19, the mobility of U in the presence of the synthetic porewater versus NaCl are very different. For samples in which the pH was adjusted using NaOH (Figure 2-18), the sorption or removal of U from the aqueous phase is significantly decreased in the synthetic porewater as shown by the smaller K_d partitioning coefficients. This is likely due to the presence of additional carbonate in the synthetic porewater, which can complex U and decrease sorption. Further, this trend continues in the presence of NH_4OH at neutral pH in Figure 2-19. However, at the higher pH in the presence of NH_4OH, a different process appears to be controlling the system, leading to greater removal in the synthetic porewater likely due to a co-precipitation process.

Figure 2-20 depicts the dissolution of Si from kaolinite (initially 5 g/L) for synthetic porewater (circles) and NaCl (diamonds). Although there are only two datapoints for the NaCl background electrolyte, these points appear to follow the trend discussed above for the synthetic porewater. Therefore, the differences in electrolyte constituents between the synthetic porewater and NaCl do not appear to have a significant effect on mineral dissolution. Further, it must be noted that the dissolution of Al was fairly consistent in the presence of NaCl similar to data shown for the synthetic porewater in Figure 2-17.

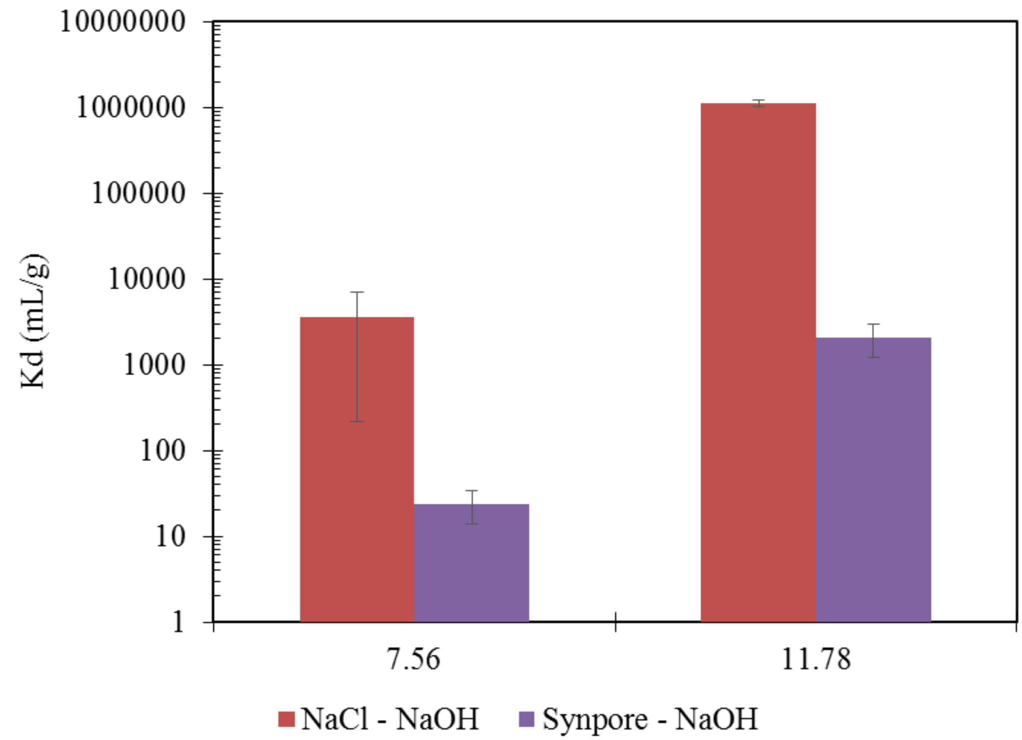


Figure 2-18. Comparison of K_d (mL/g) for 500 ppb U sorption to 5 g/L kaolinite in either synthetic porewater or NaCl at similar ionic strength at pH 7.6 and 11.8 with pH adjustment by NaOH.

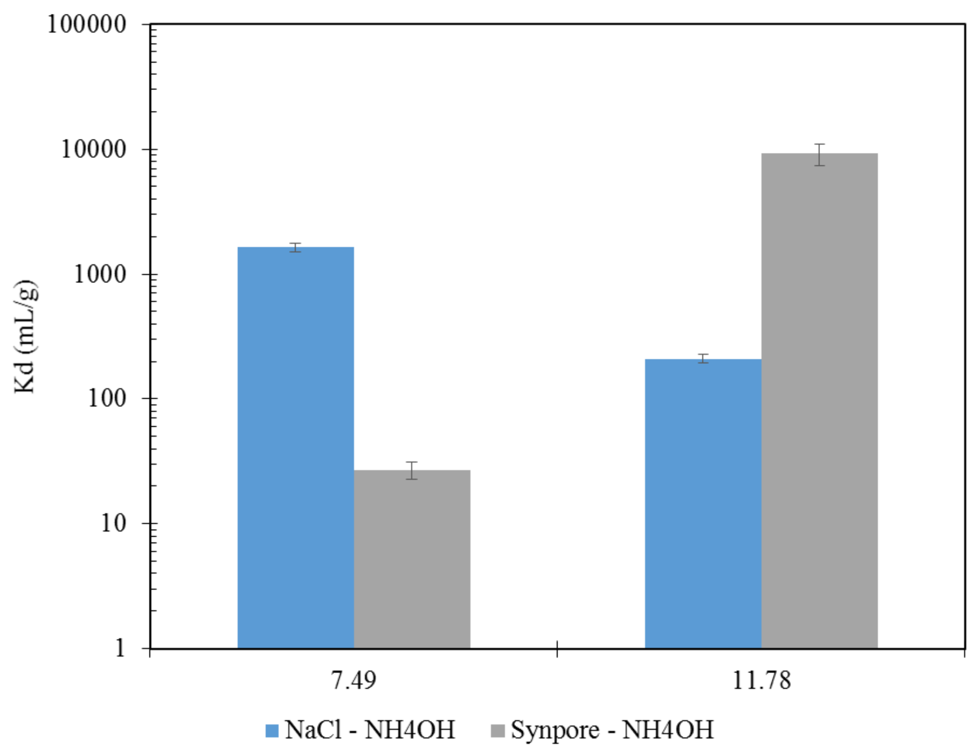


Figure 2-19. Comparison of K_d (mL/g) for 500 ppb U sorption to 5 g/L kaolinite in either synthetic porewater or NaCl at similar ionic strength at pH 7.6 and 11.8 with pH adjustment by NH_4OH.

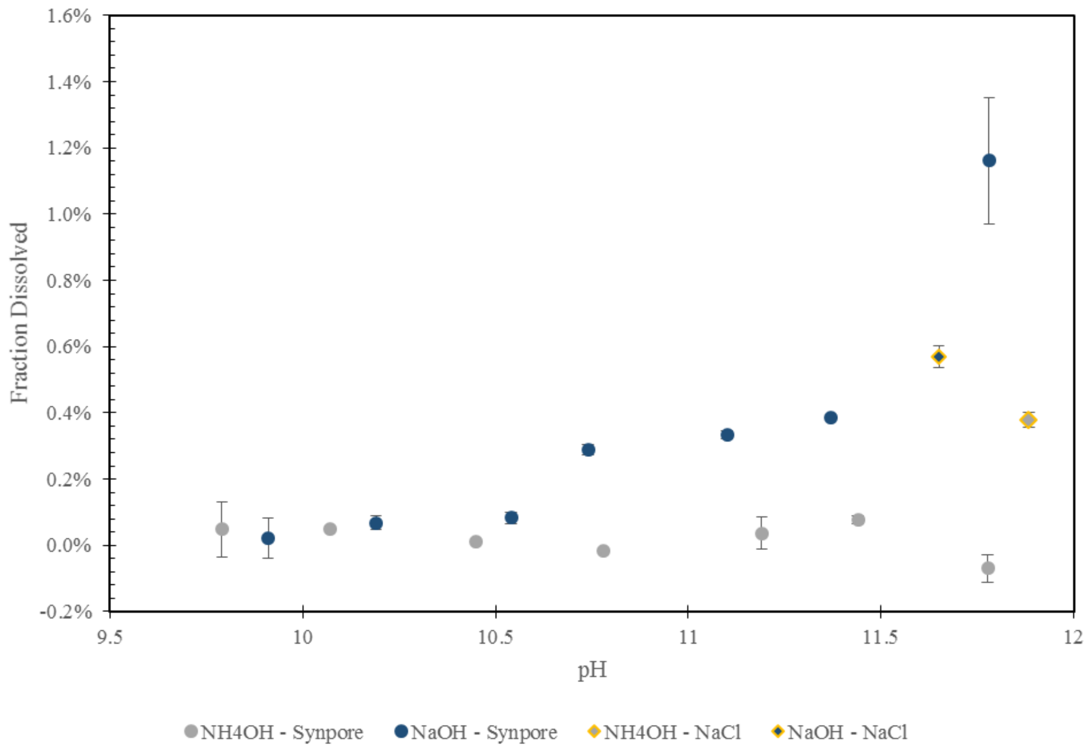


Figure 2-20. Comparison of Si dissolution from kaolinite (5 g/L) in either synthetic porewater or NaCl with pH adjustment by either NaOH or NH_4OH.

Table 2-5. Fractionation Scheme Procedures

Target Extraction Phase	Tessier (Beltrán et al., 2010; Tessier et al., 1979)	Modified Tessier (Clark et al., 1996; Serkiz et al., 2007)	BCR (Filgueiras et al., 2002)	Hanford-specific (Szecsody et al., 2010)
Solid:Liquid Ratio	Var – (1) 8:1, (2) 25:1, (3) 20:1, (4) 11:1	40:1'	40:1 (except final step is 50:1)	Not listed
Water soluble		Ultrapure water pH 5.5, 16 hour		Natural Hanford groundwater
Exchangeable	1 M $MgCl_2$ at pH 7, 1 hr	0.5 M $Ca(NO_3)_2$ pH 5.5, 16 hour		0.5 M $Mg(NO_3)_2$, 1 hour Changed – 0.0144 M $NaHCO_3$ + 0.0028 M Na_2CO_3, pH 9.45, 1 hour
Acid soluble or Carbonates	1 M NaOAc adjusted to pH 5 with acetic acid, 5 hour	0.44 M acetic acid, 0.5 M $Ca(NO_3)_2$, pH 2.5, 8 hour	HOAc 0.11 mol/L, 16 hour @ 25C	Step 1 – 1 M NaAc, 1 hour Step 2 – acetic acid at pH 2.3 (5 days)
Reducible or Fe/Mn oxides	0.04 M hydroxylamine in 25% HOAc at 96C, 6 hr	0.01 M hydroxylamine, pH 1.1, 30 min	0.1 mol/L hydroxylamine at pH 2, 16 hour @ 25C	0.1 M ammonium oxalate + 0.1 M oxalic acid, 1 hour
Oxidizable or Organics	3 mL 0.02 M HNO3 + 5 mL H_2O_2 adjusted to pH 2, 85C for 2 hour, add 3 mL H_2O_2, heat @ 85C for 3 hr, + 5 mL NH_4OAc 3.2 M @ 25C for 30 min	0.1 M $Na_2P_2O_7$, pH 10, 24 hour	10 mL H_2O_2 1 hr @ 25C, evaporate, 10 mL H_2O_2 at 85C, evaporate, 50 mL NH_4OAc 1 mol/L 16 hour @ 25C	
Residual	HF and $HClO_4$ mixture			8 MHNO$_3$, 95C, 2 hour
Long Term Available Uranium				1000 hr, 14.4 mM carbonate at pH 9.54 (same as above, exchangeable step)

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Subtask 1.3.3: The influence of microbial activity on the corresponding electrical geophysical response after ammonia injections in the vadose zone

A new DOE Fellow was hired to support this task. Alejandro Garcia is a new master's student in Geosciences beginning in the spring semester. He is currently working on a literature review in preparation for the experimental work for this task.

The review is focuses on literature involving the spectral induced polarization (SIP) geophysical method, its theory and how it pertains to biofilms occurring in porous media. The SIP uses AC current (in contrast to traditional resistivity methods which use DC current) to measure voltage and calculate complex conductivity (σ*), a property of a material associated with charge transport and storage. The complex conductivity expression is:

$$\sigma^*(\omega) = \sigma'(\omega) + i\sigma n(\omega)$$

Where σ'(ω) is the real conductivity and σn(ω) is the imaginary conductivity associated with charge storage through polarization. The theory behind using SIP to detect biofilm formation relies on the idea that bacteria have a negatively charged surface which creates polarization by attracting positively charged cations and creating a double charge layer. This polarization is then reflected in the calculated imaginary conductivity.

Outside of reviewing literature, the new DOE Fellow, Alejandro Garcia, supporting this task has been preparing paperwork required by PNNL for an internship for Spring 2016 as well as continuing to further his knowledge and expertise in Python.

Task 2: Remediation Research and Technical Support for Savannah River Site

Task 2 Overview

The acidic nature of the historic waste solutions received by the F/H Area seepage basins caused the mobilization of metals and radionuclides, resulting in contaminated groundwater plumes. FIU is performing basic research for the identification of alternative alkaline solutions that can amend the pH and not exhibit significant limitations, including a base solution of dissolved silica and the application of humic substances. Another line of research is focusing on the evaluation of microcosms mimicking the enhanced anaerobic reductive precipitation (EARP) remediation method previously tested at SRS F/H Area.

Task 2 Quarterly Progress – October 1 – December 31, 2015

Subtask 2.1. FIU's Support for Groundwater Remediation at SRS F/H –Area

A batch experiment was conducted by preparing a heterogeneous mixture containing SRS background soil (d>180 µm) and synthetic SRS water amended with U. The solutions were created to be saturated with U. These experiments are being conducted in order to assess the effect of sodium silicates on the pH of a solution using SRS soil and synthetic groundwater replicated after the site's natural groundwater composition. In order to create the SRS synthetic groundwater, a working solution was first prepared using the chemical concentrations below:

Table 2-6. Synthetic Groundwater Composition

Compound	**$CaCl_2$**	**Na_2SO_4**	**$MgCl_2$**	**KCl**	**NaCl**
Amount (g)	5.4771	1.0727	3.0943	0.3997	2.6528

After creating a well-mixed stock solution, 1 mL was then diluted into 1 L of slightly acidified (pH 3.6) deionized water to create the working solution. About 0.4 g of SRS soil was brought in contact with 20 ml of SRS synthetic groundwater modified with different concentrations of U. Three sets of samples were produced: the first set (Lot A) comprised of controls and samples that contained 50 ppb of U, the second set (Lot B) included controls and samples that contained 100 ppb of U [both Lot A and B were spiked with 70 ppm of sodium silicate (SS)], the third set (Lot C) comprised of samples containing 100 ppb of U and spiked with 140 ppm SS. All experiments were conducted in triplicate and the standard deviation was calculated. The samples were equilibrated for a period of 3 days on a shaking platform at 120 rpm. Aliquots were isolated from each sample over a 2 day period and analyzed by KPA to determine the U concentration. Table 2-7 shows the average U concentration for the different Lots followed by their standard deviation calculated from the triplicate samples. Readings where gathered over a period of 2 days; after one day, the average percent removal of U from the aqueous solution was about 48%. For both Lot A and B (composed of 50 and 100 ppb U and 70 ppm SS), there was a significantly greater removal of U than in Lot C (composed of 100 ppb U and 140 ppm SS), which was spiked with a higher sodium silicate concentration. Table 2-8 shows the pH measured over a period of 3 days followed by the relative standard deviation calculated from the triplicate samples. The pH from Lot A and B shows a slight increase over a period of 2 days, at near neutral conditions. Alternatively, Lot C had a decreasing trend from pH 9.6 to 8.4.

Table 2-7. Residual U Concentration Followed by Relative Standard Deviation for Samples Amended With 70 and 140 ppm Sodium Silicate

U Undersaturated Samples		
	Samples	Average Removal %
Day 1	Lot A	47.97± 4
	Lot B	48.11± 4
	Lot C	30.48± 0.5
Day 2	Lot A	55.27± 4
	Lot B	56.21± 3
	Lot C	35.28± 2

Table 2-8. pH Measured Over a Period of 2 Days Followed by Relative Standard Deviation for the Samples Containing U and Modified With 70 and 140 ppm Sodium Silicate

	Samples	
Day 0	Lot A	6.43± 0.02
	Lot B	6.55± 0.15
	Lot C	9.56± 0.06
Day 1	Lot A	7.2± 0.06
	Lot B	7.32± 0.15
	Lot C	9.09± 0.07
Day 2	Lot A	7.42± 0.01
	Lot B	7.42± 0.10
	Lot C	8.43± 0.36

These results are consistent with previous results, but the decrease in removal of U in Lot C can be attributed to the increase in pH, as shown in Table 2-8. When the concentration of SS was increased, the pH was also increased, decreasing the removal of U from the samples. The different mechanisms by which removal of U is achieved is currently been investigated. Future work on the samples with no SRS soil will give closer insight on the potential sorption of U in the SRS soil. Desorption and longevity of the remediation technology is under investigation presently, and will be able to provide evidence for the viability of the technology.

In an effort to better understand the chemical composition and morphology of samples collected on the 0.45 µm filters and initially spiked with the uranium concentration of 100 ppm, they were analyzed on SEM-EDS. The results of the analysis did not reveal any uranium present in the filters, despite our theoretical calculations. This experiment will be repeated again in the future; however, at this time, the focus of the experimental research is directed towards other objectives.

In order to better understand the role goethite and kaolinite play in the absorption process, an experiment was designed using pure quartz in exchange for the naturally occurring soil at the Savannah River Site. The experiment will otherwise remain consistent with the previous trials conducted using SRS synthetic groundwater, 70 ppm of sodium silicate, and 0.5 ppm of uranium (VI). Due to the presence of goethite and kaolinite in the SRS soil at an abundance of roughly 5%, yielding the same or similar uranium absorption rates with pure quartz will eliminate their known contribution to the process. On the contrary, if absorption rates with pure quartz no longer achieve the same rates as previously recorded, it can be largely attributed to the inclusion of goethite and kaolinite in the soil.

DOE Fellow Christine Wipfli presented her poster that showcased her research and the most recent progress achieved at the DOE Fellow Student Poster Competition on October 21. The event required the development of a poster displaying the highlights of the research and then a four-hour long exhibition to present the poster to university professors, ARC department staff, and FIU students.

In addition, Christine Wipfli upon returning from a summer internship produced a technical report to highlight major contributions of the tasks completed during time in DOE HQ. This report is composed of four technical case studies pertaining to different remediation strategies implemented across DOE complex sites. Although the report was completed in September, the content of the report required input and approval from representatives at each DOE site being represented in a case study. This progress is ongoing to accommodate for the various review processes. At this point in time, three out of the four case studies have completed the review and approval process, with one study outstanding. Upon completion of this final case study, the overall document will be completed and ready to be submitted to DOE and published on the FIU department website.

Several experiments were conducted to better understand the removal process of uranium bearing groundwater treated with sodium silicate. Two batch kinetic experiments were conducted to assess the contribution of the SRS soil on the removal of uranium from the aqueous phase. The first set of batch experiments used SRS soil, and the second set of batch experiments used Ottawa sand quartz, which makes up approximately 93% of the SRS soil. Less than 7% of the soil is composed of fine fraction clay, which consists mostly of kaolinite and goethite.

Approximately 0.8 g of SRS soil and Ottawa sand were placed into polypropylene vials and 40 ml of SRS synthetic groundwater (pH 3.6), containing 500 ppb of U(VI) was introduced. All samples were spiked with 70 ppm of sodium silicate (SS) and the final pH of the suspension was ~6.5. All experiments were conducted in triplicate and the standard deviation was calculated. The samples equilibrated for a period of 3 days on a platform shaker at 125 rpm. Aliquots were extracted from each sample at different time intervals over the equilibration period and analyzed by KPA to determine the residual uranium concentration.

In Figure 2-21, the results of the kinetic experiments with SRS soil and Ottawa sand (quartz) are presented. The U(VI) removal at the equilibrium for SRS soil was ~60% whereas Ottawa sand removed ~14% of uranium from the aqueous phase at equilibrium.

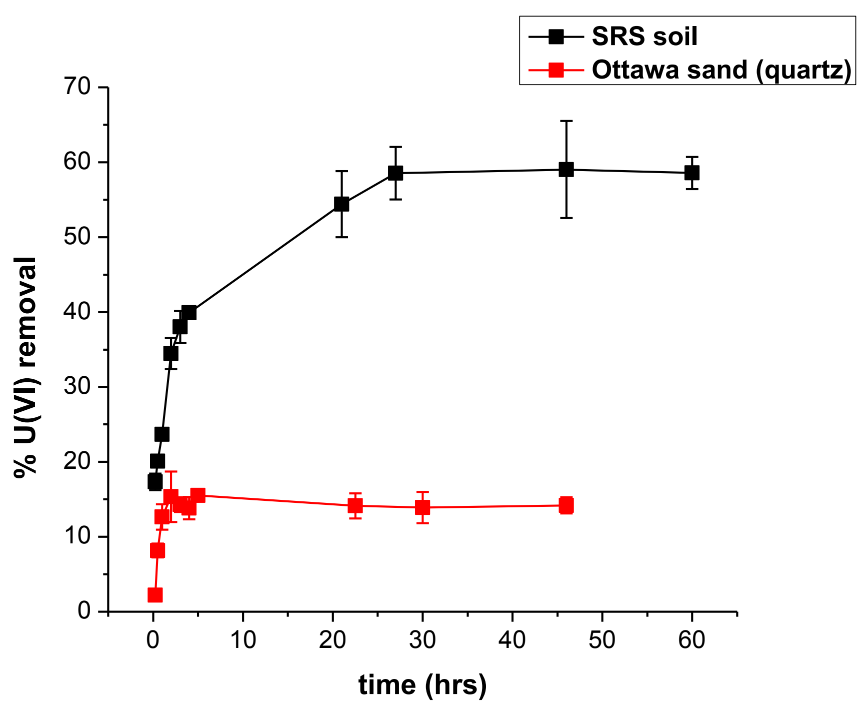


Figure 2-21. Kinetic results of U(VI) sorption at circumneutral conditions (pH ~6.5) at SRS soil and Ottawa sand (quartz).

A comparison of the results gives a clearer look at the contributions of the different fractions in the removal of uranium from the aqueous phase. Quartz, albeit making up 93% of the SRS soil, was able to remove only 14% of U(VI). The much larger U(VI) removal capacity by SRS soil can be attributed to the soil's other constituents, namely kaolinite and goethite. Furthermore, the equilibrium in the case of Ottawa sand is quite fast; maximum removal is reached within an hour, whereas in the case of SRS soil, only 24% of the initial U(VI) is removed. Equilibrium between SRS soil and the aqueous phase was already established at 21h.

Several experiments were next conducted to better understand the uranium removal mechanism from contaminated groundwater treated with sodium silicate. The focus of this experiment was shifted to the SRS synthetic groundwater composition to investigate the influence of the cations that are present in SRS synthetic groundwater on the uranium removal process. A batch experiment was conducted to assess the possibility of competition between cations in SRS synthetic groundwater and uranium for the same soil binding sites.

A batch experiment was conducted by preparing a suspension of SRS background soil (0.18<d< 2 mm) and synthetic SRS groundwater. The protocol for creating SRS synthetic groundwater has been described in detail in previous monthly reports.

Approximately 0.2 g of SRS soil was introduced to 20 mL of SRS synthetic groundwater (SRS.GRW). The different categories of samples are presented at Table 2-9. Batch experiments were conducted in the presence of sodium silicate (pH ~6.5) and without sodium silicate (pH ~3.5), as well as with and without uranium. The purpose was to track the amount of Ca, Mg, Al and Fe in the aqueous phase in the presence and absence of uranium to investigate if they affect the removal of uranium. The samples equilibrated for a period of 2 days on a platform shaker at 125 rpm. Aliquots were extracted from each sample at different time intervals over the equilibration period and analyzed by ICP-OES to determine the Ca, Mg, Al, and Fe concentrations. Finally, identical batch experiments (pH 3.5 and 6.5 after sodium silicate amendment) were carried out, but instead of using SRS synthetic groundwater, deionized water (DIW) was introduced in the samples. For the samples containing uranium, the U(VI) residual concentration was determined by means of KPA. All experiments were conducted in triplicate and the standard deviation was calculated.

Table 2-9. Schematic Representation of the Different Batch Experiments Conducted in Order to Investigate the Effect of Cations on the Uranium Sorption onto SRS Sediment

Code	U(VI), 0.5 ppm	Sodium silicate (70ppm) amendment	Medium
A	x		SRS synthetic groundwater
B	x	x	SRS synthetic groundwater
C			SRS synthetic groundwater
D		x	SRS synthetic groundwater

In Table 2-10, the average concentration of Ca and Mg in the aqueous phase is presented, followed by the standard deviation for all samples (A-D). A comparison of the concentration of

Ca and Mg in the SRS.GRW and all of the samples reveals that there are significantly greater concentrations of these cations in the supernatant solution compared to the composition of synthetic SRS groundwater. This result suggests that amounts of Ca and Mg could leach from the soil into the aqueous phase, despite the fact that calcium and magnesium oxides comprise a very small fraction of SRS sediment. Furthermore, results suggest that the amount of calcium and magnesium in the aqueous phase is not pH dependent, since code samples A and C have pH values of 3.5 whereas code samples B and D have pH values 6.5 (sodium silicate amendment). Similarly, the presence of uranium in the samples does not seem to affect the amounts of magnesium in the aqueous phase (code samples C and D do not contain uranium). Uranium removal for the code samples A (pH 3.5) was found to be zero while for code samples B (pH 6.5) was found to be 60±4%, consistent with all our previous experiments. On the other hand, a difference in the amount of calcium in the aqueous phase was observed in the presence of uranium, implying that there may be some limited ion-exchange between calcium and uranium during uranium sorption. Finally, there seems to be no difference between the different time intervals (day 1 and 2), something rather expected since in previous kinetic experiments, the equilibrium was found to be established in less than 24 hours.

Table 2-10. Ca and Mg Concentration in the Aqueous Phase and Relative Standard Deviation

		A	**B**	**C**	**D**
Day 1	Ca (ppm)	2.09±0.04	2.59±0.25	1.79±0.09	1.72±0.14
	Mg (ppm)	0.76±0.01	0.76±0.03	0.70±0.01	0.69±0.02
Day 2	Ca (ppm)	2.09±0.25	2.04±0.07	1.67±0.01	1.64±0.04
	Mg (ppm)	0.72±0.01	0.73±0.04	0.67±0.01	0.68±0.03
SRS.GRW	Ca (ppm)	0.54±0.03			
	Mg (ppm)	0.35±0.01			

In Table 2-11, the average concentration of Al and Fe in the aqueous phase is presented, followed by the standard deviation for all samples (A-D). The presence of Al and Fe in solution is an indication of aluminum and iron leaching from the soil (kaolinite and goethite respectively), since SRS synthetic groundwater does not contain any of these elements.

Table 2-11. Al and Fe Concentration in the Aqueous Phase and Relative Standard Deviation

		A	**B**	**C**	**D**
Day 1	Al (ppm)	0.71±0.08	0.66±0.07	0.66±0.09	0.56±0.1
	Fe (ppm)	0.32±0.16	0.39±0.01	0.28±0.12	0.41±0.20
Day 2	Al (ppm)	0.51±0.04	0.44±0.01	0.51±0.01	0.42±0.09
	Fe (ppm)	0.12±0.09	0.25±0.05	0.13±0.03	0.20±0.15
SRS.GRW	Al (ppm)	0			
	Fe (ppm)	0			

SRS sediments from the F/H Area are comprised roughly of 94% quartz, 5 % kaolinite, and 1% goethite; this explains the derivation of aluminum detected in the supernatant. The levels of iron and aluminum are similar across all the code groups for day 1, indicating that the leaching of iron and aluminum into the aqueous phase is not pH dependent and is not affected by the presence of uranium in the aqueous phase. On the other hand, the levels of iron and aluminum during the second day, although similar across the samples, are lower than the respective values of the first day. A possible explanation for this pattern may be the secondary precipitation of iron and aluminum at the respective pH values.

Finally, in the sorption experiments of U onto SRS sediment in deionized water with and without the addition of sodium silicate (pH 3.5 and 6.5 respectively), U(VI) removal was very similar when compared to the experiment with SRS synthetic groundwater. At pH 3.5, removal was zero, while at pH 6.5, removal was found to be 64±7%. This implies that the presence of several cations in the SRS synthetic groundwater (e.g., Na^+, K^+, Ca^{2+} and Mg^{2+}) has little or no interference with U(VI) sorption onto the sediment. The amount of calcium, magnesium, aluminum and iron leached from the sediment in the aqueous phase (DI water) is summarized in Table 2-12.

Table 2-12. Ca, Mg, Al and Fe Concentrations in the Aqueous Phase and Relative Standard Deviation

	pH 3.5	**pH 6.5**
Ca (ppm)	2.7±0.3	2.8±0.2
Mg (ppm)	0.75±0.2	0.72±0.1
Al (ppm)	0.40±0.03	0.44±0.01
Fe (ppm)	0.24±0.08	0.36±0.1

The concentrations of calcium, magnesium, iron and aluminum are very similar with the respective concentrations detected in the samples with SRS synthetic groundwater.

Subtask 2.2. Monitoring of U(VI) Bioreduction after ARCADIS Demonstration at F-Area

FIU prepared new samples to do sulfate analysis via ion chromatography (IC) in the FIU Chemistry Department, but the results were not yet available. Previous results indicated that sulfate reduction is likely occurring; however, further investigation is being planned to confirm if this is true.

DOE Fellow Aref Shehadeh prepared a poster for the DOE Fellow student competition entitled, "Optimizing Remediation of I-129 using AgCl Colloidal-Sized Particles in SRS F-Area Sediments," for the experimental work he conducted under the summer internship mentor Dr. Miles Denham (SRNL). Silver chloride (AgCl) is currently being used at the SRS F-Area for the *in situ* remediation of the radioactive iodine-129 (I-129) plume, which has progressed from a series of unlined seepage basins.

In the month of November, FIU continued to work on a paper pertaining to the experiment "*Monitoring of U(VI) bioreduction after ARCADIS demonstration at SRS F-Area*" which describes how the pH conditions at the Savannah River Site F-Area were not conducive to the formation of iron precipitates. The introduction, methodology, and preliminary results are near completion. This paper will be citing several scientific studies, which can provide evidence as to why iron precipitates were not observed in the microcosm experiments. Original data from the experiment has been condensed and analyzed into a format that will better suit the current paper. In the coming weeks, the sulfate analysis should be completed with help from the FIU Chemistry Department, and modeling of the microcosm experiment will begin using the Geochemist Workbench software.

In the month of December, FIU finalized sulfate analysis via ion chromatography for the liquid samples collected from the microcosm experiments. All samples collected for analysis were kept under anaerobic conditions in the anaerobic glove box until time of assay. A calibration curve was prepared by using a sulfate standard (Figure 2-22).

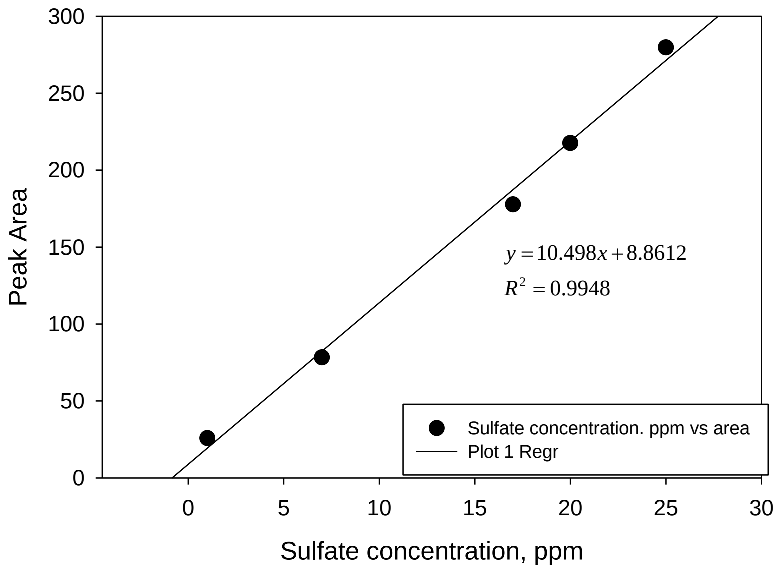


Figure 2-22. Calibration curve for sulfate analysis.

Data suggested that there is no sulfate reduction in any of batches augmented with sulfate and the concentration remained on the level of 500 ppm as originally added to the initial solutions (518-542±14.5 ppm). This might explain why XRD analysis hasn't revealed the formation of pyrite phases. The next step of research will be to conduct speciation modeling of the microcosm experiment using the Geochemist Workbench software.

Subtask 2.3: Sorption Properties of Humate Injected into the Subsurface System

During the month of October, the experiment on the humate substances (HS) desorption was completed. This experiment was conducted for 30 days due to the longer time needed for the desorption process to occur. The experiments started by weighing 1 g of SRS sediments into centrifuge tubes. A known concentration of HumaK was then pipetted into the tubes and the pH was adjusted to 4. The final volume in each tube was adjusted to 20 mL. The samples were left on the shaker for five days to be able to reach equilibrium for the adsorption process; this amount of time was previously determined in the adsorption kinetic experiment. After the fifth day, the samples were withdrawn, and the supernatant of the samples was removed and replaced by DI water with the pH adjusted to the same level (pH = 4). The pH of the samples was measured and adjusted daily to pH 4. At predetermined time intervals, samples were withdrawn and centrifuged. The concentration of the supernatant was measured by UV-vis spectrophotometer. The humate initial concentration C_0 (Figure 2-23) corresponds to the HS adsorbed to SRS sediments before desorption. The results show that the concentration of HumaK remaining adsorbed after the completion of the desorption experiments remains almost constant from day 3 to day 30. A possible explanation for this behavior could be that the pH 4 of the DI water that replaced the supernatant solution was the same as for the sorption experiments. In this case, the interactions in the sediments adsorbed layer remain relatively the same. Therefore, there is no increase in negative charges in humic molecules or the surface charge of sediments that could stimulate the desorption process. Also, humic molecules that are adsorbed strongly will not be desorbed easily, and the humic molecules that have a weak interaction with sediments (either physical or reversible adsorption) will be desorbed. In the next month, FIU will start Humak desorption experiments at different pH values (from 4 to 8).

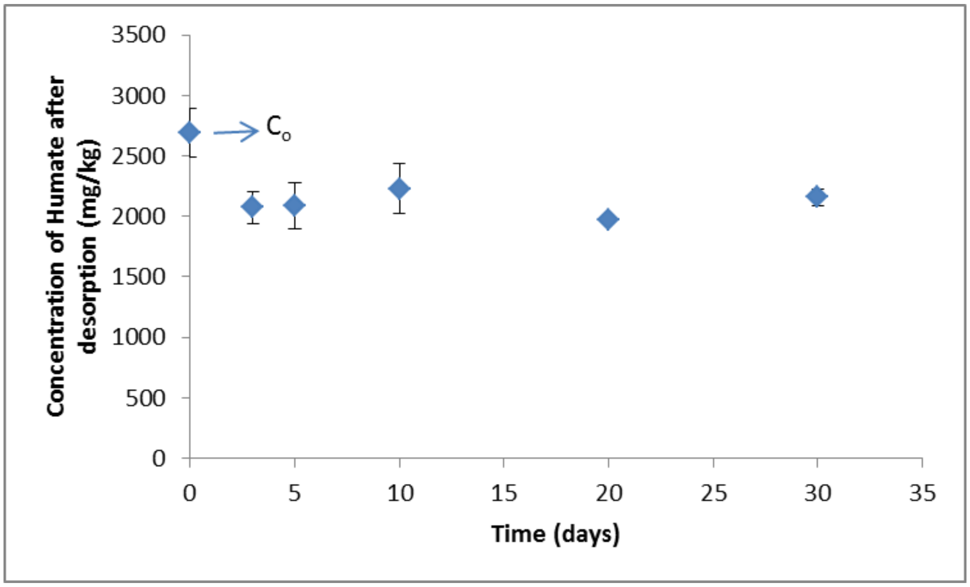


Figure 2-23.Concentration of humate after desorption, mg/kg.

DOE Fellow Hansell Gonzalez presented a poster entitled, "Study of an Unrefined Humate Solution as a Possible Remediation Method for Groundwater Contamination," for the DOE Fellows Poster Exhibition which took place on October 21, 2105. Hansell's poster contains the results on the characterization of SRS sediments and Huma-K by using scanning electron microscopy, fourier transform infrared spectroscopy, and potentiometric titrations.

During the month of November, updates on Task 2.3 *"Sorption Properties of Humate Injected into the Subsurface System"* were presented during the DOE Fellows Induction Ceremony to the new DOE Fellows and special guests. The presentation by DOE Fellow, Hansell Gonzalez, covered the latest results on the characterization of SRS sediments and Huma-K by using scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM/EDS), fourier transform infrared spectroscopy (FTIR), and potentiometric titrations.

In addition, FIU initiated the first draft of a manuscript for a peer-reviewed publication that summarizes all experimental studies conducted using HumaK as a low-cost remediation method for acidic groundwater contaminated with uranium. So far, the "Materials and Methods" section is close to being finalized. This section presents a detailed description of SRS sediments and HumaK preparation for the experiments, as well as details of the characterization techniques using SEM/EDS, FTIR, and potentiometric titration and the parameters used for analysis. It also describes experimental procedures and presents respective formulas used to calculate the amount of HumaK sorbed to SRS sediments for the kinetics, sorption at different pH values and desorption experiments.

FIU began preparation for the desorption experiment of HumaK at different pH values. The desorption studies will investigate if the change in the solution's pH plays an important role in the desorption process and determine the extent of sorption reversibility. The procedure for the desorption experiments includes pipetting a known concentration of HumaK into centrifuged tubes containing SRS sediments. Samples will be left on a rotary shaker until adsorption equilibrium is reached. After the equilibrium is attained, the supernatant will be replaced by DI water at different pH values (from 4 to 8), and the samples will be placed on the rotary shaker for five days. The concentration of the supernatant will be measured by a UV-vis spectrophotometer.

During the month of December, efforts on this subtask focused on the desorption experiments for HumaK at different pH values. Initially, a HumaK concentration of 500 mg L^{-1} was pipetted into centrifuges tubes containing 1 g of SRS sediments. The pH of the samples was adjusted to 4, and the samples were homogenized by a vortex mixer. The samples were then placed on a platform shaker until sorption reached equilibrium. During that period of time, pH was checked and adjusted daily by adding either 0.1 M HCl or 0.1 M NaOH. Once sorption equilibrium was reached, the samples were centrifuged for 30 min at 2700 RPM. The concentration of HumaK in the supernatant was analyzed by means of UV-vis spectrophotometry.

Further, an initial draft was developed describing the materials and methods of this research for a manuscript to be submitted for publication in a peer-reviewed journal. The manuscript draft summarizes all of the experimental data obtained with HumaK as a low-cost remediation method for acidic groundwater contaminated with uranium. The draft is currently undergoing internal review and revision.

The desorption experiments of HumaK at different pH values will be continued in January. Experiments will be done by replacing the supernatant with deionized water at different pH values (from 4 to 8). The pH will be checked and adjusted daily. Samples will be left on the rotary shaker for five days. After that, the samples will be centrifuged and the concentration of the HumaK desorbed in the aqueous phase will be determined spectrophotometrically.

Subtask 2.4. The Synergistic Effect of HA and Si on the Removal of U(VI)

FIU drafted and submitted a paper for the Waste Management Symposia to be held on March 6-10, 2016 in Phoenix, AZ. The paper was accepted for presentation at the conference.

> **Abstract**: 16524 – Multicomponent Batch Experiments Investigating Uranium Synergy with Humic Acid, Silica Colloids and SRS Sediments at Variable pH
> **Session**: 071A – Posters: Environmental Remediation Analysis, Technology and Treatment Systems
> **Date**: Tuesday, March 08, 2016
> **Time**: 1:30 - 5:00 PM

Batches 1, 4, and 7 containing uranium, silica and/or sediment with no humic acid addition were analyzed using ICP-OES in order to determine silica and iron concentrations. Previously, these samples were analyzed via KPA and data analysis for pH 3, which provided inconclusive results. Thus, new samples were prepared in order to compare those values. These samples have been analyzed with ICP-OES and the data is currently under processing; KPA is still required to measure the uranium concentrations. KPA analysis and data analysis for ICP-OES results will be completed in the next reporting period.

Data obtained for batches 1, 4, and 7 containing uranium, silica, and/or sediment with no humic acid was processed (Table 2-13). Batch 1 filtered samples, which contain only silica and uranium, revealed a high silica percent removal with pH 3 yielding the highest removal at 99% and subsequently decreasing until pH 8, with a removal percent of 88%. Batch 4 (silica, uranium, sediment) filtered samples had an average removal of ~97% between all pH values. Batch 7 was analyzed and revealed no silica presence as expected; batch 7 does not contain silica and only contains sediment and uranium. Unfiltered samples were also analyzed, though must be reprocessed; the internal standard yttrium revealed a discrepancy yielding inconclusive data; the samples will be reanalyzed and the data will presented in the next report.

Table 2-13. Silica Removal for Filtered Samples

pH 3	Si Avg Removal, %	Std Deviation
Batch 1	99.378	0.317
Batch 4	95.633	1.423
Batch 7	-	-
pH 4	**Si Avg Removal, %**	**Std Deviation**
Batch 1	97.741	2.045
Batch 4	96.361	0.548
Batch 7	-	-
pH 5	**Si Avg Removal, %**	**Std Deviation**
Batch 1	96.395	1.718
Batch 4	97.058	0.932
Batch 7	-	-
pH 6	**Si Avg Removal, %**	**Std Deviation**
Batch 1	92.299	1.610
Batch 4	96.405	1.443
Batch 7	-	-
pH 7	**Si Avg Removal, %**	**Std Deviation**
Batch 1	88.724	3.888
Batch 4	98.670	0.638
Batch 7	-	-
pH 8	**Si Avg Removal, %**	**Std Deviation**
Batch 1	88.129	2.122
Batch 4	97.718	0.102
Batch 7	-	-

Data obtained for batches 1, 4, and 7 containing uranium, silica, and/or sediment with no humic acid was also processed (Table 2-14). Unfiltered samples were analyzed during the month of November; however, there was discrepancy in the internal standard yttrium intensity. Fresh unfiltered samples were prepared and analyzed; silica removal data obtained during this analysis is shown in Table 2-14. Batch 1 showed approximately 50% removal while Batch 4 data was inconsistent; most of Batch 4 samples showed no detectable concentration of silica.

Table 2-14. Silica Removal for Unfiltered Samples

Sample-Description, pH 3	Si Avg Removal, %	Std Deviation
Batch 1	77.600	5.070
Batch 4	Not Detected	Not Detected
Batch 7	-	-
Sample-Description, pH 4	**Si Avg Removal, %**	**Std Deviation**
Batch 1	54.600	7.770
Batch 4	Not Detected	Not Detected
Batch 7	-	-
Sample-Description, pH 5	**Si Avg Removal, %**	**Std Deviation**
Batch 1	55.600	1.550
Batch 4	Not Detected	Not Detected
Batch 7	-	-
Sample-Description, pH 6	**Si Avg Removal, %**	**Std Deviation**
Batch 1	48.200	4.350
Batch 4	45.100	9.060
Batch 7	-	-
Sample-Description, pH 7	**Si Avg Removal, %**	**Std Deviation**
Batch 1	51.300	6.480
Batch 4	Not Detected	Not Detected
Batch 7	-	-
Sample-Description, pH 8	**Si Avg Removal, %**	**Std Deviation**
Batch 1	41.600	2.890
Batch 4	Not Detected	Not Detected
Batch 7	-	-

Subtask 2.5. Investigation of the Migration and Distribution of Natural Organic Matter Injected into Subsurface Systems

FIU drafted and submitted a paper for the Waste Management Symposia to be held on March 6-10, 2016 in Phoenix, AZ. The paper was accepted for presentation at the conference.

> **Abstract**: 16523 – Migration and Distribution of Natural Organic Matter Injected into Subsurface Systems
> **Session**: 071B – Posters: Environmental Remediation Field Investigation and Remediation
> **Date**: Tuesday, March 08, 2016
> **Time**: 1:30 - 5:00 PM

The work completed for this task will assemble, integrate, and develop a practical and implementable approach to quantify and simulate potential natural organic matter (NOM, such as humic and fulvic acids, humate, etc.) deployment scenarios over the range of conditions at DOE sites. Initial laboratory experiments and an initial set of simplified models have been developed at SRNL. Under this task, additional batch and column studies and testing will be conducted at

FIU to provide the transport parameters for an extension of the current model scenarios. The following was accomplished during this performance period:

- Column 1 was drained and soil was divided into 6 sections and a small amount of each section was oven dried at 35°C for 2-days. A representative sample from each section was used to prepare samples for SEM analysis.
- One sample was taken from the homogeneous mixture of soil used in the column and was used for SEM analysis.
- SEM analysis is in progress. Results will be reported once the analysis is completed.
- Completed SEM-EDS analysis of representative sediment samples from Column 1 that was oven dried at 35°C for 2 days.
- EDS analysis revealed that the concentration of carbon in the sample increased as it moved down the column. TOC analysis is planned to get quantitative data of humic acid retained in the column.
- Column 2 was taken apart and six soil samples were taken 2 inches apart from each other and dried in an oven at 35°C for 2 days.
- Samples will be analyzed via SEM and TOC analysis and data will be reported.
- The test plan was updated based on discussions with Miles Denham and Brian Looney at SRNL.
- Initiated drafting of experimental procedures for approval by FIU's radiation control committee since this year's experiments involve uranium solution injection.
- FIU began drafting a poster for the Waste Management titled, "*Migration and Distribution of Natural Organic Matter Injected into Subsurface Systems*."

Task 3: Surface Water Modeling of Tims Branch

Task 3 Overview

This task will perform modeling of surface water, and solute/sediment transport specifically for mercury and tin in Tims Branch at the Savannah River Site (SRS). This site has been impacted by 60 years of anthropogenic events associated with discharges from process and laboratory facilities. Tims Branch provides a unique opportunity to study complex systems science in a full-scale ecosystem that has experienced controlled step changes in boundary conditions. The task effort includes developing and testing a full ecosystem model for a relatively well defined system in which all of the local mercury inputs were effectively eliminated via two remediation actions (2000 and 2007). Further, discharge of inorganic tin (as small micro-particles and nanoparticles) was initiated in 2007 as a step function with high quality records on the quantity and timing of the release. The principal objectives are to apply geographical information systems and stream/ecosystem modeling tools to the Tims Branch system to examine the response of the system to historical discharges and environmental management remediation actions.

Task 3 Quarterly Progress – October 1 – December 31, 2015

Subtask 3.1. Modeling of Surface Water and Sediment Transport in the Tims Branch Ecosystem

- FIU drafted and submitted a paper for the Waste Management Symposia to be held on March 6-10, 2016 in Phoenix, AZ. The paper was accepted for presentation at the conference.

  Abstract: 16203 – Development of an Integrated Hydrological Model for Simulation of Surface Runoff and Stream Flow in Tims Branch Watershed
  Session: 071A – Posters: Environmental Remediation Analysis Technology and Treatment Systems
  Date: Tuesday, March 08, 2016
  Time: 1:30 - 5:00 PM

- FIU is continuing the literature review on similar type watershed models developed using MIKE SHE/MIKE 11 as well as previous hydrology modeling efforts at SRS. Some of the publications reviewed include:
  - Dai, Z., Li, C., Trettin, C., Sun, G., Amatya, D., and Li, H., 2010, Bi-criteria evaluation of the MIKE SHE model for a forested watershed on the South Carolina coastal plain: Hydrology and Earth System Sciences, v. 14, no. 6, p. 1033-1046.
  - Graham, D. N., and Butts, M. B., 2005, Flexible, integrated watershed modelling with MIKE SHE: Watershed models, v. 849336090, p. 245-272.
  - Wijesekara, G., 2013, An integrated modeling system to simulate the impact of land-use changes on hydrological processes in the Elbow River watershed in Southern Alberta [Ph.D.: University of Calgary.
  - Wijesekara, G., Farjad, B., Gupta, A., Qiao, Y., Delaney, P., and Marceau, D., 2014, A Comprehensive Land-Use/Hydrological Modeling System for Scenario Simulations in the Elbow River Watershed, Alberta, Canada: Environmental Management, v. 53, no. 2, p. 357-381.
- MIKE SHE model development included:
  - Unsaturated zone data preparation
    - Soil re-classification: re-classification of the soil into 4 soil types.
    - Literature review on soil characteristics and parameters (porosity, hydraulic conductivity, etc.) to find soil hydrogeological parameters to be used in MIKE SHE model.
    - Literature review on unsaturated zone MIKE model set up: previous model set up and approaches.
- MIKE 11 model preparation:
  - Continued development of cross sections as seen in figure below (approx. 50% work completed).

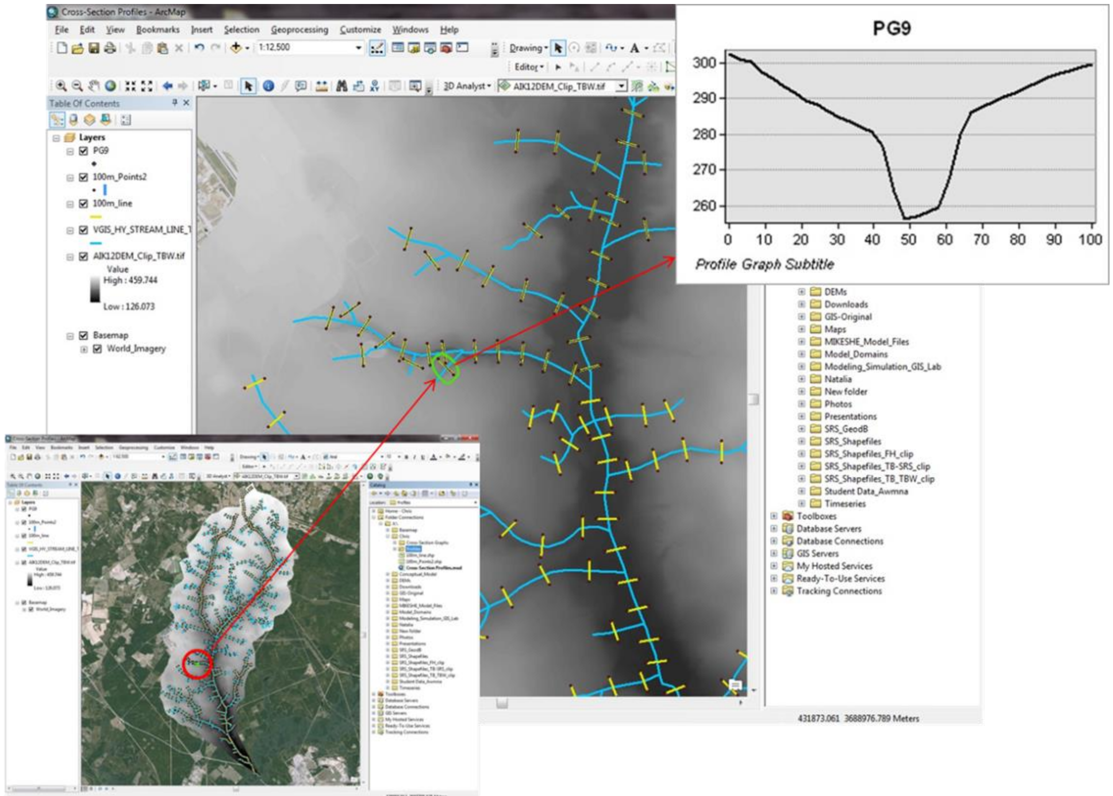


Figure 2-24. An ArcMap view of the Tims Branch delineated cross sections (left and center); the cross section profile of the cross section #PG9 (right).

- Implementation of SWAT (Soil and Water Assessment Tool) model for the Tims Branch watershed for comparative purposes. The SWAT model is a river basin scale model developed to predict the impact of land management practices on water, sediment and agricultural chemical yields. SWAT is a public domain model actively supported by the USDA at the Grassland, Soil and Water Research Laboratory in Temple, Texas.
- The SWAT model setup involves several steps: (1) data preparation as per model requirement; (2) watershed delineation and sub-basin discretization; (3) Hydrological response unit (HRU) definition; (4) preparing weather and other SWAT input tables; (5) parameter sensitivity analysis; and (6) calibration and uncertainty analysis.
- The Digital Elevation Model (DEM) was used to delineate the watershed and to analyze the drainage patterns of the land surface terrain. Sub-basin parameters such as slope gradient, slope length of the terrain, and the stream network characteristics were derived from the DEM. The land use map was reclassified into SWAT land cover/plant types. A user look up table was created to identify the SWAT code for the different categories of land cover/land use on the map as per the required format.

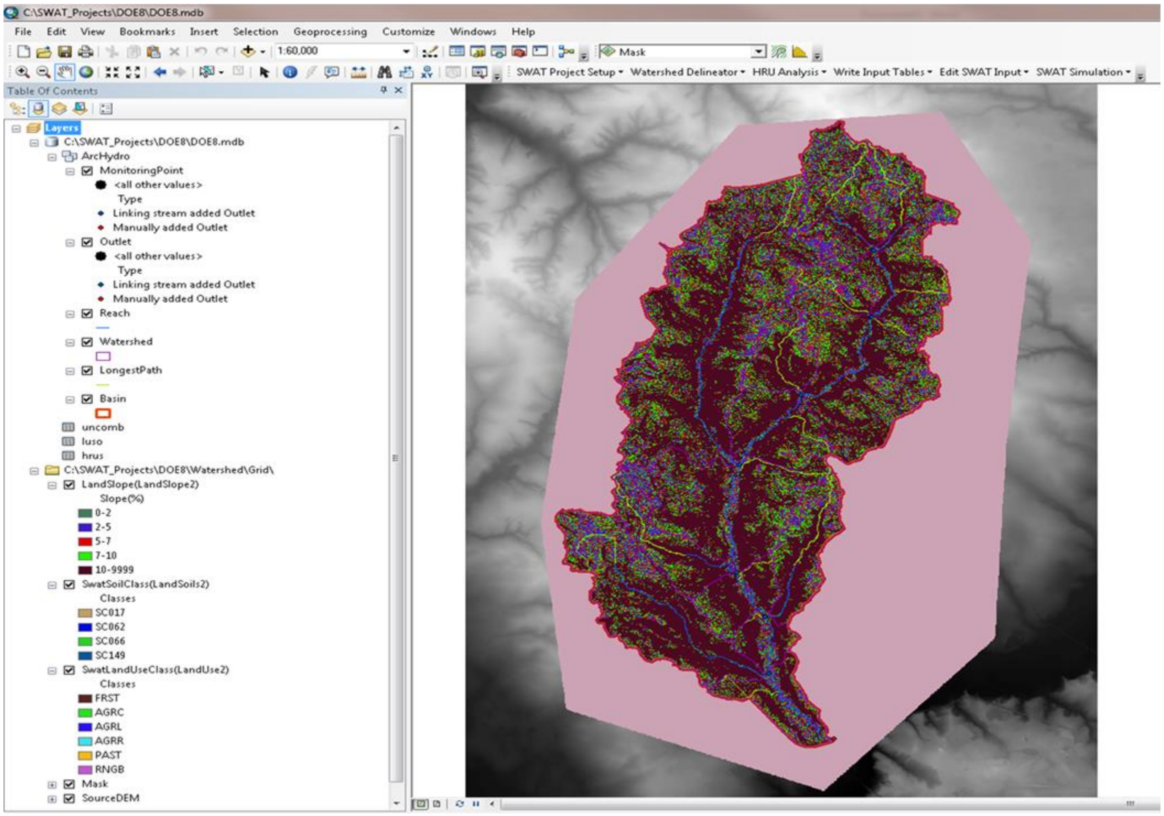


Figure 2-25. Delineation of the Tims Branch watershed using the SWAT model.

- FIU has completed the update of the MIKE SHE overland flow module to incorporate the relevant parameters required for simulation of overland flow using the revised model boundary. Simulations are currently being run to test the model and refine the parameters as necessary.
- Preparation of the unsaturated zone data is also still in progress. This includes:
    - Soil re-classification: re-classification of the soil into 4 soil types.
    - Literature review on soil characteristics and parameters (porosity, hydraulic conductivity, etc.) to find soil hydrogeological parameters to be used in MIKE SHE model.
    - Literature review on unsaturated zone MIKE model set up: previous model set up and approaches.
- GIS data review:
    - The existing groundwater table shapefiles were carefully studied and it was determined that the data provided more of a spatial distribution with no timeseries data. As such, groundwater table timeseries data will be acquired from available sources and agencies and input into the model to serve as initial data/calibration/boundary conditions in the MIKE SHE hydrology model.
    - As part of the unsaturated flow development, and based on the previous literature review, a two layer unsaturated zone has been defined. Each zone requires soil characteristics and classifications. This part is under construction.

    - In addition, timeseries data of Leaf Area Index and Root Depth are being retrieved from online resources. This data is significant for ET module development in MIKE SHE.

- MIKE 11: More than 80% of the cross sections for the Tims Branch study area have been prepared. The DOE Fellows are being trained to use ArcHydro for watershed delineation to be applied in future model development and river chainage and linkage definitions in accordance with MIKE 11.

- Implementation of SWAT (Soil and Water Assessment Tool) model

    - Implementation of SWAT model for the Tims Branch watershed is 50% completed.

    - Delineation of the Tims Branch watershed: The Digital Elevation Model (DEM) was used to delineate the watershed and to analyze the drainage patterns of the land surface terrain. Delineation of the watershed is 100% completed. Sub-basin parameters such as slope gradient, slope length of the terrain, and the stream network characteristics were derived from the DEM. The land use map was reclassified into SWAT land cover/plant types. A user look up table was created to identify the SWAT code for the different categories of land cover/land use on the map as per the required format.
    - The hydrological response unit (HRU) definition: Subdividing the sub watershed into areas having unique land use, soil and slope combinations makes it possible to study the differences in evapotranspiration and other hydrologic conditions for different land covers, soils and slopes. The HRU definition process is 100% completed.
- Students were encouraged to prepare a poster for the WM 2016 symposium. Three abstracts were submitted to WM2016 as student poster presentations.

- A poster entitled "*Integrated Modeling System for Analysis of Watershed Water Balance: A Case Study in the Tims Branch Watershed, South Carolina*" was prepared and presented at the American Geophysical Union (AGU) Fall 2015 meeting in December by Dr. Shimelis Setegn (Figures 2-26 and 2-27).

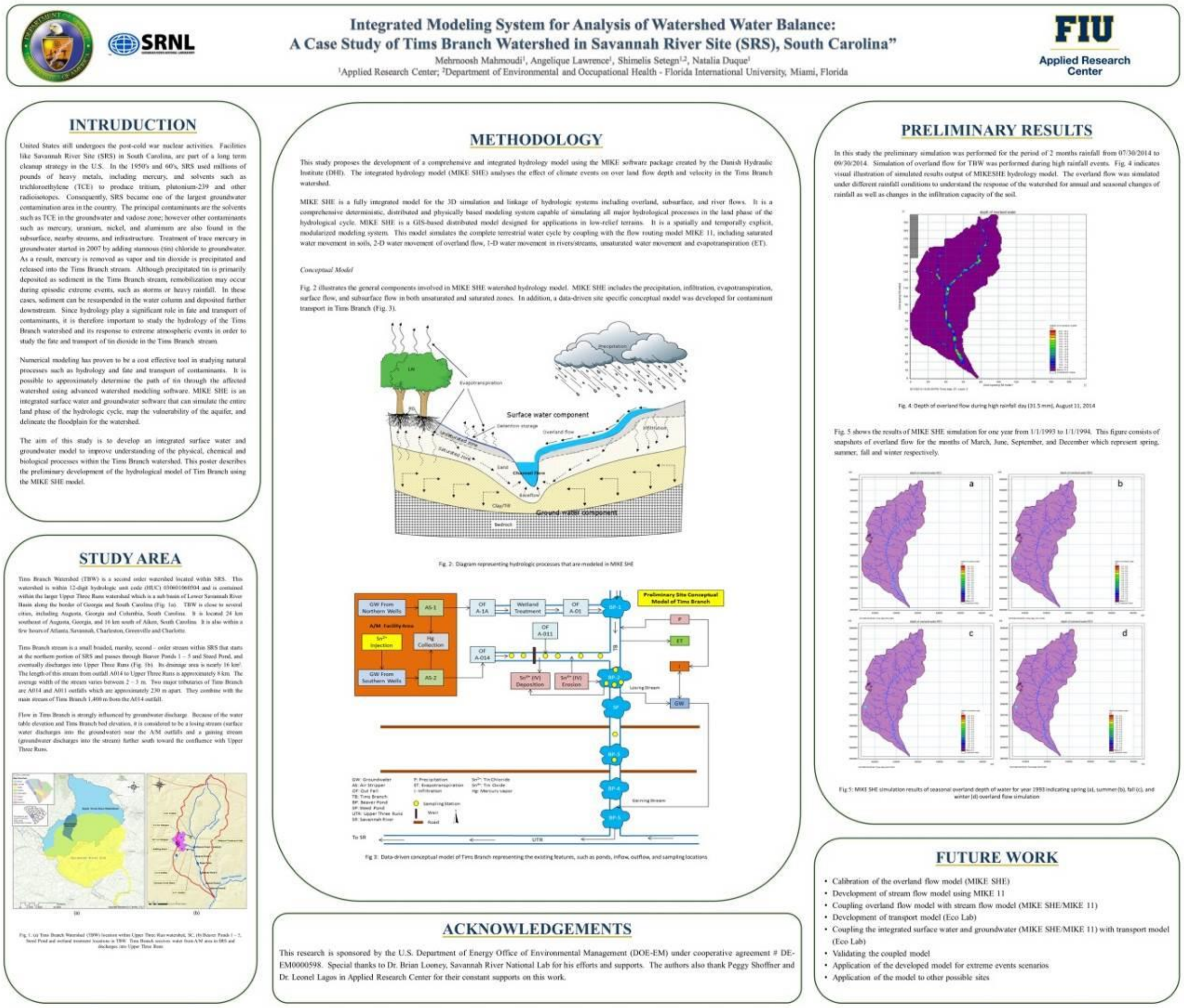


Figure 2-26. Poster presented at American Geophysics Union (AGU) Fall 2015 meeting.

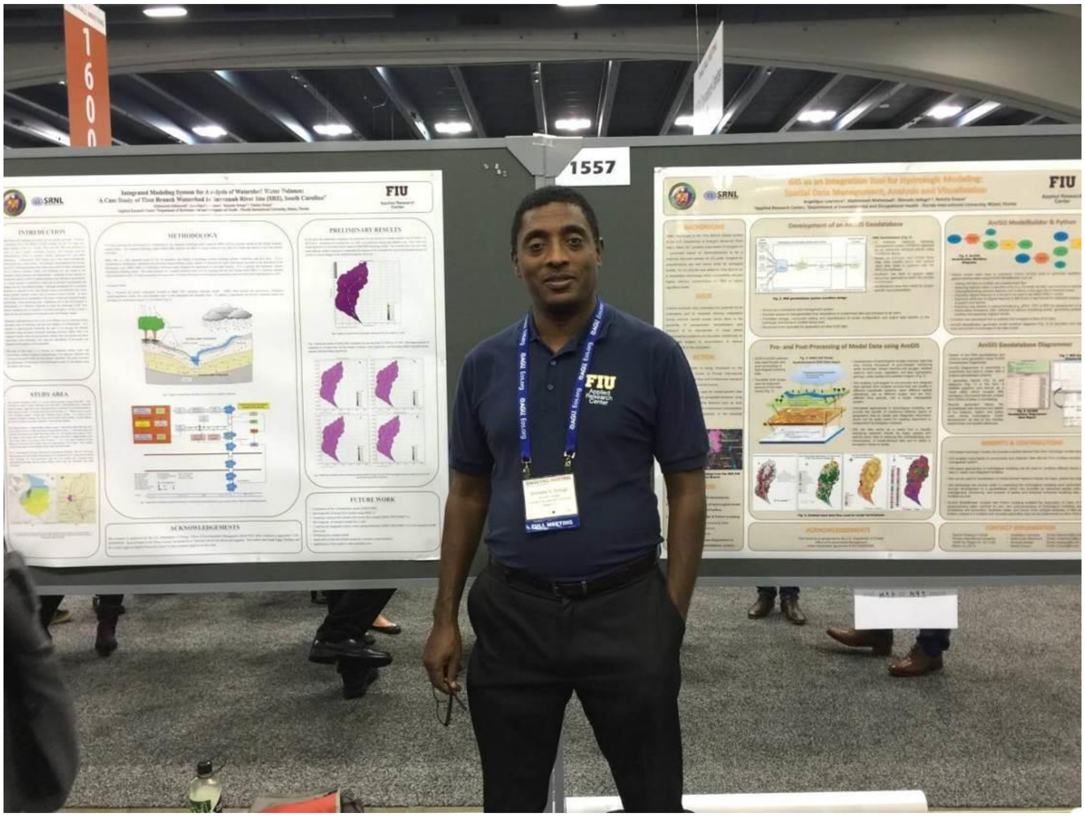


Figure 2-27. Dr. Shimelis Setegn presenting FIU research at the AGU Fall 2015 meeting.

- FIU has completed the update of the MIKE SHE overland flow module to incorporate the relevant parameters required for simulation of overland flow using the revised model boundary. A summary of the task milestone was submitted to DOE on December 30, 2015. Updated model data and parameters were summarized in the milestone report. Figure 2-28 illustrates an example of a preliminary simulation of overland flow with the new domain boundary. Table 2-15 shows the vegetation data used in model development.

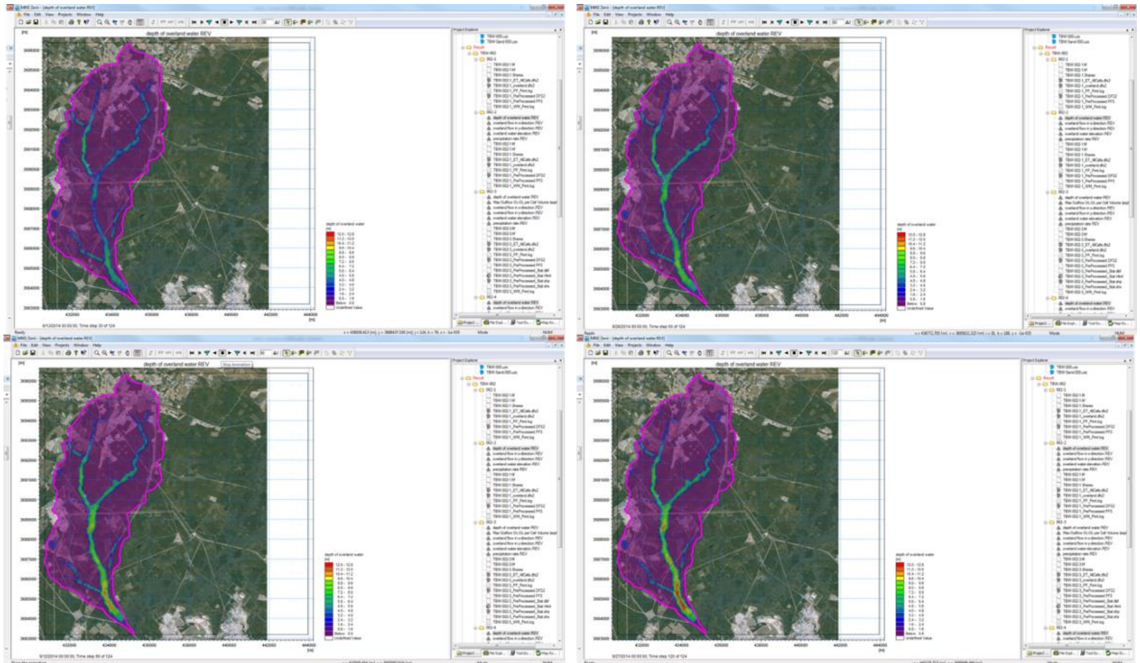


Figure 2-28. Preliminary simulation of overland flow in Tims Branch watershed.

Table 2-15. Vegetation data, Leaf Area Index (LAI) and Root Depth (RD)

Vegetation ID	LAI	RD (mm)
Barren Land	1.31	4000
Cultivated Crops	3.62	1500
Deciduous Forest	5.5	2000
Developed Low Intensity	2.5	2000
Developed Medium Intensity	2.0	2000
Developed Open Space	3.0	2000
Emergent Herbaceous Wetland	6.34	2000
Evergreen Forest	5.5	1800
Hay/pasture	1.71	1500
Mixed Forest	5.5	2400
Open Water	0.0	0.0
Quarries	1.31	4000
transitional	1.31	4000
Urban/Recreational Grasses	2.0	2000
Woody Wetland	6.34	2000

- Preparation of the unsaturated zone data is also still in progress. This includes:
  - The re-classification of the soil into 4 soil types which has been well documented in the milestone report.
  - Literature review on soil characteristics and parameters (porosity, hydraulic conductivity, etc.) to find soil hydrogeological parameters to be used in MIKE SHE model.
  - Literature review on unsaturated zone MIKE model set up: previous model set up and approaches.
  - The initial depth of water and the groundwater table data across the watershed were imported into the MIKE model. This data was prepared using various GIS tools.
- GIS data review:
  - The existing groundwater table shapefiles were carefully studied and it was determined that the data provided more of a spatial distribution with no timeseries data. As such, groundwater table timeseries data will be acquired from available sources and agencies and input into the model to serve as initial data/calibration/boundary conditions in the MIKE SHE hydrology model.
  - As part of the unsaturated flow development, and based on the previous literature review, a two layer unsaturated zone has been defined. Each zone requires soil characteristics and classifications. This part is under construction.
  - In addition, timeseries data of Leaf Area Index and Root Depth are being retrieved from online resources. This data is significant for ET module development in MIKE SHE.
- MIKE 11: More than 80% of the cross sections for the Tims Branch study area have been prepared. The DOE Fellows are being trained to use ArcHydro for watershed delineation to be applied in future model development and river chainage and linkage definitions in accordance with MIKE 11. Figure 2-29 displays the cross sections in the watershed.

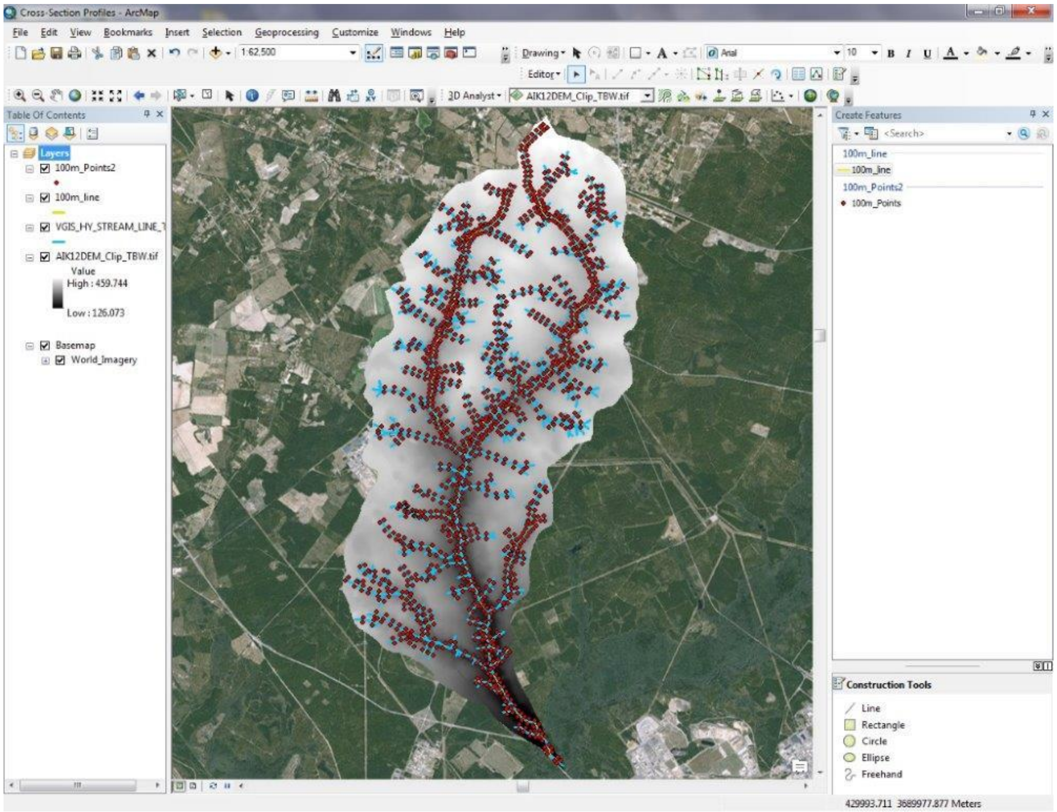


Figure 2-29. Cross sections of Tims Branch and streams across the entire watershed.

Subtask 3.2. Application of GIS Technologies for Hydrological Modeling Support

- FIU drafted and submitted a paper for the Waste Management Symposia to be held on March 6-10, 2016 in Phoenix, AZ. The paper was accepted for presentation at the conference.

  Abstract: 16202 – Using GIS for Processing, Analysis and Visualization of Hydrological Model Data
  Session: 071B – Posters: Environmental Remediation Field Investigation and Remediation
  Date: Tuesday, March 08, 2016
  Time: 1:30 - 5:00 PM

- New GIS shapefiles and maps were created for the expanded study domain which now incorporates the entire Tims Branch watershed as opposed to just the portion that lies within the SRS boundary. Maps of some of the significant model input parameters are shown below for the original study domain and the new revised study domain.

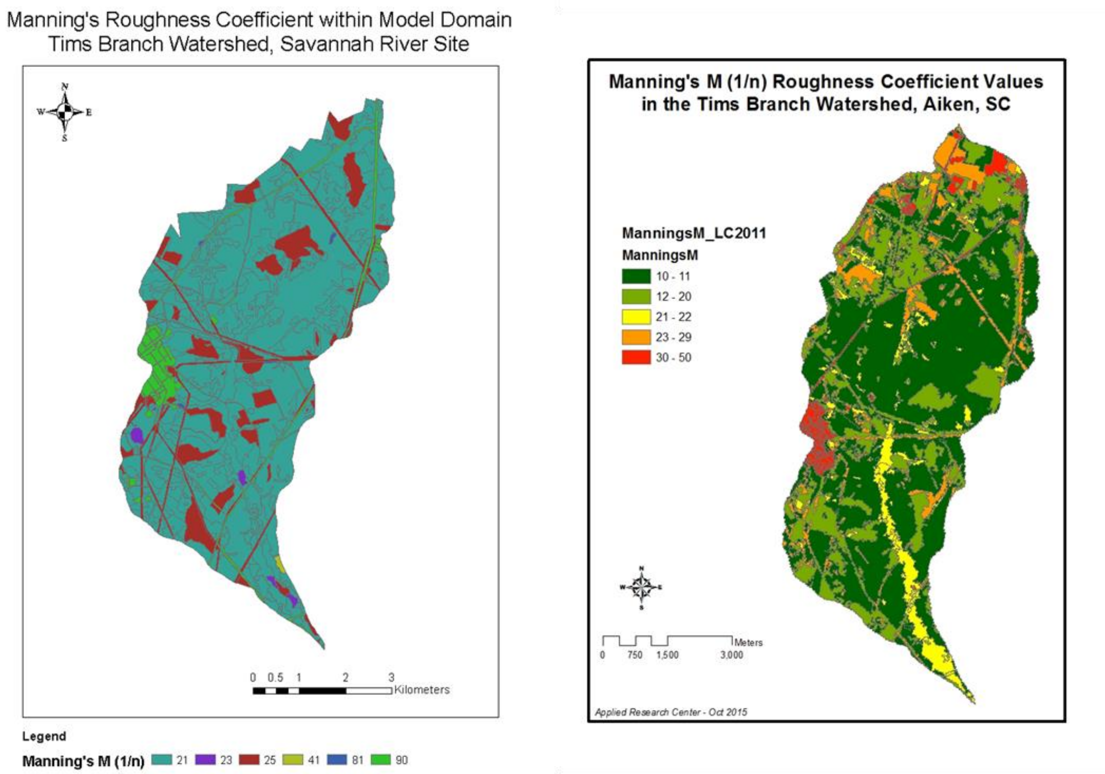


Figure 2-30. Map of the Manning's M (1/n) roughness coefficients in the Tims Branch watershed for the original study domain (left) and the new revised study domain (right).

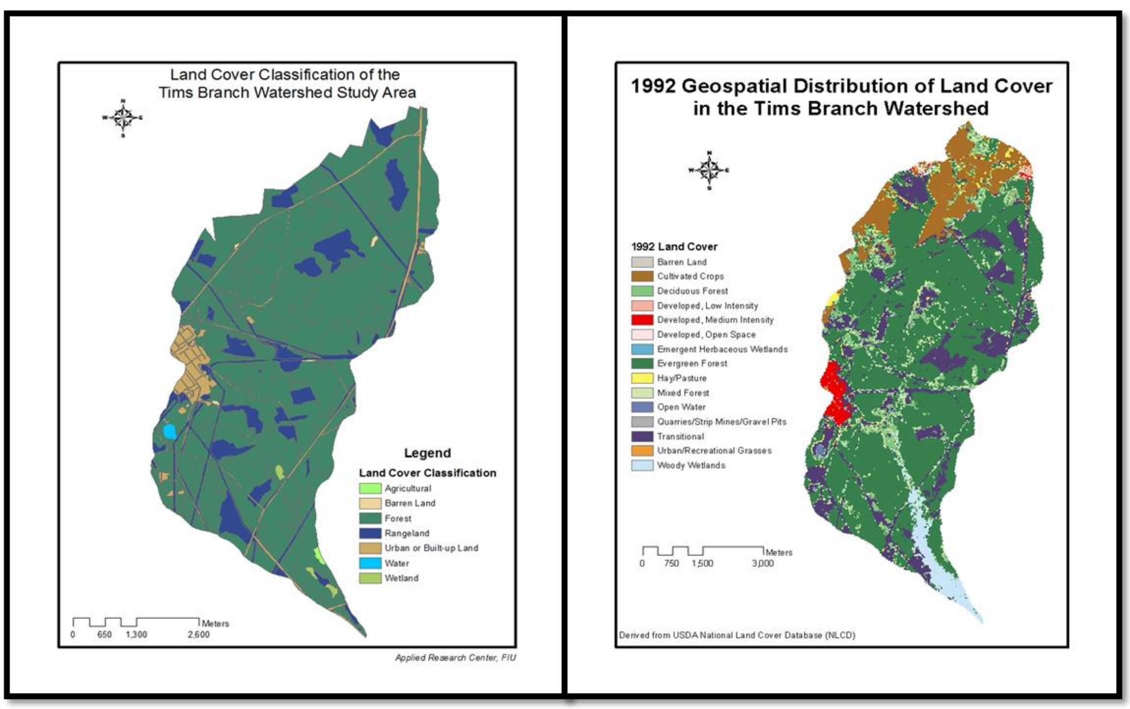


Figure 2-31. Map of the land cover classification in the Tims Branch watershed for the original study domain (left) and the new revised study domain (right).

- Basic preliminary geospatial analyses were conducted on the land cover data that was downloaded from the National Land Cover Database (NLCD). Land cover grid files for the years 1992, 2001, 2006 and 2011 were converted to GIS shapefiles and the attribute data used to graph the land cover changes. Thematic maps of the land cover for each year were also created. Further manipulation of the downloaded rasters and converted shapefiles is planned for a more in-depth comparative data analysis.

Land Cover	1992 (% Total)	2001 (% Total)	2006 (% Total)	2011 (% Total)
Open Water	0%	0%	0%	0%
Developed, Open Space	0%	1%	1%	1%
Developed, Low Intensity	0%	1%	1%	1%
Developed, Medium Intensity	1%	1%	1%	1%
Developed, High Intensity	0%	0%	0%	1%
Barren Land	0%	3%	3%	3%
Quarries/Strip Mines/Gravel Pits	0%	0%	0%	0%
Transitional	16%	0%	0%	0%
Deciduous Forest	5%	0%	0%	0%
Evergreen Forest	56%	63%	54%	53%
Mixed Forest	5%	1%	1%	1%
Shrub/Scrub	0%	13%	16%	23%
Herbaceuous	0%	6%	11%	6%
Hay/Pasture	1%	2%	2%	1%
Cultivated Crops	9%	1%	1%	1%
Urban/Recreational Grasses	0%	0%	0%	0%
Woody Wetlands	3%	5%	5%	5%
Emergent Herbaceuous Wetlands	0%	1%	1%	1%

Figure 2-32. Percentage (%) Distribution of Land Cover for 1992, 2001, 2006 and 2011 derived from the National Land Cover Database.

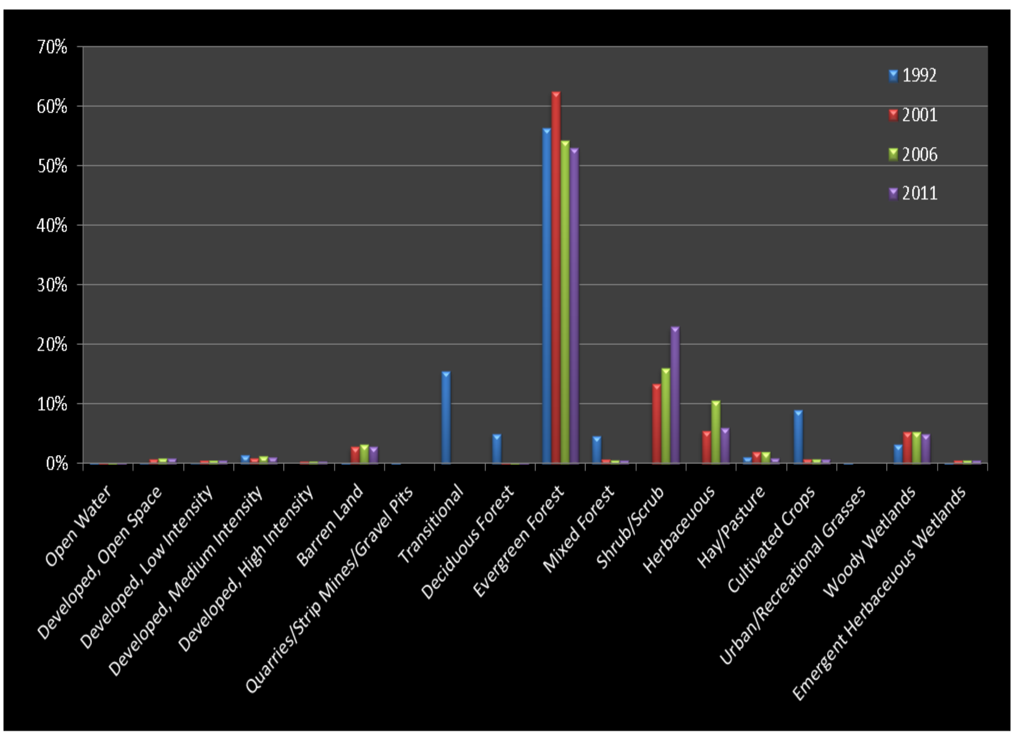


Figure 2-33. Percentage (%) Distribution of Land Cover for 1992, 2001, 2006 and 2011 derived from the National Land Cover Database.

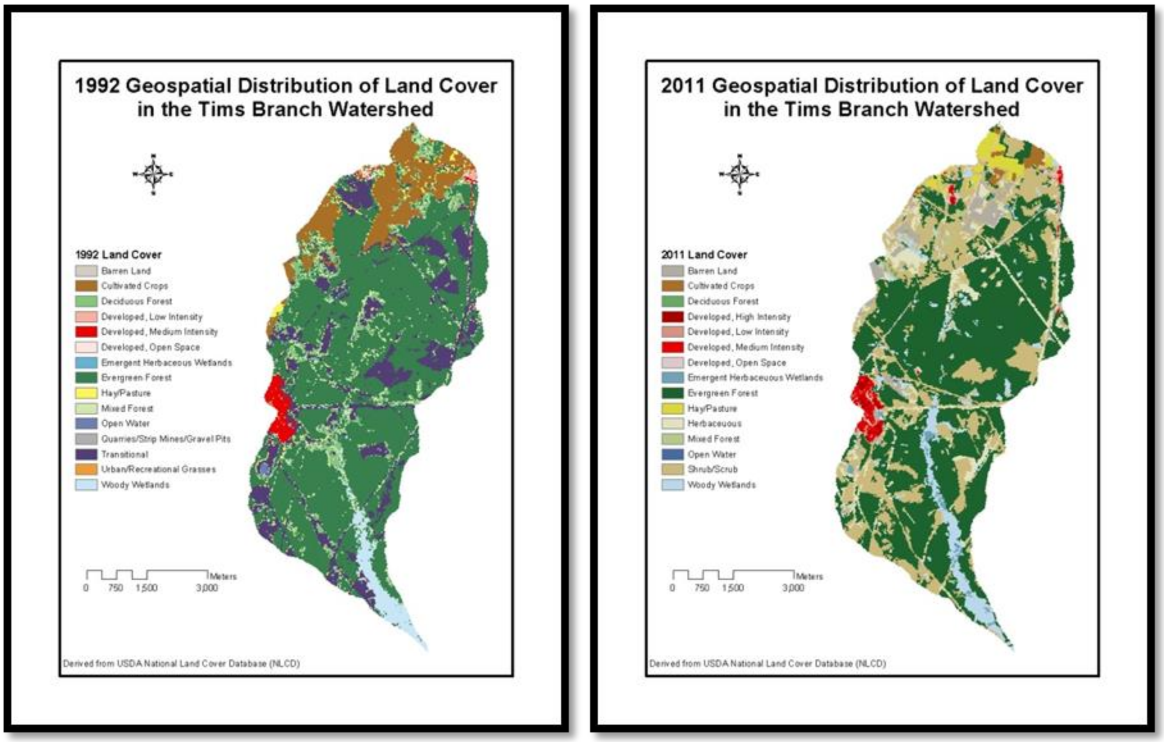


Figure 2-34. The maps above show the geospatial distribution the land cover classes existing in 1992 and 2011. Significant differences observed: (1) Introduction of high intensity developed land in 2011; (2) Large increase in shrubs between 1992 and 2011; (3) Disappearance of transitional vegetation between 1992 and 2011; (4) Decrease in cultivated crops between 1992 and 2011.

- FIU continued the development of revised GIS shapefiles and maps for the expanded study domain which now incorporates the entire Tims Branch watershed as opposed to just the portion that lies within the SRS boundary.
- The Tims Branch stream network was derived from the USGS National Hydrography Dataset (NHD), which is a GIS-based digital vector dataset that represents drainage network features such as rivers, streams, canals, lakes, ponds, coastline, dams, and stream gauges. The NHD was delineated and georeferenced to the USGS 1:24,000 scale topographic base map meeting National Map Accuracy Standards (NMAS). The NHD files contain flow network attributes imbedded in the data that allow GIS scientists to trace flow directions and facilitate delineation of features such as nodes, cross sections and cross section profiles required for developing the stream flow model using MIKE 11. The NHD and the USGS Watershed Boundary Dataset (WBD) can be used by a GIS with other data themes such as elevation, boundaries, transportation, and control structures to produce general reference maps, however, because they both use a linked addressing system based on reach codes and other basic NHD and WHD features, in-depth geospatial analyses are possible to study cause and effect relationships, such as how a source of poor water quality upstream might affect a fish population downstream.
- A poster entitled "*GIS as an Integration Tool for Hydrologic Modeling: Spatial Data Management, Analysis and Visualization*" was prepared and presented at the American Geophysical Union (AGU) Fall 2015 meeting in December by Dr. Shimelis Setegn (Figure 2-35).

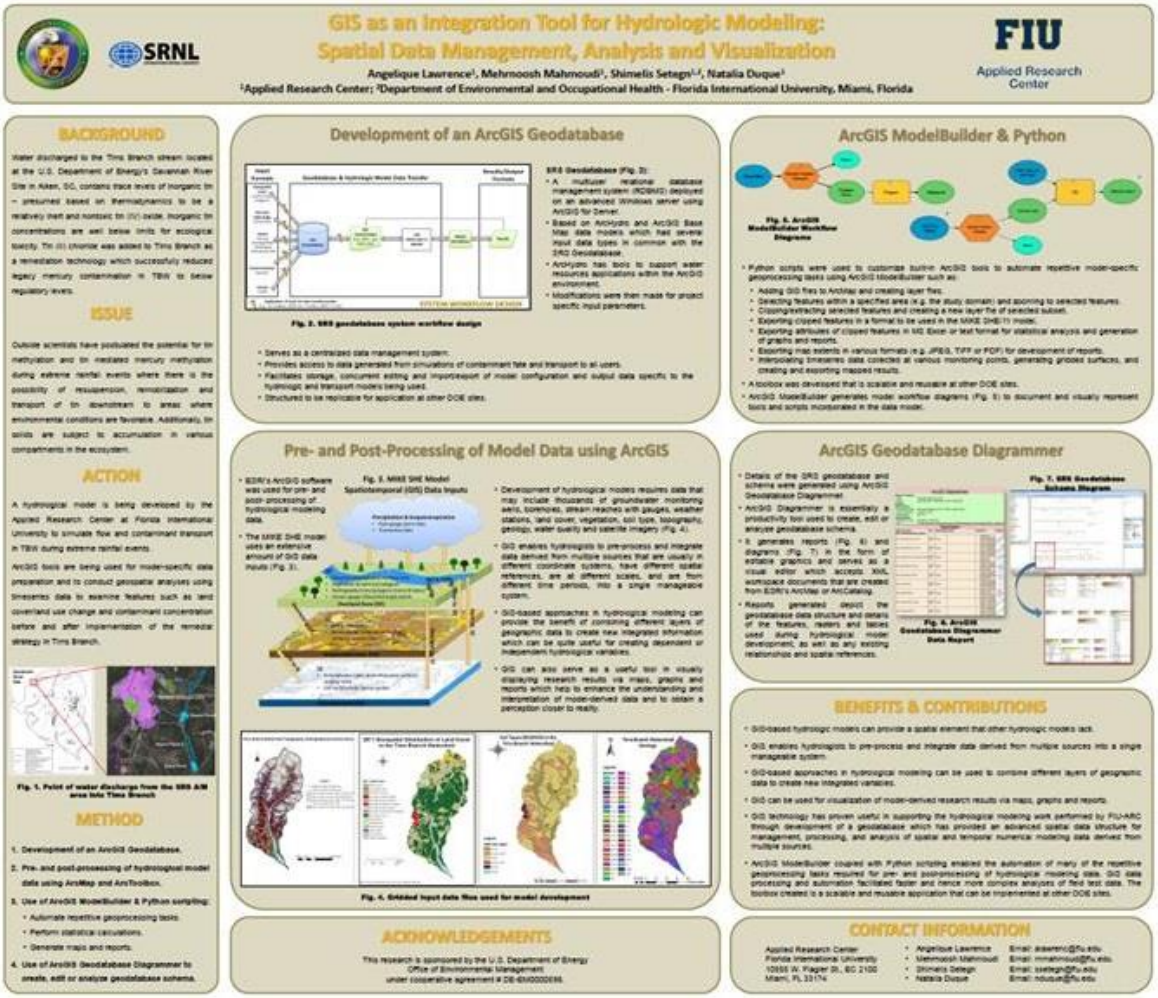


Figure 2-35. Poster presented at American Geophysics Union (AGU) Fall 2015 meeting.

- FIU continued the development of revised GIS shapefiles and maps for the expanded study domain which now incorporates the entire Tims Branch watershed (TBW) as opposed to just the portion that lies within the SRS boundary. The following outlines the procedure for development of a geospatial layer to represent the initial water depth in Tims Branch watershed:
    - A DEM of the Tims Branch watershed was converted from feet to meters by multiplying the raster file by 0.3048 and then clipping the file to the TBW domain.
    - A point near outfall A-11 was used as a reference point for the initial water depth.
        - Location: 432,839.875; 3,688,280.806 meters
        - Elevation: 71.63 meters

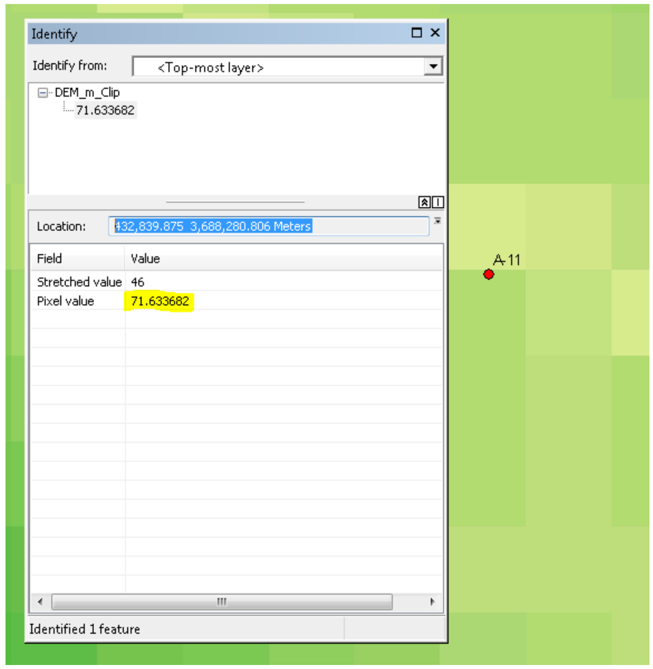


Figure 2-36. Reference point for initial topographic elevation.

- A value of 10 cm was then added to the ground surface elevation at the selected reference point. This new elevation is the water elevation (H). A uniform potentiometric line was assumed along the domain.
  - Water elevation along the domain: 71.73 meters (above mean sea level)

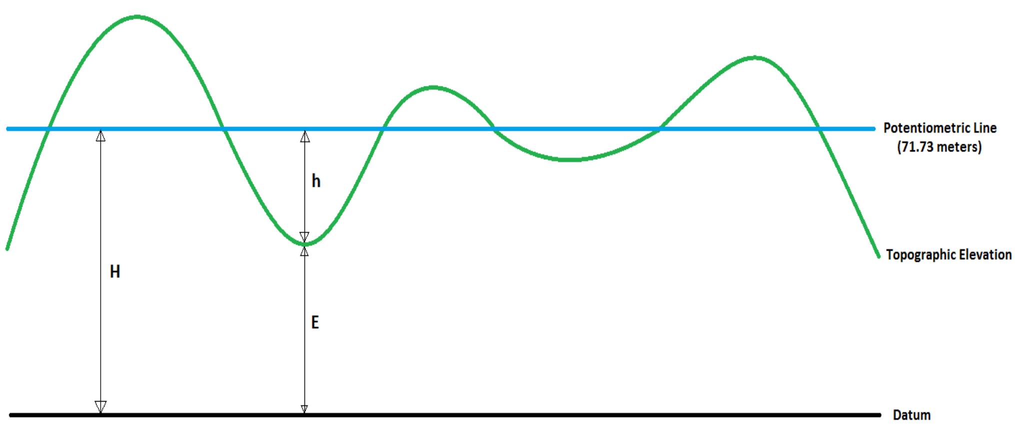


Figure 2-37. Topographic elevation.

- Using the ArcGIS raster calculator the following operation was then performed on the entire raster file:

$$h = H - E$$

Where: h = initial water depth, H = water elevation (from datum), and E = ground surface elevation.

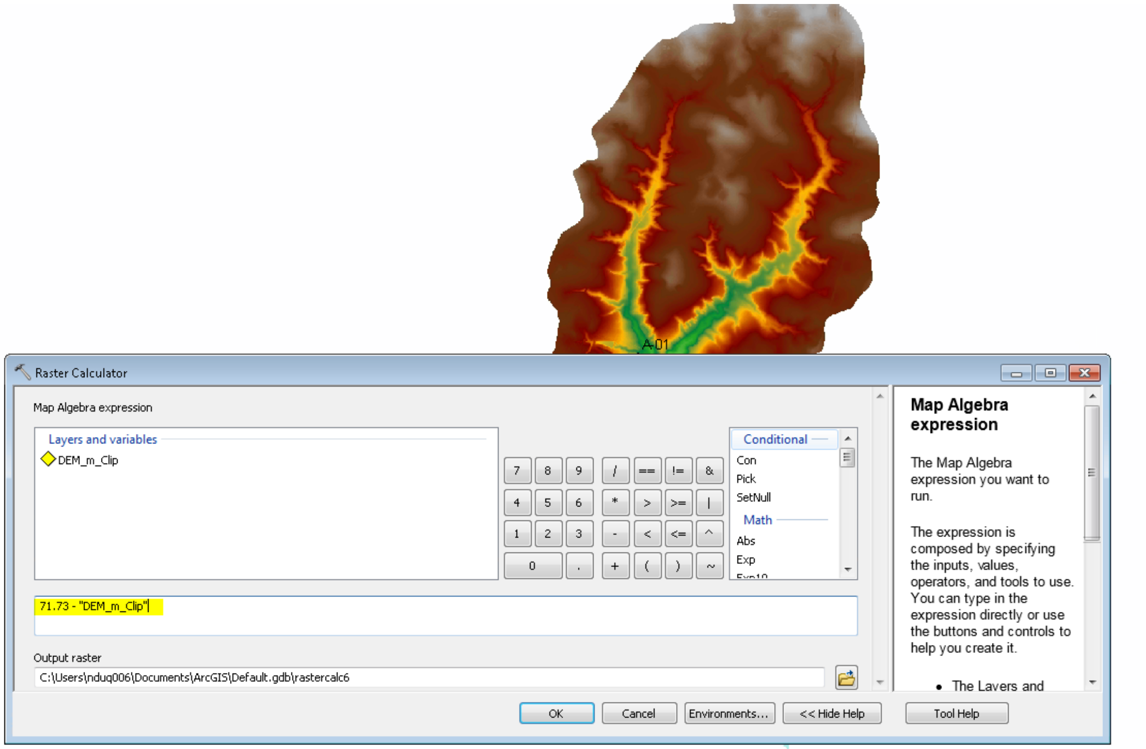


Figure 2-38. ArcGIS raster calculator.

- The resulting water depth file contained some negative numbers. These values refer to the locations with higher elevation which mainly stay dry. Therefore, water depth at these locations is zero. Using the ArcGIS raster calculator, a value of zero (0) was assigned to all water elevation with negative values in the resulting raster.

  Expression: Con("water_depth" < 0, 0 , "water_depth")

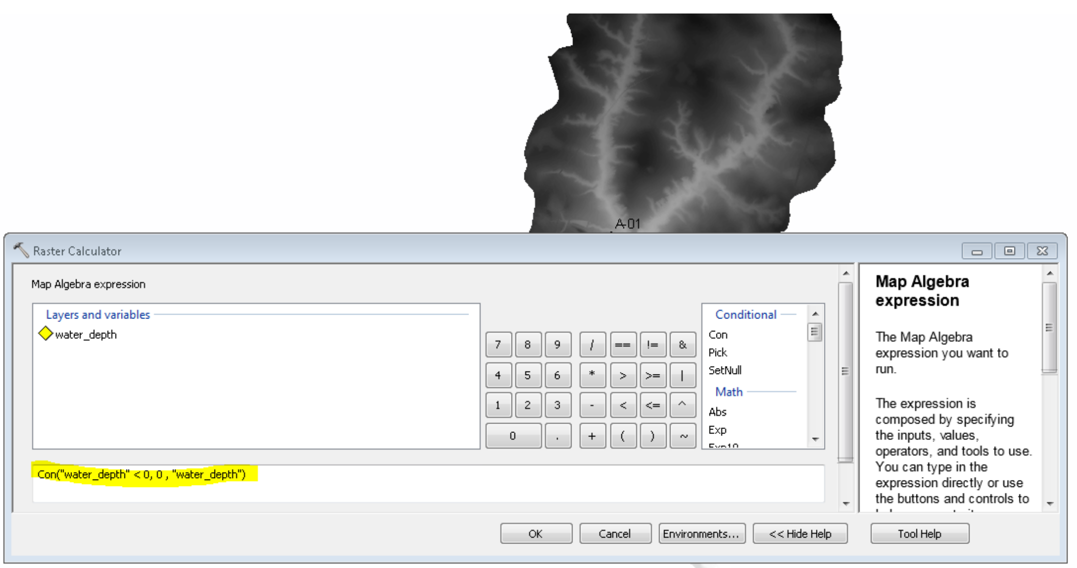


Figure 2-39. ArcGIS raster calculator.

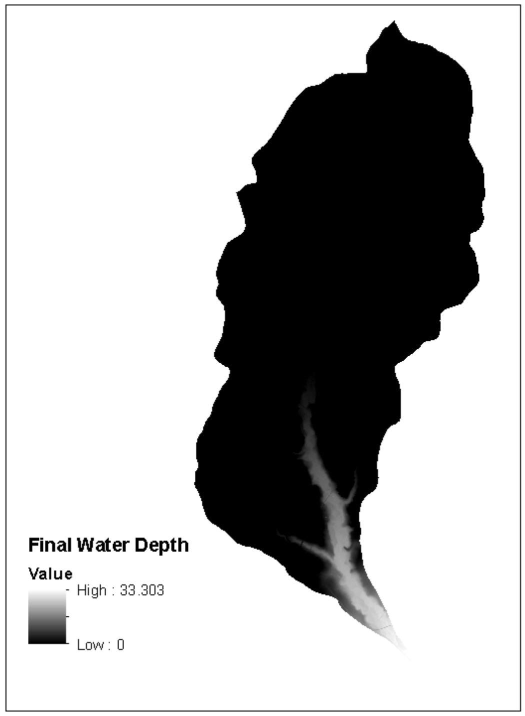


Figure 2-40. Final water depth.

- Finally, using the "Raster to Point" tool in ArcToolbox, the resulting water depth raster file was converted into a GIS point shapefile which can be input into the MIKE SHE model.

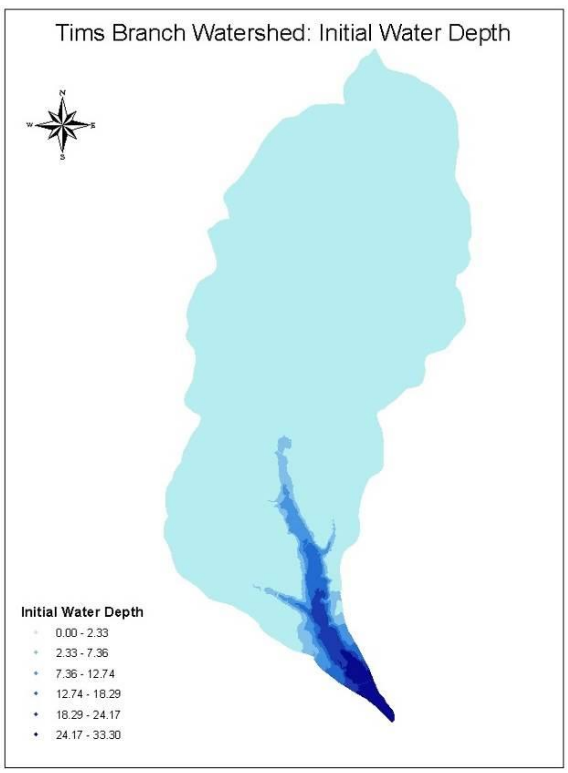


Figure 2-41. GIS point shapefile for initial water depth.

Subtask 3.3. Biota, Biofilm, Water and Sediment Sampling in Tims Branch

- The proposed work outlined in the Project Technical Plan for this task was approved by Brian Looney (SRNL) and John Seaman (SREL). A new ISCO system was ordered by SREL for a planned sediment sampling effort in Tims Branch/Steed Pond. This unit will be equipped with a velocity meter and geochemical probes. A request was made by FIU for some additional water and sediment samples to be collected for Sn analysis and to acquire additional data parameters that could be used for the Tims Branch modeling effort. The last update in December was that the automated monitoring stream system was due to be delivered soon. In addition, approximately two dozen sediment samples were collected from the Steed Pond/Tims Branch of which only two were found with elevated contaminants.

Task 4: Sustainability Plan for the A/M Area Groundwater Remediation System

<u>Task 4 Overview</u>

The research and analysis performed under this task is being performed to support DOE EM-13 (Office of D&D and Facilities Engineering) under the direction of Mr. Albes Gaona, program lead for DOE's Sustainable Remediation Program.

Subtask 4.1. Sustainable Remediation Analysis of the M1 Air Stripper

The goal of the SRS M Area groundwater remediation system is to provide hydraulic containment of the contaminated groundwater. The focus of FIU's Green and Sustainable Remediation (GSR) analysis is to provide analyses and recommendations for improving the electro-mechanical components and operations of this remediation system (e.g., air stripper, pumps). These improvements should result in a more sustainable system that saves energy, cuts greenhouse gas emissions, and saves financial and other resources.

- FIU is developing analyses and improvements for the pumps and air stripper in particular.
- FIU is developing a commercial quote for a solar voltaic system with batteries for electrical power for the groundwater remediation system. The advanced, commercial batteries are much lower in cost than 3 years ago. Pumps and the stripper blower motor are expected to be the primary users of electrical power in the system.
- The following solar companies were contacted to provide a price quote on such a solar system for the groundwater remediation system with a continuous load of 150 kW:
    - Sunwize Power & Battery
    - Alder Energy Systems
    - Southern Atlantic Solar
    - Baker Renewable Energies
    - Hannah Solar Government Service
- FIU expects quotes by the first week in November.

FIU became aware that there is a current proposal to install a photovoltaic solar system at SRS and has asked SRNL for the proposal to ensure our proposed solar systems are aligned with SRS goals and plans for solar power usage at the site.

FIU is developing a draft report by December 18, 2015 for Savannah River Site entitled, "A Sustainability Analysis for the M1 Air Stripper and Pumps of the M Area Groundwater Remediation System at the Savannah River Site."

This will complete research on this task as it closes. Comments on the draft report will be collected in January and a final report will be sent to SRNL in late January. In addition, DOE Fellow and student, Yoel Rotterman, will present a student poster and a short paper at Waste Management Symposium (March 6-10, 2016) based upon data and analyses in this report.

During November FIU received 2 quotes for solar power systems capable of powering the M Area Groundwater Remediation System. FIU is working on getting modifications to these quotes as well as additional quotes for turnkey photovoltaic systems. Systems connected to the grid as well as ones with energy storage that does not require grid connection are being evaluated.

Much of the report was written this past month including sections on the history of contaminant removal; the packing materials; the pumps; and the blower. Some key concepts in the report include:

- A solar power, photovoltaic system capable of powering the air stripper and pumps in the M Area groundwater remediation system;
- Rationale for optimizing the pumping rates and schedules of individual recovery wells;
- Preferred pumps to install when current pumps require replacement;
- Use of direct current power for air blower system allowing adjustment to any air flow speed and lower electrical power consumption.

FIU is awaiting additional information from SRNL in December on the column packing material, additional air stripper information, and summary data on an analysis done in 2015 by SRNL for a photovoltaic system at SRS but at a different location.

FIU received extensive data on solar irradiance and efficiency for a solar photovoltaic system in South Carolina located near SRS. FIU will use this actual data to modify quotes from solar companies that assume an average of 5 hours of full sunlight irradiance throughout the year.

- FIU submitted a draft report on December 18, 2015 for Savannah River Site entitled, "*A Sustainability Analysis for the M1 Air Stripper and Pumps of the M Area Groundwater Remediation System at the Savannah River Site*."
- This completes research on this task as it closes. Comments on the draft report will be collected in January and a final report will be sent to SRNL in late January. In addition, DOE Fellow Yoel Rotterman will present a student poster at Waste Management Symposium (March 6-10, 2016) based upon data and analyses in this report.

Task 5: Remediation Research and Technical Support for WIPP

<u>Task 5 Overview</u>

This new task is in collaboration with research scientist Donald Reed in support of Los Alamos National Laboratory's field office in Carlsbad, New Mexico. This research center has been tasked with conducting experiments in the laboratory to better understand the science behind deep geologic repositories for the disposal of nuclear waste. The majority of their work is conducted in high ionic strength systems relevant to the Waste Isolation Pilot Plant (WIPP) located nearby. WIPP is currently the only licensed repository for the disposal of defense waste in the world. However, the facility is not currently operating, following an airborne release from a waste drum which failed to contain waste following an exothermic reaction of the waste. This was due to incompatibility of mixed waste received from LANL (organic adsorbent mixed with nitrate salt waste). Although off-site releases of $^{239/240}Pu$ and ^{241}Am were detected slightly above background, they were still below levels deemed unsafe to the public. FIU-ARC is now initiating a new task to support basic research efforts requested to update risk assessments for the WIPP site.

The objective of this task is to support LANL researchers in the basic science research required to address concerns in risk assessment models for the re-opening of the WIPP site for acceptance of defense waste.

<u>Task 5 Quarterly Progress – October 1 – December 31, 2015</u>

- On October 20-21, Dr. Donald Reed, a Team Leader of Actinide Chemistry and Repository Science Program at Los Alamos National Laboratory, visited ARC, participated as a judge in the DOE Fellows poster competition and presented a lecture for ARC staff and students on actinide solubility and speciation in the Waste Isolation Pilot Plant (WIPP) Transuranic Repository. This lecture was conducted within the framework of the Applied Research Center Distinguished Lecture Series.
- ARC staff also participated in a discussion with Dr. Reed on the research conducted by ARC for the DOE EM sites and the collaboration between ARC and LANL to advance science of WIPP research. Dr. Reed and ARC staff both expressed interest in the collaboration to advance science of WIPP research, which is a high priority mission for the DOE HQ.
- FIU is working to create collaborative research projects with the Actinide Chemistry and Repository Science Program (ACRSP) team in Carlsbad to establish a greater understanding of the effects of ionic strength and brine composition on the sorption of actinides in the Waste Isolation Pilot Plant (WIPP) and DOE-EM relevant host materials and synthetic groundwaters. This collaborative research will be initiated at the Los Alamos National Lab's Carlsbad facilities where Dr. Hilary Emerson (postdoctoral staff member at FIU's Applied Research Center) will be working under the direction of Dr. Don Reed and Dr. Timothy Dittrich. All work performed at the LANL facilities will be done under the existing CEMRC/NMSU safety program under which all LANL ACRSP research activities are currently performed. This research will advance science in high ionic strength systems, which were not well researched in the past. The first step in this collaboration will be to create a task focusing on sorption of actinides as a function of ionic strength (0.001 – 7.5 M NaCl, GWB and ERDA-6 groundwaters), pH (6 – 10), and host material (dolomite). The benefit of this collaboration to DOE-EM/WIPP will be an improved understanding of the radionuclides sorption behavior helping to interpret site-specific data and the relationship with FIU leading to joint peer-review publications on the topic.
- Some of the experimental protocols from experiments conducted at LANL facilities will be transferred to ARC to allow studies to be conducted in ARC's laboratories.
- During the month of December, dates for Dr. Hilary Emerson to travel to Los Alamos National Lab's CEMRC Carlsbad facilities were finalized (currently scheduled for

February 15 – March 8, 2016). During the month of January, travel, housing plans and radiation working training will be finalized with collaborators at Los Alamos.

- During January, dolomite samples will be received and analyzed at FIU ARC by XRD, SEM and BET surface area. In addition, technical plans for the column experiments to be performed at the CEMRC facility in Carlsbad will be finalized with collaborators at LANL. Initial experiments to be accomplished during the first visit to Carlsbad will focus on the fate of trivalent actinides and lanthanides (Pu, Am, Eu, Nd) at variable ionic strength and relevant conditions to WIPP.

Milestones and Deliverables

The milestones and deliverables for Project 2 for FIU Year 6 are shown on the following table. Milestone 2015-P2-M1 was completed with the submittal of draft papers to the Waste Management 2016 Symposium. The Task 4 deliverable, "Draft sustainable remediation report for the M1 air stripper," was submitted by its due date 12/18/2015. The Task 3 milestone (2015-P2-M2) "Complete refinement of MIKE SHE model configuration parameters for the simulation of overland flow using revised model domain (Subtask 3.1)" was also completed by its due date 12/30/2015.

FIU Year 6 Milestones and Deliverables for Project 2

Task	Milestone/ Deliverable	Description	Due Date	Status	OSTI
Project	2015-P2-M1	Submit draft papers to Waste Management 2016 Symposium	11/6/2015	Complete	
Task 1: Hanford Site	Deliverable	Progress report on the experimental results on autunite mineral biodissolution (Subtask 1.2)	2/15/2016	On Target	OSTI
	Deliverable	Progress report on batch experiments for ammonia injection task (Subtask 1.3.1)	6/22/2016	On Target	OSTI
	Deliverable	Literature Review of Geophysical Resistivity Measurements and Microbial Communities (Subtask 1.3.3)	3/18/2016	On Target	
Task 2: SRS	Deliverable	Progress report on batch experiments on sodium silicate application in multi-contaminant systems (Subtask 2.1)	4/11/2016	On Target	OSTI
	Deliverable	Progress report on the synergy between colloidal Si and HA on the removal of U(VI) (Subtask 2.4)	4/21/2016	On Target	OSTI
	Deliverable	Progress report on column experiments to investigate uranium mobility in the presence of HA (Subtask 2.5)	5/20/2016	On Target	OSTI
Task 3: Tims	2015-P2-M2	Complete refinement of MIKE	12/30/2015	Complete	

Branch		SHE model configuration parameters for the simulation of overland flow using revised model domain (Subtask 3.1)			
	2015-P2-M3	Complete input of MIKE SHE model configuration parameters for simulation of evapotranspiration (Subtask 3.1)	2/29/2016	On Target	
	2015-P2-M4	Complete input of MIKE SHE model configuration parameters for simulation of unsaturated flow (Subtask 3.1)	3/31/2016	On Target	
	Deliverable	Progress Report for Subtask 3.1: Modeling of surface water and sediment transport in the Tims Branch ecosystem	4/29/2016	On Target	OSTI
	Deliverable	Progress Report for Subtask 3.2: Application of GIS technologies for hydrological modeling support	4/29/2016	On Target	OSTI
	2015-P2-M5	Complete input of MIKE SHE model configuration parameters for simulation of flow in the saturated zone (Subtask 3.1)	6/30/2016	On Target	
Task 4: Sustainability Plan	Deliverable	Draft sustainable remediation report for the M1 air stripper	12/18/2015	Complete	OSTI

Work Plan for Next Quarter

Task 1: Remediation Research and Technical Support for the Hanford Site

Subtask 1.1 – Sequestering Uranium at the Hanford 200 Area Vadose Zone by in situ Subsurface pH Manipulation using NH_3 Gas

- Continue with isopiestic measurements.
- Prepare new samples for 100mM sodium silicate and re-prepare NaCl standard in another cup due to cup's rusting.
- Set up flow –through experiment to investigate for solid phase's dissolution.
- Complete preparation of new samples followed by initial solid and liquid phase analysis by SEM/EDS and KPA, respectively. The results of this initial analysis will determine the methods used for continued characterization attempts, which may include XRD, EMPA, and/or TEM analysis
- Complete proceedings paper for WM-2016

Subtask 1.2. Investigation on Microbial-Meta-Autunite Interactions - Effect of Bicarbonate and Calcium Ions

- Determination of Ca and P concentrations in the samples by means of ICP-OES

- Perform protein analysis (bicinchoninc acid assay) by means of UV-vis spectroscopy
- Analyze autunite samples exposed to bacterial presence by means of SEM/EDS.
- Complete proceedings paper for the WM-2016

Subtask 1.3. Evaluation of Ammonia Fate and Biological Contributions During and After Ammonia Injection for Uranium Treatment

- The batch sorption experiments will continue to be optimized to understand sorption of U and NH_3 under conditions relevant to the Hanford Site based on the lessons learned.
- Speciation modeling will be completed for comparison with the experimental results.
- Statistical analysis will be used to compare the samples that had pH adjusted by NaOH versus by NH_4OH (t-test).

Task 2: Remediation Research and Technical Support for Savannah River Site

Subtask 2.1. FIU's Support for Groundwater Remediation at SRS F/H –Area

- Perform kinetic experiments on the retention of U(VI) by SRS F/H area soil after sodium silicate amendment (restoration of circumenutral conditions)
- Perform batch comparison experiments in sodium silicate amended samples containing SRS F/H area soil, as well as pure minerals (quartz and kaolinite)
- Assess the longevity of the remediation methods (sodium silicate amendment) in batch experiments

Subtask 2.2 – Monitoring of U(VI) Bioreduction after ARCADIS Demonstration at F-Area

- Conduct Minteq speciation modeling to confirm the results from the batch experiment and to determine in which conditions the iron precipitates would likely form.
- Finalize sulfate analysis via ion chromatography on the supernatant solutions collected from the microcosm tubes, which were augmented with the 500 ppm sulfate.

Subtask 2.3.Sorption Properties of the Humate Injected into the Subsurface System

- Perform desorption experiment of HumaK sorbed onto SRS sediments at different pH values (4-8).
- Study the effects of salts ($NaNO_3$) on desorption of HumaK.

Subtask 2.4 – The synergetic effect of HA and Si on the removal of U(VI)

- Analyze samples for batches 1, 4 and 7 via ICP-OES to determine the concentrations of silica and iron; process the data and report the findings.
- Initiate experiments with 30 ppm of HA.
- Initiate sediment samples analysis via SEM/EDS
- Complete draft technical paper for Waste Management Symposia-2016.

Subtask 2.5 – Investigation of the migration and distribution of natural organic matter injected into subsurface systems

- Drain columns 1 and 7 and collect sediment for analysis via SEM and TOC analyzer.

- Update the experimental procedure based on the discussion with Dr.Miles Denham and Dr.Brian Looney and initiate new experiments to study the migration and distribution of HA and its effect on uranium mobility.
- Complete draft technical paper for waste management symposia.

Task 3: Surface Water Modeling of Tims Branch

Subtask 3.1. Modeling of Surface Water and Sediment Transport in the Tims Branch Ecosystem

- Submit draft paper to Waste Management 2016 Symposium by 11/6/15.
- Complete refinement of MIKE SHE model configuration parameters for the simulation of overland flow using revised model domain (Subtask 3.1)

Subtask 3.2. Application of GIS Technologies for Hydrological Modeling Support

- Submit draft paper to Waste Management 2016 Symposium by 11/6/15.
- Complete the download and pre-processing of model-specific geospatial and timeseries configuration parameters to fill in data gaps needed for the revised Tims Branch watershed model domain using GIS tools.
- Update GIS maps of Tims Branch hydrology, geology, land use, vegetation cover, topography, etc. for revised model domain.

Subtask 3.3. Biota, Biofilm, Water and Sediment Sampling in Tims Branch

- Coordinate with John Seaman (SREL) to collect data from the sampling effort they planned to conduct between September-October in Tims Branch/Steed Pond. A request was made by FIU for some additional water and sediment samples to be collected for Sn analysis and to acquire additional data parameters that could be used for the Tims Branch modeling effort.

Task 4: Sustainability Plan for the A/M Area Groundwater Remediation System

- Perform analysis of available data of air mass flow rates and contaminant removal efficiency. Determine the overall system efficiency and provide recommendations for the optimal air flow rates which will provide the lowest overall cost of operation.
- Perform analysis of the contaminant concentration from all recovery wells feeding the M1 air stripper and the current packing material and recommend component and process changes to improve the efficiency, lower the electrical energy usage and lessen the environmental footprint of the stripper operation.
- Develop an engineering analysis with input from SRNL and the SRS remediation contractor for improvement to the M1 air stripper.
- Perform analysis of a renewable energy system to power the M1 air stripper to include solar and/or wind. This would include costs for components properly sized and for installation.
- Draft sustainable remediation report for the M1 air stripper.
- Submit draft paper to Waste Management 2016 Symposium by 11/6/15.

Task 5: Remediation Research and Technical Support for WIPP

- Work in collaboration with LANL to identify research topic of interest to advance the science in high ionic-strength systems relevant to WIPP conditions.

Project 3
Waste and D&D Engineering & Technology Development

Project Manager: Dr. Leonel E. Lagos

Project Description

This project focuses on delivering solutions under the decontamination and decommissioning (D&D) and waste areas in support of DOE HQ (EM-13). This work is also relevant to D&D activities being carried out at other DOE sites such as Oak Ridge, Savannah River, Hanford, Idaho and Portsmouth. The following tasks are included in FIU Year 6:

Task No	Task
Task 1: Waste Information Management System (WIMS)	
Subtask 1.1	Maintain WIMS – database management, application maintenance, and performance tuning
Subtask 1. 2	Incorporate new data files with existing sites into WIMS
Task 2: D&D Support to DOE EM for Technology Innovation, Development, Evaluation and Deployment	
Subtask 2.1	D&D Technology Demonstration & Development and Technical Support to SRS's 235-F Facility Decomissioning
Subtask 2.2	Technology Demonstration and Evaluation
Subtask 2.3	Support to DOE EM-13 and the D&D Community
Task 3: D&D Knowledge Management Information Tool	
Subtask 3.1	Web and Mobile Application for D&D Decision Model
Subtask 3.2	Mobile Applications/Platforms for DOE Sites
Subtask 3.3	Development & Integration of International KM-IT Pilot for UK Collaboration
Subtask 3.4	Outreach and Training (D&D Community Support)
Subtask 3.5	Data Mining and Content Management
Subtask 3.6	D&D KM-IT Administration and Support

Task 1: Waste Information Management System (WIMS)

Task 1 Overview

This task provides direct support to DOE EM for the management, development, and maintenance of a Waste Information Management System (WIMS). WIMS was developed to receive and organize the DOE waste forecast data from across the DOE complex and to automatically generate waste forecast data tables, disposition maps, GIS maps, transportation details, and other custom reports. WIMS is successfully deployed and can be accessed from the web address http://www.emwims.org. The waste forecast information is updated at least annually. WIMS has been designed to be extremely flexible for future additions and is being enhanced on a regular basis.

Task 1 Quarterly Progress - October 1 – December 31, 2015

During this performance period, FIU performed database management, application maintenance, and performance tuning to the online WIMS in order to ensure a consistent high level of database and website performance.

FIU drafted and submitted a paper on the Waste Information Management System for the Waste Management Symposia to be held on March 6-10, 2016 in Phoenix, AZ. The paper was accepted for presentation during the conference.

> **Abstract**: 16463 – Waste Information Management System with 2015-16 Waste Streams
> **Session**: 050A - Posters: Waste Characterization
> **Date**: Tuesday, March 08, 2016
> **Time**: 8:30 AM - 12:00 PM

FIU has discussed the 2016 data set collection with DOE. The completed data set is expected to be sent to FIU in the March 2016 timeframe. FIU will integrate and publish the 2016 data on the existing WIMS platform. The need to update WIMS to the current platform and software tools was reiterated to DOE. Currently, the maintenance of WIMS is dependent on the legacy Microsoft.Net framework, database, and application development environment running on a 10-year old machine. If the database, application and the framework are not upgraded, then there is a very high risk that the system will become unavailable to the stakeholders in the future.

Task 2: D&D Support to DOE EM for Technology Innovation, Development, Evaluation and Deployment

Task 2 Overview

This task provides direct support to DOE EM for D&D technology innovation, development, evaluation and deployment. For FIU Year 6, FIU will assist DOE EM-13 in meeting the D&D needs and technical challenges around the DOE complex. FIU will expand the research in technology demonstration and evaluation by developing a phased approach for the demonstration, evaluation, and deployment of D&D technologies. One area of focus will be working with the Savannah River Site to identify and demonstrate innovative technologies in support of the SRS 235-F project. FIU will further support the EM-1 International Program and the EM-13 D&D program by participating in D&D workshops, conferences, and serving as subject matter experts.

Task 2 Quarterly Progress – October 1 – December 31, 2015

DOE Fellows supporting this task include Jesse Viera (undergraduate, mechanical engineering), Janesler Gonzalez (undergraduate, mechanical engineering), Meilyn Planas (undergraduate, electrical engineering), and Orlando Gomez (graduate, physics). DOE Fellow Yoel Rotterman (undergraduate, mechanical engineering) will be supporting this task starting in January 2016.

Task 2.1.1: Incombustible Fixatives

FIU drafted and submitted a paper for the incombustible fixatives research for the Waste Management Symposia to be held on March 6-10, 2016 in Phoenix, AZ. The paper was accepted for presentation at the conference.

Abstract: 16393 - Incombustible Fixatives for D&D Activities
Session: 025 - Application of Innovative D&D Technologies Part 1
Date: Monday, March 07, 2016
Time: 1:30 PM - 5:00 PM

The objective of this research task is to improve the operational performance of fixatives by enhancing their fire resiliency. Most fixatives begin to see degradation between 200-400 degrees, at which time radioisotopes could potentially be released into the environment. The layering or combining of an intumescent coating with the fixative is being investigated as a way to mitigate the release of radioisotopes during fire and/or extreme heat conditions.

Since 9/11/2001, there have been significant improvements in fire retardant/fire resistant technologies, with intumescent coatings being at the forefront of this development. Intumescent coatings develop a thick char to insulate the substrate and protect it from fire and extreme heat conditions. Applying that technology to fixatives through layering and combining should increase its fire resiliency and mitigate the risk of contamination under those extreme conditions.

FIU completed Phase I of the proof-of-principle series of tests (milestone 2015-P3-M2.1) which were designed to expose a selection of fixatives, strippable coatings, and decontamination gels alongside fixatives layered with an intumescent coating to a direct flame source (Figures 3-1 to 3-3). At the conclusion of Phase I, the initial results were promising. All of the fixatives-only test coupons that were exposed to the direct flame began to display significant degradation in less than two minutes, with some of the fixatives actually igniting and becoming flammable upon contact with a direct flame source. However, the fixatives that were layered with an intumescent coating developed a thickening char as designed (intumescent coatings, when exposed to an extreme heat source, expand and develop an insulating char), thereby protecting both the fixative and substrate for upwards of 35 minutes from the direct flame. Most promising was the discovery that in most instances, the fixative under the intumescent coating was relatively intact with no noticeable signs of degradation. There was sufficient data to support further testing and the proof-of-concept that the fire resiliency of fixatives used in D&D activities can be significantly enhanced by layering with an intumescent coating, thereby mitigating any potential release of radioisotopes during fire or extreme heat conditions.

Figure 3-1. ARC constructed a platform that would allow the safe execution of a series of proof-of-principle experiments. The far left panel is wood only, the middle panel is wood with a leading industry fixative, and the far right panel is wood with a leading industry fixative layered with an intumescent coating.

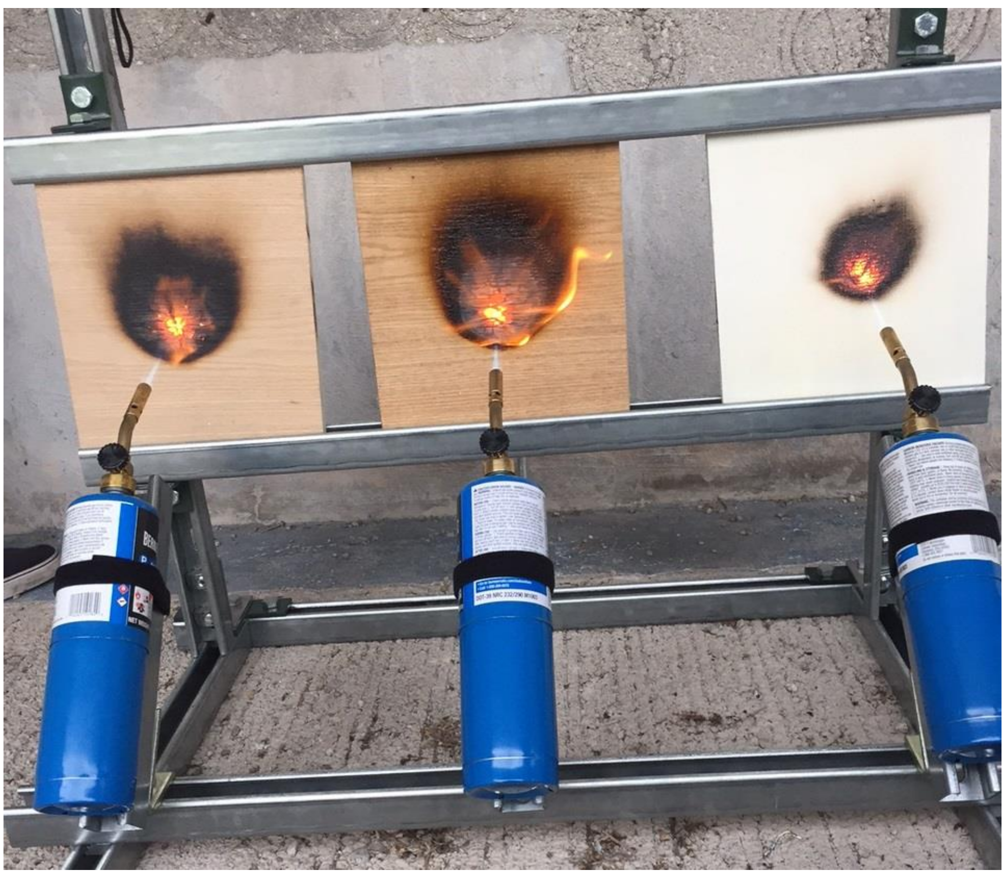


Figure 3-2. At the 2-minute mark, both the left and middle panels began to show significant degradation and burn through while the right panel began to develop a thickening char from the expanding intumescent coating.

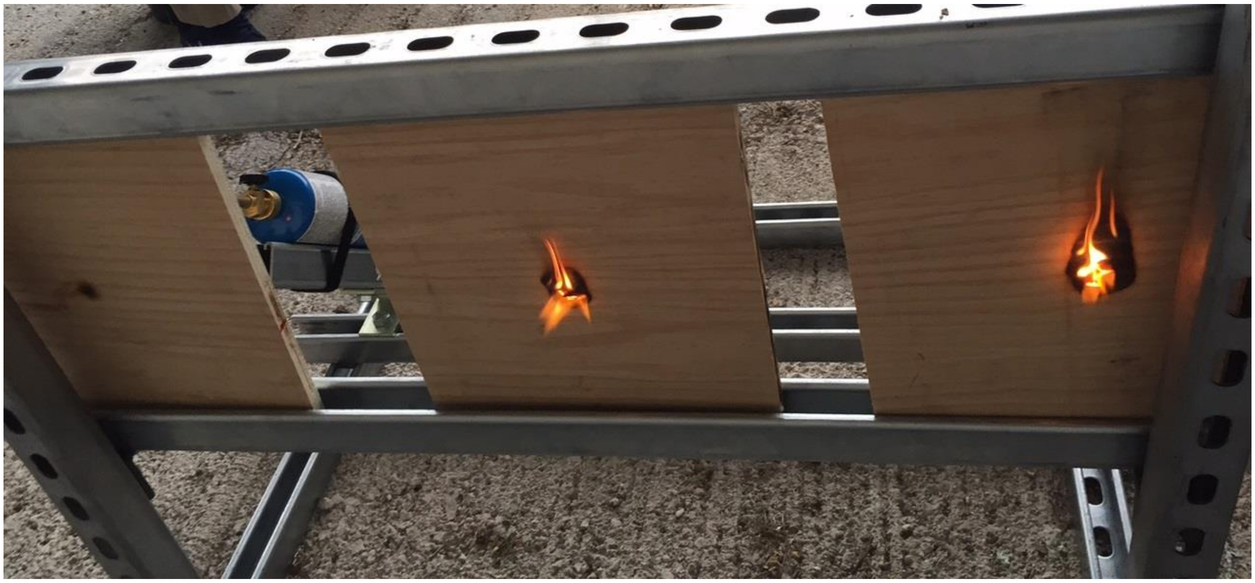


Figure 3-3. At 5-minute mark, both the left and middle panels displayed serious degradation and burn through while the right panel with an intumescent coating remained largely intact.

Due to results from the Phase I proof of principle series of tests, there was sufficient data to support the hypothesis that fire resiliency of fixatives used in D&D activities can be enhanced by layering them with an intumescent coating, and therefore preparations began for Phase II of the Test Plan. Phase II is intended to conduct controlled tests on uncontaminated coupons using different substrates by incrementally increasing the temperatures in a muffle furnace. Similar to Phase I, ARC is applying each of the 5 fixatives to coupons in accordance with the manufacturer's directions (fixative-only coupons), and will then apply the various fixatives plus a layer of intumescent coating to a second series of coupons (fixatives plus intumescent coating). Coupons include 4" x 4" red oak and sheet metal in order to facilitate placement in the muffle furnace. The same application and curing procedures as in Phase I is being followed.

ARC is subjecting the cured fixative-only coupons as well as the fixative-plus-intumescent-coating coupons to incrementally increasing temperatures (e.g.; 100°F, 200°F, 300°F, and so forth) in a muffle furnace for a time period of 2 hours at each temperature, allowing the coupons to cool for 1 hour, and then recording the effect of the heat on the fixative. Effects observed and recorded include the amount of weight lost, thickness degradation, and a visual inspection for evidence of failure, including peeling, cracking, blistering, abnormal discoloration, or loss of adhesion. The intent is to determine at what temperature each of the designated fixatives begin to breakdown and display negative effects that could degrade its intended purpose, specifically fixing radioactive contaminants. Once the temperature threshold has been reached for a particular coupon, the incremental heating process ceases for that sample and the results are recorded.

ARC will follow the same procedures for the second series of coupons (fixative plus intumescent coating), and the results will be consolidated and analyzed as well. Again, the intent of this phase is to identify the temperature at which each product begins to degrade, thus making it vulnerable to a potential release of a radioactive contaminant, and then observe whether that same fixative layered with an intumescent coating maintains its integrity longer and under more extreme heat conditions.

Preliminary results for the fixatives baselined to date demonstrated significant degradation at temperature ranges between 250-300° degrees Fahrenheit within five minutes of exposure. During this phase of testing, FIU is using a product called GloGerm to simulate a radioactive contaminant. GloGerm is useful to "track" particles on the coupon and in the fixative during degradation. When exposed to a black light, the GloGerm glows.

Figure 3-4 shows a sheet metal coupon to which the GloGerm gel was applied and an industry fixative was then applied to the manufacturer's recommended thickness. Once allowed to cure, the coupon was placed in a muffle furnace and exposed to heat. At 250° F, the fixative began to melt and flow into the petri dish along with the simulated contaminant.

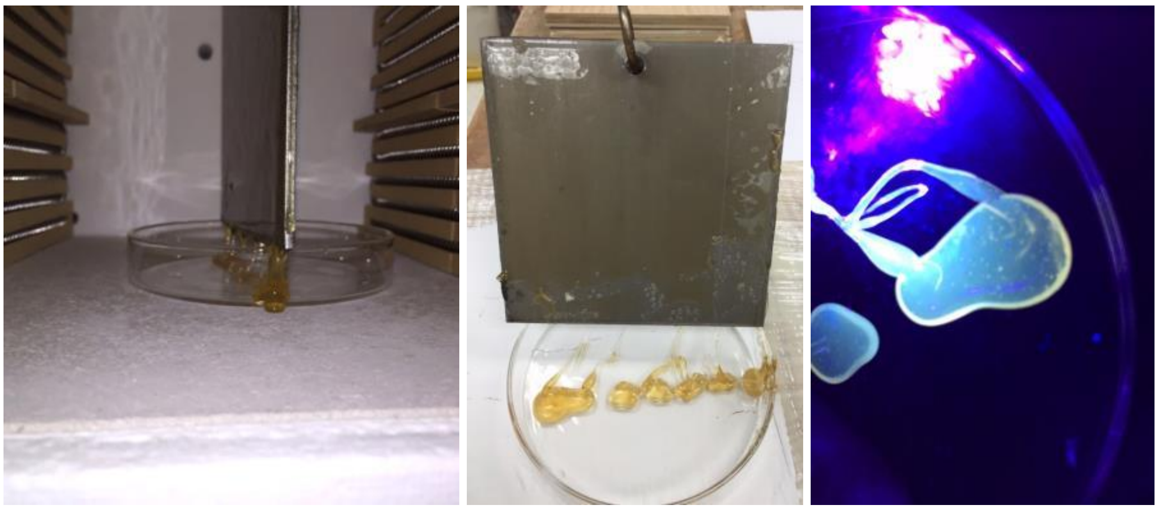


Figure 3-4. Sheet metal coupon placed in muffle furnace (left), fixative flowed from metal coupon into petri dish with exposure to heat at 250° F (middle); GloGerm transported from coupon to petri dish fluorescing under black light.

Figure 3-5 shows a coupon where GloGerm gel was applied to a red oak substrate and then layered with an industry fixative to the recommended thickness. Note there is initially no contamination in the petri dish. At approximately 200° F, as the fixative expanded, so did the GloGerm. At 250-300° F, the fixative began to melt and flow into the petri dish along with the simulated contaminant.

Figure 3-5. Red oak coupon and clean petri dish (left), fixative began to flow from coupon into petri dish with exposure to heat at 250° F (middle and right).

The next step for the Phase II testing will be adding a layer of an intumescent coating on top of the fixative to determine if it helps increase the resiliency of the fixatives when exposed to heat.

Initial attempts at layering the intumescent coating have presented challenges. Small fissures and cracks in the intumescent coating have occurred during the curing process. Enhancing the bonding/adhesion of the intumescent coating will be a topic for future research. During preliminary tests, the fixative began to flow through these cracks and fissures in the intumescent coating once exposed to temperatures between 400 and 500° F (Figure 3-6).

Figure 3-6. Fixative with GloGerm flowing through fissure in intumescent coating when exposed to heat at 400-500° F (left); image of petri dish showing fluorescing GloGerm.

Additionally, ARC completed preparation of 12” x 12” coupons on steel, red oak, and sheetrock with the same set of fixatives and shipped the samples to SRNL to support their complimentary research, specifically enhancing the radiation resiliency of fixatives and their application under varying environmental conditions.

Task 2.1.2: Development of a Decision Model for Contamination Control Products

During this reporting period, FIU continued to review and update the contamination control product list. This list is being used to develop the web and mobile decision model on the D&D KM-IT framework, described under Task 3, and scheduled to be deployed in mid-January 2016. Once the web-based application is complete, it will be made available through the D&D Knowledge Management Information Tool portal for beta testing and input from field site users.

Task 2.1.3: Robotic Technologies for SRS 235-F

This is a new task for FIU Year 6. The SRS 235-F facility has a need to identify a remote system that can make one-time entry to highly contaminated areas. The one-time-entry requirement indicates that the technology will not be retrieved at the end of the work but would remain inside the facility due to the high levels of contamination. FIU will perform research to identify robotic technology systems applicable to the challenges and needs of the SRS 235-F Facility. Research will include working with SRNL to define the requirements for the robotic technology and utilizing the Robotic Database in D&D KM-IT to search and identify potential technologies that meet the defined requirements. A summary report will be developed to document the results.

Task 2.1.4: Fogging Research and Evaluation

Details of the summer internships as well as research results are detailed in the summer internship technical reports that Jesse and Janesler developed during September. These reports will be posted to the DOE Fellows website (fellows.fiu.edu) in October after review and site approval.

Task 2.2: Technology Demonstration and Evaluation

The primary objective of this new task is to standardize and implement proven processes to refine and better synchronize DOE-EM technology needs, requirements, testing, evaluation, and acquisition by implementing a three-phased technology test and evaluation model. The development of uniformly accepted testing protocols and performance metrics is an essential component for testing and evaluating D&D technologies.

During this reporting period, FIU coordinated agenda items and the committee schedule with ASTM International's E10 Committee on Nuclear Technologies and Applications and E10.03 - Radiological Protection for Decontamination and Decommissioning of Nuclear Facilities and Components for the January 2016 conference in San Antonio, TX. The chairs for both committees requested preparation of an executive brief to support the initiative of developing and promulgating uniform testing protocols and performance metrics for D&D technologies across the stakeholder community. The contents of the brief will also outline the proposed way ahead for bringing this initiative to fruition. FIU began preparation of this executive brief.

FIU also developed a manuscript titled, "The Expanding Nuclear Niche and Growing Requirement for Standardized Testing Protocols and Performance Metrics for D&D Technologies," which was approved for publication in ASTM International's Standardization News magazine by the editor-in-chief. The article is expected to be published in the March/April 2016 edition.

Task 3: D&D Knowledge Management Information Tool (KM-IT)

Task 3 Overview

The D&D Knowledge Management Information Tool (KM-IT) is a web-based system developed to maintain and preserve the D&D knowledge base. The system was developed by Florida International University's Applied Research Center (FIU-ARC) with the support of the D&D community, including DOE-EM (EM-13 & EM-72), the former ALARA centers at Hanford and Savannah River, and with the active collaboration and support of the DOE's Energy Facility Contractors Group (EFCOG). The D&D KM-IT is a D&D community driven system tailored to serve the technical issues faced by the D&D workforce across the DOE Complex. D&D KM-IT can be accessed from web address http://www.dndkm.org.

Task 3 Quarterly Progress – October 1 – December 31, 2015

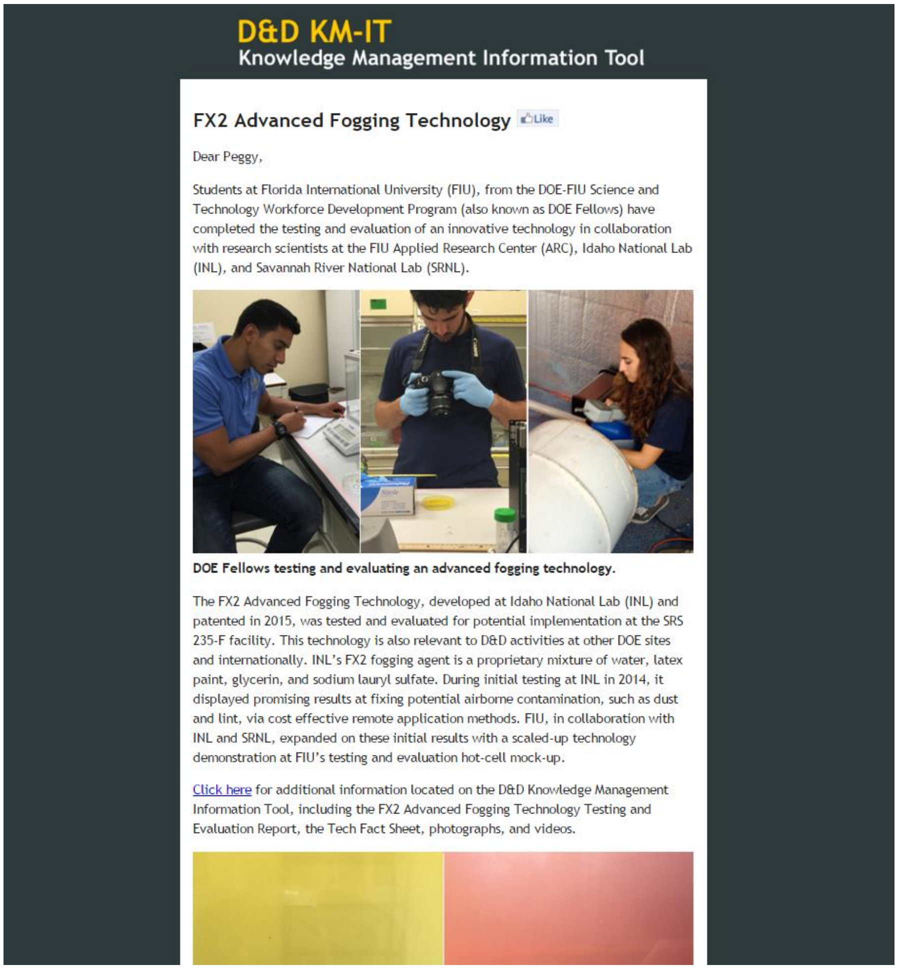
A paper on the D&D Knowledge Management Information Tool was drafted and submitted for the Waste Management Symposia to be held on March 6-10, 2016 in Phoenix, AZ. The paper was accepted for presentation at the conference.

Abstract: 16465 - Robotics Technologies on Knowledge Management Information Tool (KM-IT) Platform
Session: 087C - Posters: Robotic Technologies
Date: Wednesday, March 09, 2016
Time: 8:30 AM - 12:00 PM

DOE Fellows and other FIU students are supporting D&D KM-IT by reviewing the information in the vendor and technology modules and updating contact information. As of January 4, the system included a total of 1259 technologies and 924 vendors.

The FIU team continued working on developing the desktop and mobile decision support model for fixatives. Once the development of the fixatives database was completed, FIU started testing the class libraries that will fetch information from the fixative database for the web page user interface. The latest Bootstrap framework is being used to develop the front-end tier of the application for the home screen and summary search. The data tables and relationships are being established for all the entities (criteria, sub-criteria, and products) and sample data is being imported for development using the SQL server data integration services. The design of the web/mobile design with the Bootstrap framework is being developed to fit to the D&D KM-IT theme. The code in the framework is being developed to have the drop-down menus reflect the user's selections. Once this is completed, the backend work, such as linking the database to the design of the website, will be performed.

FIU finalized a newsletter for D&D KM-IT, highlighting the testing and evaluation of the FX2 advanced fogging agent developed by INL. The newsletter test was sent to DOE for review on October 28. After the DOE review and subsequent revisions were completed, the newsletter was distributed to the D&D KM-IT registered users on November 23, 2015. Figure 3-7 shows an image of the newsletter.



D&D KM-IT
Knowledge Management Information Tool

FX2 Advanced Fogging Technology Like

Dear Peggy,

Students at Florida International University (FIU), from the DOE-FIU Science and Technology Workforce Development Program (also known as DOE Fellows) have completed the testing and evaluation of an innovative technology in collaboration with research scientists at the FIU Applied Research Center (ARC), Idaho National Lab (INL), and Savannah River National Lab (SRNL).

DOE Fellows testing and evaluating an advanced fogging technology.

The FX2 Advanced Fogging Technology, developed at Idaho National Lab (INL) and patented in 2015, was tested and evaluated for potential implementation at the SRS 235-F facility. This technology is also relevant to D&D activities at other DOE sites and internationally. INL's FX2 fogging agent is a proprietary mixture of water, latex paint, glycerin, and sodium lauryl sulfate. During initial testing at INL in 2014, it displayed promising results at fixing potential airborne contamination, such as dust and lint, via cost effective remote application methods. FIU, in collaboration with INL and SRNL, expanded on these initial results with a scaled-up technology demonstration at FIU's testing and evaluation hot-cell mock-up.

Click here for additional information located on the D&D Knowledge Management Information Tool, including the FX2 Advanced Fogging Technology Testing and Evaluation Report, the Tech Fact Sheet, photographs, and videos.

Figure 3-7. Newsletter on advanced fogging technology.

FIU completed the development of a Google Web Analytic report for D&D KM-IT for the third quarter of 2015 (July to September) and submitted it to DOE on December 3, 2015. This report included information from Google Analytics and Google Web Master tools and a narrative to explain the results. The following figure provides an infographic of the web analytic data.

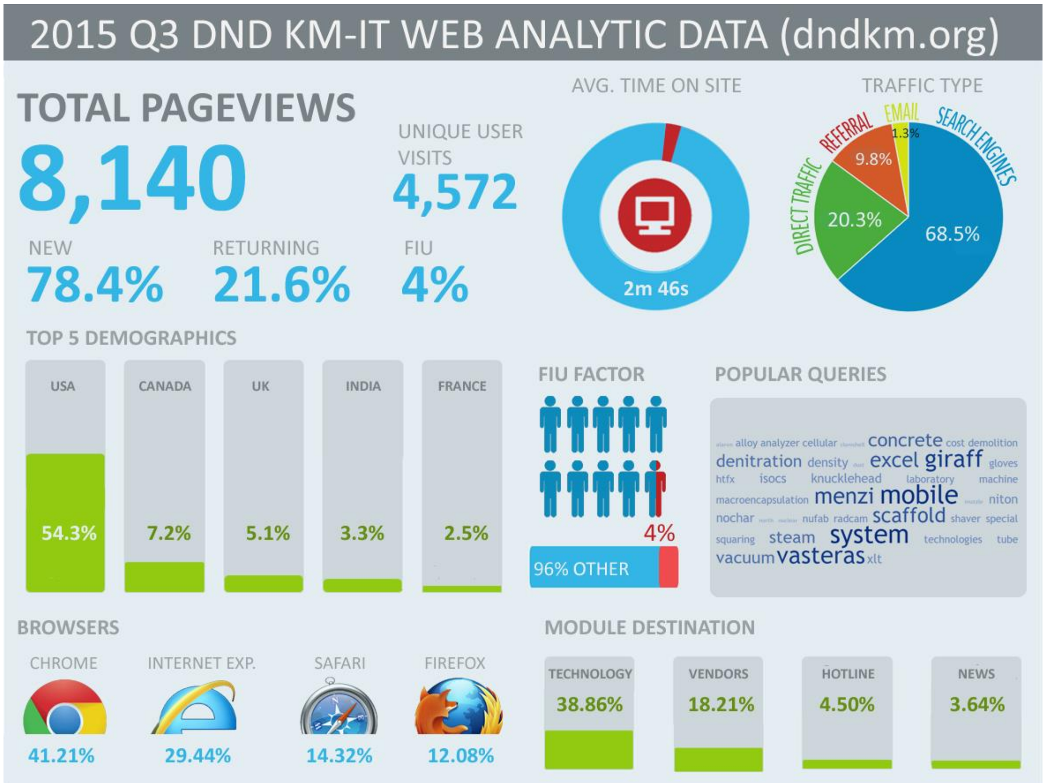


Figure 3-8. Infographic for 2015 Q3 based on web analytic data.

The following figure shows the demographics of D&D KM-IT registered users and subject matter specialists (SMS) across the United States.

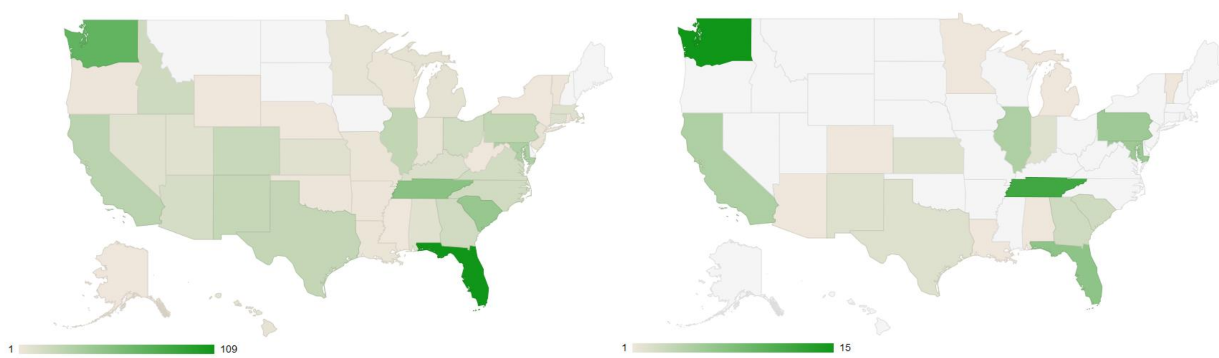


Figure 3-9. Demographics of D&D KM-IT registered users (left) and SMS (right).

A few of the highlights from this report include:

- This report reflects a slight improvement in combined visits of 13.7% over 2015 Q2 and last year's Q3. There were a total of 4,572 sessions during this quarter by 1,840 users.
- Innovative Technology Summary Reports continue to dominate the popular documents on D&D KM-IT with nine (9) out of the top ten (10) documents being ITSRs.
- The number of visits from Canada almost doubled during this quarter to 7.2% of all visits.
- Registered users increased by 97 while SMS increased by 7 over last quarter. Most of these new registrations are credited to the 2015 ANS Utility Working Conference and

Vendor Technology Expo that took place at Amelia Island, FL during the month of August.

Milestones and Deliverables

The milestones and deliverables for Project 3 for FIU Year 6 are shown on the following table. Draft papers based on project research were submitted to Waste Management 2016 (milestones 2015-P3-M1.1 and 2015-P3-M3.1). The first D&D KM-IT Workshop to DOE EM staff at HQ is being reforecast based on the schedule and availability of DOE EM staff. Milestone 2015-P3-M2.1 was met with the completion of the Phase 1 testing of fixatives for the incombustible fixatives task.

FIU Year 6 Milestones and Deliverables for Project 3

Task	Milestone/ Deliverable	Description	Due Date	Status	OSTI
Task 1: WIMS	2015-P3-M1.1	Import 2016 data set for waste forecast and transportation data	Within 60 days after receipt of data	On Target	
	2015-P3-M1.2	WM 2016 Paper for WIMS	11/6/2015	Complete	
Task 2: D&D	2015-P3-M2.1	Completion of Phase 1 testing of incombustible fixatives	12/31/2015	Complete	
	2015-P3-M2.2	Participate in ASTM E10 Committee Meeting to introduce a requirement for standardized D&D testing protocols & performance metrics	01/31/2016	On Target	
	Deliverable	Summary Report on Robotic Technologies for SRS 235-F Facility	05/29/2016	On Target	OSTI
	Deliverable	Draft Test Plan for Phase II incombustible fixatives testing and evaluation	06/30/2016	On Target	OSTI
	2015-P3-M2.3	Participate in ASTM International's Executive Steering Committee Meeting to solicit final approval for development of standardized testing protocols and performance metrics for D&D technologies.	06/30/2016	On Target	
	Deliverable	Decision brief to DOE-EM 13 on recommended technologies to test for FY'17 using FIU's 3-Phased Technology Test and Evaluation Model.	07/29/2016	On Target	
	Deliverable	Draft technical reports for demonstrated technologies	30-days after demo	On Target	OSTI
	Deliverable	Draft Tech Fact Sheet for technology evaluations/ demonstrations	30-days after demo	On Target	
Task 3: D&D KM-IT	2015-P3-M3.1	Waste Management Symposium Paper for D&D KM-IT	11/06/2015	Complete	
	Deliverable	First D&D KM-IT Workshop to DOE EM staff at HQ	11/30/2015** TBD based on availability of DOE EM staff	Reforecast	
	2015-P3-M3.2	Deployment of pilot web-based D&D Decision Model application	01/16/2016	On Target	
	2015-P3-M3.3	Completion of development & integration of	03/04/2016	On Target	

		International KM-IT pilot for UK collaboration			
	Deliverable	Preliminary Metrics Progress Report on Outreach and Training Activities	02/29/2016	On Target	
	Deliverable	First D&D KM-IT Workshop to D&D community	03/31/2016	On Target	
	2015-P3-M3.4	Four Wikipedia integration edits/articles	03/31/2016	On Target	
	2015-P3-M3.5	Deployment of pilot mobile application for D&D Decision Model	05/20/2016	On Target	
	Deliverable	Second D&D KM-IT Workshop to DOE EM staff at HQ	05/31/2016**	On Target	
	Deliverable	First infographic to DOE for review	07/25/2016	On Target	
	Deliverable	Second infographic to DOE for review	08/08/2016	On Target	
	2015-P3-M3.6	Completion of Global KM-IT Integration	08/14/2016	On Target	
	Deliverable	Metrics Progress Report on Outreach and Training Activities	08/15/2016	On Target	
	Deliverable	Second D&D KM-IT Workshop to D&D community	08/25/2016	On Target	
	Deliverable	Draft Security Audit Report	30-days after audit	On Target	
	Deliverable	D&D KM-IT Web Analysis Report	Quarterly	On Target	
	Deliverable	Draft Tech Fact Sheet for new modules or capabilities of D&D KM-IT	30-days after deployment of new module	On Target	

***Completion of this deliverable depends on scheduling and availability of DOE EM staff*

Work Plan for Next Quarter

- Task 1: Perform database management, application maintenance, and performance tuning to WIMS.
- Task 1: Present technical paper on WIMS to WM16.
- Task 1: Receive 2016 data set for waste forecast and transportation data from DOE and begin integration into WIMS.
- Task 2: Present technical paper on the incombustible fixatives research to WM16.
- Task 2: Complete execution of the phase II testing for evaluating a set of contamination control products and intumescent coatings, selected by FIU and SRS.
- Task 2: Participate in ASTM International's E10 Committee on Nuclear Technologies and Applications and E10.03 - Radiological Protection for Decontamination and Decommissioning of Nuclear Facilities and Components at the January 2016 conference in San Antonio, TX. Complete and present the executive brief to support the initiative of developing and promulgating uniform testing protocols and performance metrics for D&D technologies across the stakeholder community.
- Task 2: Finalize the manuscript titled, "The Expanding Nuclear Niche and Growing Requirement for Standardized Testing Protocols and Performance Metrics for D&D

Technologies," for publication in ASTM International's Standardization News magazine by the editor-in-chief (March/April 2016 edition).

- Task 2: Collaborate with SRNL to define the requirements for a remote system to support D&D activities at the SRS 235-F facility as well as across the DOE complex.
- Task 3: Present technical paper on D&D KM-IT to WM16.
- Task 3: Complete development and deployment for DOE review for the pilot web-based D&D Decision Model application.
- Task 3: Develop website analytics report for the fourth quarter (Oct to Dec) of 2015 and submit to DOE for review.
- Task 3: Develop website analytics report for calendar year 2015 and submit to DOE for review.
- Task 3: Complete first D&D KM-IT Workshop to DOE EM staff at HQ, based on scheduling and availability of DOE EM staff.
- Task 3: Complete development and integration of the international KM-IT pilot for UK collaboration for review by DOE.
- Task 3: Complete four new Wikipedia integration edits/articles in support of D&D topics.
- Task 3: Perform outreach and training, community support, data mining and content management, and administration and support for the D&D KM-IT system, database, and network.

Project 4
DOE-FIU Science & Technology Workforce Development Initiative

Project Manager: Dr. Leonel E. Lagos

Project Description

The DOE-FIU Science and Technology Workforce Development Initiative has been designed to build upon the existing DOE/FIU relationship by creating a "pipeline" of minority engineers specifically trained and mentored to enter the Department of Energy workforce in technical areas of need. This innovative program was designed to help address DOE's future workforce needs by partnering with academic, government and DOE contractor organizations to mentor future minority scientists and engineers in the research, development, and deployment of new technologies, addressing DOE's environmental cleanup challenges.

Project Overview

The main objective of the program is to provide interested students with a unique opportunity to integrate course work, Department of Energy (DOE) field work, and applied research work at ARC into a well-structured academic program. Students completing this research program would complete the M.S. or Ph.D. degree and immediately be available for transitioning into the DOE EM's workforce via federal programs such as the Pathways Program or by getting directly hired by DOE contractors, other federal agencies, and/or STEM private industry.

Project Quarterly Progress

Fellows continue their support to the DOE-FIU Cooperative Agreement by actively engaging in EM applied research and supporting ARC staff in the development and completion of the various tasks. The program director continues to work with DOE sites and HQ to fully engage DOE Fellows with research outside ARC where Fellows provide direct support to mentors at DOE sites, DOE-HQ, and DOE contractors. All Fellows also participated in a weekly meeting conducted by the program director, a conference line has been established to enable DOE Fellows conducting internship to join to weekly meeting and update program director on their internship. During each of these meetings, one DOE Fellow presents the work they performed during their summer internship and/or EM research work they are performing at ARC.

The DOE Fellows Fall 2015 application process was completed. A total of 26 applications were received. FIU students' applications were reviewed, and selected candidates were interviewed by the DOE Fellows selection committee during the month of October. Six (6) new DOE Fellows were selected. The following lists the 12 FIU STEM students selected for the Class of 2015 (six from the Spring 2015 recruitment and six from the Fall 2015 recruitment).

Table 4-1. New DOE Fellows from Spring and Fall 2015 Recruitment

New DOE Fellows from Spring 2015 Recruitment	New DOE Fellows from Fall 2015 Recruitment
Silvina Di Pietro *graduate (Ph.D.), chemistry*	Sarah Bird *undergraduate, environmental engineering*
Erim Gokce *undergraduate, mechanical engineering*	Alejandro Garcia *graduate (M.S.), geoscience*
Orlando Gomez *graduate (Ph.D.), physics*	Iti Mehta *undergraduate, mechanical engineering*
Alejandro Hernandez *undergraduate, chemistry*	Alexis Smoot *undergraduate, environmental engineering*
Awmna Rana *undergraduate, chemistry & biological sciences*	Gene Yllanes *undergraduate, electrical engineering*
Christopher Strand *undergraduate, civil & environmental engineering*	Sebastian Zanlongo *graduate (Ph.D.), computer science*

One (1) DOE Fellow, Awmna Kalsoom Rana, presented her research poster at the FIU McNair Scholars Research Conference held at the main FIU campus on October 14-16, 2015 (Figure 4-1). The title of the poster was “Geospatial Analysis of Time Series Data Used for Hydrological Modeling of the Fate and Transport of Contaminants in Tims Branch Watershed.”

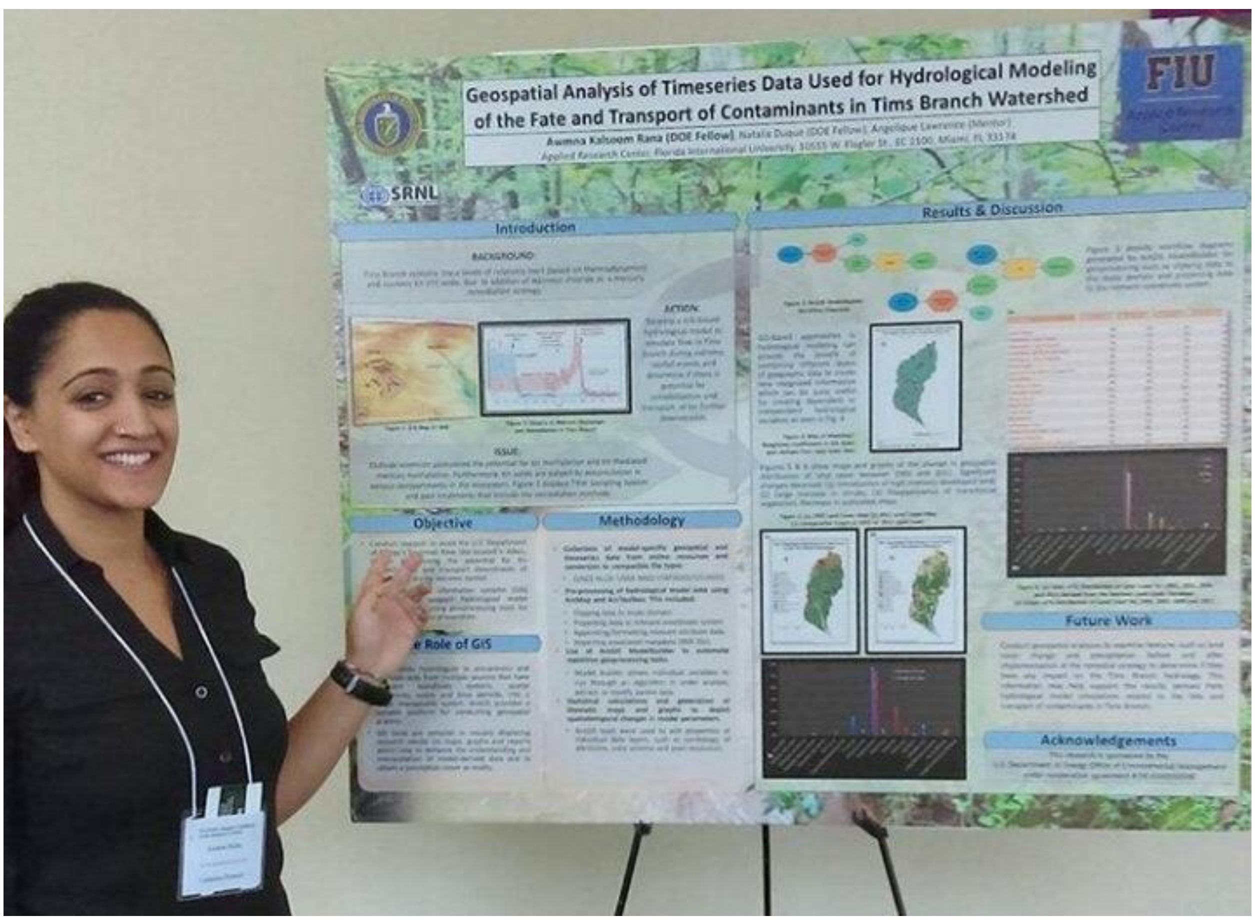


Figure 4-1. DOE Fellow Awmna Rana presenting her research at the FIU McNair Scholars Research Conference.

The DOE Fellows Program Lecture Series at the Applied Research Center had the honor of hosting a presentation by Dr. Donald Reed on October 20, 2015, during his visit to Florida International University (FIU). Dr. Donald Reed is the current team leader for the Actinide Chemistry and Repository Science Program at Los Alamos National Laboratory. The title of his talk was “Actinide Solubility and Speciation in the WIPP Transuranic Repository.” Dr. Reed also participated in a discussion with ARC staff on the research conducted by ARC for the Department of Energy (DOE) sites. Dr. Reed and ARC staff both expressed interest in the collaboration to advance the science of WIPP research, which is a high priority mission for DOE Office of Environmental Management.

DOE Fellows also participated in the DOE Fellows lecture series featuring Mr. Karthik Subramanian (Chief Technology Officer) from Washington River Protection Solutions (WRPS). Mr. Subramanian talked about “The Role of Technology in Hanford Tank Waste Disposition” and also participated in discussions with ARC researchers.

FIU conducted the annual DOE Fellows Poster Exhibition and Competition on October 21, 2015 (Figures 4-2 and 4-3). The purpose of this event was to showcase the DOE Fellows' research accomplishments for the past year as a result of their participation in various U.S. Department of Energy - Environmental Management (DOE-EM) related applied research projects. A total of 18 posters were exhibited. Some of the projects showcased by the students were a result of their summer internship assignments at DOE sites, national laboratories, and DOE Headquarters (DOE-HQ) in Washington, DC. Additional posters reflected the DOE Fellows’ DOE-EM applied research that they conduct at ARC as part of the DOE-FIU Cooperative Agreement sponsored research. For some of the graduate students, these projects are also a part of their thesis towards a master’s or Ph.D. degree. This year’s panel of judges included Dr. Don Reed (Team Leader for Actinide Chemistry and Repository Science Program at LANL), Dr. Michael Sukop (Professor in the Department of Earth and Environment at FIU), Dr. Anthony McGoron (Associate Dean & Professor in the Department of Biomedical Engineering at FIU), and Dr. Michael Robinson (Instructor in the Department of Computing and Information Sciences at FIU). The poster exhibition and competition was conducted at FIU’s Engineering Center’s Panther Pit and was attended by FIU faculty, ARC personnel, and FIU students.

The posters presented included:

- **Implementation for 64-Bit Instruction Algorithm for Hyperion**
  DOE Fellow Andrew De La Rosa
- **Nonmetallic Materials Testing for Hanford’s HLW Transfer System**
  DOE Fellow Anthony Fernandez
- **Development of a Micromachined Ultrasonic Transducer System for NDT Analysis of High Level Waste Pipes at Hanford**
  DOE Fellow Brian Castillo
- **Sodium Silicate Treatment for Uranium (VI) Bearing Groundwater at F/H Area at Savannah River Site**
  DOE Fellow Christine Wipfli
- **Incombustible Fixatives**
  DOE Fellow Janesler Gonzalez

- **Mock-Up Scrubber System**
  DOE Fellow Jesse Viera
- **Stainless Steel Corrosion: Feed Properties Affecting Material Selection for LAWPS Piping at the Hanford Site**
  DOE Fellow John Conley
- **Radial Jet Impingement Correlation Investigation of the Pulse Jet Mixers**
  DOE Fellow Maximiliano Edrei
- **Analysis of Solar Generated Power in the Southeastern United States**
  DOE Fellow Natalia Duque
- **Miniature Motorized Inspection Tool for DOE Hanford Site Tank Bottoms**
  DOE Fellow Ryan Sheffield
- **3D Visualization**
  DOE Fellow Jorge Deshon
- **Optimizing Remediation of I-129 using AgCl Colloidal-Sized Particles in SRS F-Area Sediments**
  DOE Fellow Aref Shehadeh
- **Geospatial Analysis of Timeseries Data Used for Hydrological Modeling of the Fate and Transport of Contaminants in Tims Branch Watershed**
  DOE Fellow Awmna Kalsoom Rana
- **Study of an Unrefined Humate Solution as a Possible Remediation Method for Groundwater Contamination**
  DOE Fellow Hansell Gonzalez Raymat
- **Processing of Diffusion Samplers to Test Remediation of Uranium by Humate**
  DOE Fellow Kiara Pazan
- **Heat Transfer Calculations for the Use of an Infrared Temperature Sensor**
  DOE Fellow Meilyn Planas
- **Characterization of the Uranium-Bearing Products of the Ammonia Injection Remediation Method**
  DOE Fellow Robert Lapierre
- **Climate Change Vulnerability Assessment and Adaptation Plan for the DOE Sites**
  DOE Fellow Yoel Rotterman

Figure 4-2. DOE Fellows with Student Poster Competition Judging Panel.

Figure 4-3. DOE Fellows presenting research at Student Poster Competition.

On November 5, 2015, Florida International University's (FIU's) Applied Research Center (ARC) conducted the ninth (9th) annual DOE Fellows Induction Ceremony. This year, twelve (12) FIU STEM students were inducted as DOE Fellows. Dr. Monica Regalbuto (Assistant Secretary for Environmental Management, DOE EM) was one of the keynote speakers for the ceremony. Other distinguished guests included Ms. Rosa Elmetti (Technical Advisor for International Programs, DOE EM), Mr. John De Gregory (Technical Program Monitor, Office of D&D and Facility Engineering, DOE EM), Dr. Jeff Griffin (Associate Laboratory Director, Environmental Stewardship, SRNL), Mr. Jose Sanchez (Director, Coastal and Hydraulics Laboratory, US Army Engineering Research and Development Center (ERDC), US Army Corps of Engineers), Dr. Carlos Ruiz (Senior Research Scientist, ERDC, Army Corps of Engineers), Dr. Kevin Cooper (Dean of Applied Research & Entrepreneurial Activities, Indian River State College), Mr. Jim Voss (Managing Director, Waste Management Symposia, Inc.), Mr. Jorge Rosenblut (President, Strategies & Business Development, Corp.), Mr. Vijay Alreja (VJT Founder and President, VJ Technologies, Inc.), Ms. Rosey Villagomez (Marketing Coordinator, IHI Southwest Technologies, Inc. and NitroCision, LLC), and Mr. Christopher Wright (Director of Operations, Cabrera Services, Inc.). FIU was represented at the event by Dr. Kenneth Furton (Provost, Executive Vice President, FIU), Dr. Andrés Gil (Vice President for Research), Dr. Ranu Jung (Interim Dean, College of Engineering and Computing), Dr. Inés Triay (ARC Executive Director) and Dr. Leonel E. Lagos (Principal Investigator for DOE-FIU Cooperative Agreement and Director, DOE Fellows Program), as well as FIU faculty, staff, and students.

During the ceremony, a Memorandum of Understanding (MOU) was signed by Dr. Monica Regalbuto and Dr. Kenneth Furton (Figure 4-4). This MOU between DOE and FIU commemorates a renewal of a strong partnership between the two organizations that started in 1995. The renewal is part of the five (5) year, $20M DOE-FIU Cooperative Agreement between DOE and FIU. The new five year cycle officially started on August 29, 2015 and has a period of performance ending on August 28, 2020. Under this new agreement, FIU will continue conducting scientific research in support of DOE EM environmental restoration mission and the STEM workforce development program (DOE Fellows Program) that provides research and employment opportunities to FIU STEM minority students.

A Memorandum of Agreement (MOA) was also signed by Dr. Jeff Griffin and Dr. Andres Gil, to commemorate the strong research collaboration relationship between the Applied Research Center at FIU and the Savannah River National Laboratory in the areas of deactivation and decommissioning (D&D), environmental remediation (ER), high level waste treatment and disposal, radiochemistry, nuclear forensics and cyber security.

Ms. Regalbuto and the other distinguished guests had the opportunity to participate in morning tours of the ARC research laboratories and listen to DOE Fellows presenting their research work. Presentations were given by Dr. Lagos and DOE Fellows Ryan Sheffield and Hansell Gonzalez. Dr. Lagos presented an overview of the DOE Fellows program. DOE Fellow Ryan Sheffield presented his DOE EM research on developing a miniature motorized inspection tool for DOE Hanford Site tank bottoms. DOE Fellow Hansell Gonzalez presented his DOE EM research on unrefined humate solutions as a possible remediation method for groundwater contamination. Tours of the ARC facilities included visits to the ARC test and evaluation facility for a demonstration on the incombustible fixatives research; the radiological laboratory; the modeling, simulation & GIS laboratory; the soil and groundwater laboratory; the IT and cyber research laboratory; and the robotics and sensors laboratory for a demonstration of the inspection tools being developed for double-shell tanks at the Hanford Site (Figure 4-5). In addition, eighteen (18) DOE Fellows had the opportunity to showcase their research by presenting posters as part of the afternoon events.

Figure 4-4. Memorandum of Understanding (MOU) signing by Dr. Monica Regalbuto and Dr. Kenneth Furton, Dr. Ines Triay and Dr. Leonel Lagos.

Figure 4-5. Ms. Regalbuto and the other distinguished guests watching the demonstration of the inspection tools being developed for double-shell tanks at the Hanford Site.

Figure 4-6. New DOE Fellows at the 2015 Induction Ceremony.

During this year's induction ceremony, 12 new FIU STEM students were inducted as DOE Fellows:

- Sarah Bird – undergraduate, environmental engineering
- Silvina Di Pietro – graduate (Ph.D.), chemistry
- Alejandro Garcia, graduate (M.S.), geoscience
- Erim Gokce – undergraduate, mechanical engineering
- Orlando Gomez, graduate (Ph.D.), physics
- Alejandro Hernandez, undergraduate, chemistry
- Iti Mehta – undergraduate, mechanical engineering
- Awmna Rana, undergraduate, chemistry & biological sciences
- Alexis Smoot – undergraduate, environmental engineering
- Christopher Strand – undergraduate, civil & environmental engineering
- Gene Yllanes – undergraduate, electrical engineering
- Sebastian Zanlongo – graduate (Ph.D.), computer science

In addition, awards were presented to the DOE Fellows that won the DOE Fellows Poster Exhibition and Competition held on October 21, 2015. First place was awarded to Ms. Christine Wipfli for her poster titled, "Sodium Silicate Treatment for Uranium (VI) Bearing Groundwater at F/H Area at Savannah River Site." Second place went to Mr. Hansell Gonzalez Raymat for his poster titled, "Study of an Unrefined Humate Solution as a Possible Remediation Method for Groundwater Contamination." Third place was awarded to Mr. Anthony Fernandez for his poster titled "Nonmetallic Materials Testing for Hanford's HLW Transfer System."

For the seventh year, the DOE Fellow of the Year Award and the Mentor of the Year Award were presented at the ceremony. DOE Fellows were requested to nominate their ARC mentors and ARC mentors were requested to nominate the DOE Fellows. An ARC committee was established to review and select the winners from the submitted nominations. The 2015 Mentor of the Year Award went to Postdoctoral Research Fellow Dr. Vasileios Anagnostopoulos. The 2015 DOE Fellow of the Year Award was awarded to Mr. Hansell Gonzalez Raymat (DOE Fellows Class of 2013). A new award for the 2015 Emerging DOE Fellow of the Year was awarded to Mr. Jesse Viera (DOE Fellows Class of 2014) and Mr. Jorge Deshon (DOE Fellows Class of 2014).

The six (6) new DOE Fellows hired during the fall recruitment were assigned to an ARC staff member to act as their mentor and supervise their research work. The following table lists the new DOE Fellows as well as their ARC mentor and DOE related project.

Table 4-2. New DOE Fellows from Spring/Summer Recruitment

Name	Classification	Major	ARC Mentor	Project Support
Alejandro Garcia	Graduate - M.S.	Geoscience	Dr. Yelena Katsenovich	FIU's Support for Groundwater Remediation at PNNL
Alexis Smoot	Undergrad - B.S.	Environmental Eng.	Dr. Ravi Gudavalli	Synergistic Effects of Silica and Humic Acid on U(VI) Removal
Gene Yllanes	Undergrad - B.S.	Electrical Eng.	Dr. David Roelant	Evaluation of FIU's SLIM for Estimating the Onset of Deep Sludge Gas Release Events
Iti Mehta	Undergrad - B.S.	Mechanical Eng.	Dr. Aparna Aravalli	Investigation Using an Infrared Temperature Sensor to Determine the Inside Wall Temperature of DSTs
Sarah Bird	Undergrad - B.S.	Environmental Eng.	Dr. Ravi Gudavalli	Modeling of the Migration and Distribution of Natural Organic Matter injected into Subsurface Systems
Sebastian Zanlongo	Graduate - Ph.D.	Computer Science	Dr. Dwayne McDaniel	TBD

The new DOE Fellows also completed the required environmental health and safety (EH&S) training prior to engaging in laboratory work, including:

- Laboratory Hazard Awareness Training
- HazCom: In Sync with GHS
- Fire Safety
- Environmental Awareness Part II
- Small Spills and Leaks
- EPA Hazardous Waste Awareness and Handling
- Personal Protective Equipment
- Safe Use of Fume hoods
- Radiation Safety

Dr. Lagos conducted orientation sessions for the new class of DOE Fellows and discussed the expectations of the program, including program components such as hands-on research on DOE related challenges, summer internships, and potential future employment with DOE EM, national laboratories and DOE contractors.

Two of our current DOE Fellows graduated with a bachelor's degree in environmental engineering and participated in the FIU graduation ceremony held on December 14, 2015.

- **Kiara Pazan** (Environmental Engineering) – DOE Fellow Class of 2014
- **Aref Shehadeh** (Environmental Engineering) – DOE Fellow Class of 2014

In addition, Andrew De La Rosa left the DOE Fellows program to accept employment with Lockheed Martin.

Figure 4-7. DOE Fellows graduating from FIU: Kiara Pazan and Aref Shehadeh.

The American Nuclear Society (ANS) has approved the establishment of an ANS student section at Florida International University (FIU). The FIU ANS student section will be officially launched in January 2016 with a visit to FIU from ANS president Eugene "Gene" Grecheck for a special ceremony to present the Student Section Charter. This event is currently scheduled for January 28, 2016. Chapter officers include Ryan Sheffield (President), Maximiliano Edrei (Vice President), Awmna Rana (Secretary), Janesler Gonzalez (Committee Head), and Jesse Viera (Treasurer). Dr. Leo Lagos from FIU's Applied Research Center is serving as the FIU Chapter Faculty Advisor.

Figure 4-8. ANS Student Chapter at FIU. From left to right: Awmna Rana (Communication Officer), Carolina Padron (Secretary), Ryan Sheffield (President), Maximiliano Edrei (Vice President), Janesler Gonzalez (Officer - special programs), Jesse Viera (Treasurer) and Dr. Leo Lagos (FIU Chapter advisor).

The DOE Fellows who participated in a summer 2015 internship are preparing and presenting an oral presentation at the weekly DOE Fellows meetings. The schedule for these presentations is provided below.

Table 4-3. Presentations on Summer 2015 Internships

DOE Fellow	***Internship Location***	***Summer Mentor(s)***	***Date***
John Conley	*WRPS, Richland, WA*	*Terry Sams/ Dave Shuford*	*Sept 11, 2015*
Andrew De La Rosa	*Oak Ridge National Lab – Cyber & Information Security Research*	*Joseph Trien*	*Sept 18, 2015*
Kiara Pazan & Aref Shehadeh	*SRNL, Savannah River, SC*	*Miles Denham/ Margaret Millings*	*Oct 09, 2015*
Christine Wipfli	*DOE-HQ EM-12, Cloverleaf, Germantown, Maryland*	*Skip Chamberlain/ Kurt Gerdes*	*Nov 20, 2015*
Maximiliano Edrei	*National Energy Technology Lab, Morgantown, WV*	*Chris Guenther*	*Nov 20, 2015*
Natalia Duque	*SRNL, Savannah River, SC*	*Ralph Nichols/ Carol Eddy-Dilek/ Brian Looney*	*Dec 10, 2015*
Yoel Rotterman	*DOE-HQ EM-13, Forrestal, Washington D.C.*	*Albes Ganoa/ John De Gregory*	*TDB*
Ryan Sheffield	*DOE-HQ EM-20, Cloverleaf, Germantown, Maryland*	*James Poppiti*	*TDB*
Jorge Deshon	*SRNL, Savannah River, SC*	*John Bobbitt/ Steven Tibrea*	*TDB*
Janesler Gonzalez & Jesse Viera	*Idaho National Lab*	*Rick Demmer/ Steve Reese*	*TDB*
Anthony Fernandez	*Washington River Protection Solutions (WRPS), Richland, WA*	*Ruben Mendoza/ Gregory Gauck*	*TDB*
Meilyn Planas	*WRPS, Richland, WA*	*Terry Sams*	*TDB*
Claudia Cardona	*PNNL, Richland, WA*	*Jim Sczcsody*	*TDB*

A paper was drafted and submitted to the Waste Management Symposia to be held on March 6-10, 2016 in Phoenix, AZ. The paper was accepted for presenting at the conference.

Abstract: 16513 - Development of a Workforce for the Nuclear Industry
Session: 028 - Workplace Management and Performance Solutions
Date: Monday, March 07, 2016
Time: 1:30 PM - 5:00 PM

In addition, DOE Fellows completed development of 100-word abstracts on their research to be presented at Waste Management 2016 during the student poster session. The DOE Fellows also began working on short videos briefly describing their research for submittal to the conference. Twenty (20) DOE Fellows will participate in the conference.

1. Kinetic and Mechanism Studies of U(VI) Bearing Groundwater Treated with Sodium Silicate at the Savannah River Site - Alejandro Hernandez (DOE Fellow)
2. Nonmetallic Materials Testing for Hanford's HLW Transfer System - Anthony Fernandez (DOE Fellow)
3. Application of Geospatial Tools to Support Development of a Hydrological Model of the Tims Branch Watershed, Aiken, SC - Awmna Rana (DOE Fellow)
4. Development of a Data Acquisition Unit for the Remote Permanent Mounting of Ultrasonic Transducers for the Integrity Analysis of Hanford Transfer Component - Brian Castillo (DOE Fellow)
5. A Study of Sodium Silicate Treatment for the U(VI)-impacted Acidic Groundwater at Savannah River Site's F/H Area - Christine M. Wipfli (DOE Fellow)
6. Topographic Analysis of Timeseries Data to Support the Hydrology Model of the Tims Branch Watershed, Aiken, SC - Christopher Strand (DOE Fellow)
7. Modifications and Enhancements to the Robotic Pipe Inspection Tool to be utilized for the DOE High Level Waste Project at the Hanford Site - Erim Gokce (DOE Fellow)
8. Rapid Imaging of Solids in High Level Waste Tanks at Hanford - Gene Yllanes (DOE Fellow)
9. Study of an Unrefined Humate Solution as a Possible Remediation Method for Groundwater Contamination at SRS - Hansell Gonzalez Raymat (DOE Fellow)
10. Innovative Process for Abatement of Mercury - Janesler Gonzalez (DOE Fellow)
11. The Expanding Nuclear Niche and Growing Requirement for Standardized Testing Protocols and Performance Metrics for D&D Technologies - Jesse Viera (DOE Fellow), Orlando Gomez (DOE Fellow)
12. Stainless Steel Corrosion: Feed Properties Affecting Material Selection for LAWPS Piping at Hanford Site - John Conley (DOE Fellow)
13. Fixatives Decision Model on KM-IT Platform - Jorge Deshon (DOE Fellow)
14. Radial Jet Impingement Correlation Investigation - Maximiliano Edrei (DOE Fellow)
15. Heat Transfer Calculations for the Use of an Infrared Temperature Sensor - Meilyn Planas (DOE Fellow)
16. A Model to Simulate Flow in Tims Branch, Savannah River Site, SC - Natalia Duque (DOE Fellow)
17. The Characterization of Uranium Phases Produced by the NH3 Injection Remediation Method under Hanford 200 Area Conditions - Robert Lapierre (DOE Fellow)

18. Development of a Miniature Motorized Inspection tool for the Hanford DOE Site Tank Bottoms - Ryan Sheffield (DOE Fellow)

19. Ammonia Gas Injection for Remediation of Uranium Contamination - Silvina Di Pietro (DOE Fellow)

20. Green & Sustainable Remediation Analysis of a Packed Tower Air Stripper Used to Remediate Groundwater Contaminated with CVOCs - Yoel Rotterman (DOE-Fellow)

FIU continued to aggressively identify federal entry-level career opportunities within DOE on USA Jobs and forward those vacancy announcements to the DOE Fellows, with a particular emphasis on federal positions within DOE EM, the national labs, or DOE tier-1 contractors. ARC mentors also completed identifying those DOE Fellows who are preparing to transition from academia to the workforce. Training sessions on resume preparation and the USA Jobs application process are being incorporated into monthly meetings for the DOE Fellows.

Program Director Dr. Leonel Lagos met with Melody Bell (Associate Deputy Assistant Secretary, Human Capital, DOE EM) and John De Gregory (Technical Monitor for the DOE-FIU Cooperative Agreement) on October 23 to discuss the transition of DOE Fellows from the program to employment with DOE EM, national laboratories, and DOE contractors. The direct involvement and commitment from DOE will include the creation of transition programs. DOE HR also commits to conducting two (2) job fair/employment workshops for the DOE Fellows each year. In addition, FIU will engage directly with the HR offices at DOE national laboratories and contractors as well as coordinate workshop hiring socials with DOE contractors, national laboratories, etc. at designated conferences (e.g., WM, ANS, etc.)

Three (3) DOE Fellows (Ms. Kiara Pazan, Mr. Aref Shehadeh, and Ms. Meilyn Planas) applied to DOE job announcements on USA Jobs (see below). ARC mentors did not identify any specific job announcements being released by DOE EM on USA Jobs for the months of October, November, or December.

A. Job Title: Recent Graduate Energy Industry Analyst
Department: Department of Energy
Agency: Federal Energy Regulatory Commission (FERC)
Job Announcement Number: FERC-DE-2016-0013

B. **Job Title:** Student Trainee (Electrical Engineering)
Department: Department of Energy
Agency: Federal Energy Regulatory Commission (FERC)
Job Announcement Number: FERC-DE-2016-0015

C. **Job Title:** Student Trainee (Energy Industry)
Department: Department of Energy
Agency: Federal Energy Regulatory Commission (FERC)
Job Announcement Number: FERC-DE-2016-0016

One DOE Fellow (Kiara Pazan) received a callback requesting an interview with the US Army Corps of Engineers based on one of her application submitted through USA Jobs, indicating that her application passed the keyword search and initial evaluation by the hiring manager. Her interview is scheduled for January 2016.

All of the Fellows continued their research in the four DOE EM applied research projects under the cooperative agreement and research topics identified as part of their summer internships at DOE sites, national labs, and/or DOE HQ. Each DOE Fellow is assigned to DOE EM research projects as well as ARC mentors. A list of the current Fellows, their classification, areas of study, ARC mentor, and assigned project task is provided below.

Table 4-4. Project Support by DOE Fellows

Name	Classification	Major	ARC Mentor	Project Support
Alejandro Garcia	Graduate - B.S.	Geoscience	Dr. Yelena Katsenovich	FIU's Support for Groundwater Remediation at PNNL
Alejandro Hernandez	Undergrad - B.S.	Chemistry	Dr. Vasileios Anagnostopoulos	Groundwater Remediation at SRS F/H -Area
Alexis Smoot	Undergrad - B.S.	Envr. Eng.	Dr. Ravi Gudavalli	Synergistic Effects of Silica and Humic Acid on U(VI) Removal
Andrew De La Rosa	Graduate – M.S.	Computer Eng.	Mr. Himanshu Upadhyay	Information Technology for Environmental Management
Anthony Fernandez	Undergrad - B.S	Mechanical Eng.	Mr. Amer Awwad	Evaluation of Nonmetallic Components in the Waste Transfer System
Aref Shehadeh	Undergrad - B.S.	Envr. Eng.	Dr. Yelena Katsenovich	Monitoring of U(VI) Bioreduction after ARCADIS Demonstration at F-Area
Awmna Kalsoom	Undergrad - B.S.	Chemistry	Ms. Angelique Lawrence	Surface Water Modeling of Tims Branch
Brian Castillo	Undergrad - B.S.	Biomedical Eng.	Ms. Aparna Aravelli	Development of a Micromachined Ultrasonic Transducer System for Analysis of High Level Waste Pipes at Hanford
Christin Pino	Undergrad - B.S.	Chemistry	Dr. Ravi Gudavalli	Synergistic Effects of Silica and Humic Acid on U(VI) Removal
Christine Wipfli	Undergrad - B.S.	Envr. Eng.	Dr. Vasileios Anagnostopoulos	Groundwater Remediation at SRS F/H Area
Christopher Strand	Undergrad - B.S.	Civil & Env. Eng.	Dr. Noosha Mahmoudi	Surface Water Modeling of Tims Branch
Claudia Cardona	Graduate - Ph.D.	Envr. Eng.	Dr. Yelena Katsenovich	Sequestering Uranium at the Hanford 200 Area Vadose Zone
Erim Gokce	Undergrad - B.S.	Mechanical Eng.	Mr. Anthony Abrahao	Development of Inspection Tools for DST Primary Tanks
Gene Yllanes	Undergrad - B.S.	Electrical Eng.	Dr. David Roelant	Evaluation of FIU's SLIM for Estimating the Onset of Deep Sludge Gas Release Events

Hansell Gonzalez	Graduate - Ph.D.	Chemistry	Dr. Yelena Katsenovich	Sorption Properties of Humate Injected into the Subsurface System
Iti Mehta	Undergrad - B.S.	Mechanical Eng.	Dr. Aparna Aravalli	Investigation Using an Infrared Temperature Sensor to Determine the Inside Wall Temperature of DSTs
Janesler Gonzalez	Undergrad - B.S.	Mechanical Eng.	Mr. Joseph Sinicrope	Incombustible Fixatives
Jesse Viera	Undergrad - B.S.	Mechanical Eng.	Mr. Joseph Sinicrope	Incombustible Fixatives
John Conley	Undergrad - B.S.	Mechanical Eng	Mr. Amer Awwad	Evaluation of Nonmetallic Components in the Waste Transfer System
Jorge Deshon	Undergrad - B.S.	Computer Eng.	Dr. Himanshu Upadhyay	Information Technology for Environmental Management
Kiara Pazan	Undergrad - B.S.	Envr. Eng.	Dr. Ravi Gudavalli	Modeling of the Migration and Distribution of Natural Organic Matter injected into Subsurface Systems
Maximiliano Edrei	Graduate – M.S.	Mechanical Eng.	Dr. Dwayne McDaniel	Computational Fluid Dynamics Modeling of HLW Processes in Waste Tanks
Meilyn Planas	Undergrad - B.S.	Electrical Eng.	Mr. Joseph Sinicrope	Incombustible Fixatives
Natalia Duque	Graduate – M.S.	Envr. Eng.	Dr. Noosha Mahmoudi	Surface Water Modeling of Tims Branch
Orlando Gomez	Graduate - Ph.D.	Physics	Mr. Joseph Sinicrope	Incombustible Fixatives
Robert Lapierre	Graduate – M.S.	Chemistry	Dr. Yelena Katsenovich	Sequestering Uranium at the Hanford 200 Area Vadose Zone
Ryan Sheffield	Undergrad - B.S.	Mechanical Eng.	Mr. Hadi Fekrmandi	Development of Inspection Tools for DST Primary Tanks
Sarah Bird	Undergrad - B.S.	Envr. Eng.	Dr. Ravi Gudavalli	Modeling of the Migration and Distribution of Natural Organic Matter injected into Subsurface Systems
Sebastian Zanlongo	Graduate – Ph.D.	Computer Science	Dr. Dwayne McDaniel	TBD
Silvina Di Pierto	Graduate - Ph.D.	Chemistry	Dr. Hilary Emerson	Evaluation of Ammonia for Uranium Treatment
Yoel Rotterman	Undergrad - B.S.	Mechanical Eng.	Dr. David Roelant	Sustainable Remediation

The research being conducted by the DOE Fellows in support of DOE EM includes the following brief descriptions.

DOE Fellow Hansell Gonzalez Raymat has been studying a commercially available low cost unrefined humic substance known as HumaK as a possible amendment for the remediation of acidic groundwater at the Savannah River Site (SRS). Humic substances can remove contaminants such as uranium, which is one of the key contaminants of concern in the groundwater at SRS. In his study, he has performed a detailed characterization of SRS sediments and HumaK by means of SEM/EDS, FTIR, and potentiometric titrations, which will elucidate the mechanism of HumaK sorption onto SRS aquifer sediments after injection. Also, he has performed batch sorption experiments using SRS sediments and HumaK to investigate various parameters that may affect the sorption behavior of HumaK onto SRS sediments such as pH, contact time, and concentration.

DOE Fellow Jesse Viera has been pursing efforts testing and evaluating the FX2 Advanced Fogging Technology in conjunction with Idaho National Laboratory. The development of this fixative will assist in fixing radioactive particles that are found in decommissioned sites. In addition, Jesse and his team are currently conducting research and testing on possible incombustible fixatives. If successful, an incombustible fixative can provide safer decommissioned facilities for the Department of Energy by trapping radioactive fixatives while maintaining high fire resiliency.

DOE Fellow Jorge Deshon's contributions to the Deactivation and Decommissioning Knowledge Management Information Tool (D&D KM-IT) involve reviewing and editing the SQL database through the use of Microsoft Excel. This allowed the Vendor and Technology modules to be kept up-to-date for customers using the information tool. In an effort to generate more traffic through the D&D KM-IT site, editing of secondary sites such as Wikipedia were completed to increase search results through the web. Currently, the task right now is to make a more mobile-friendly version of the D&D KM-IT site by making it more responsive. This is paired along with the task of developing native apps through the use of C# with the Microsoft Visual Studio integrated development environment.

DOE Fellow Anthony Fernandez is supporting the testing of non-metallic materials to determine if EPDM rubber and Garlock can withstand high-level nuclear waste applications. The testing performed on the materials will include variable temperatures, variable lengths of time, high caustic conditions (specifically sodium hydroxide, NaOH). The effects of varying radioactive conditions will be obtained mathematically. The results obtained from performing multiple aging procedures on the materials will help determine the material that will be used in future Hanford waste transfer lines, which will be constructed for the new Waste Treatment Plant.

DOE Fellow Natalia Duque is supporting the development of a hydrodynamic model of flow for Tims Branch located at the Savannah River Site. When completed, the model will be capable of simulating extreme events such as heavy rainfall and flooding, and it will be coupled with a contaminant transport module to predict the fate of contaminants, such as tin, under varying environmental conditions. In addition, Natalia has been helping the hydrological group in the development of various Geographical Information Systems (GIS) files to further understand the hydrodynamics of Tims Branch Watershed and to be used as inputs in the development of different models.

Milestones and Deliverables

The milestones and deliverables for Project 4 for FIU Year 6 are shown on the following table. Development, review, and site approval of the DOE Fellow summer 2015 internship reports were completed. The reports are available and have been posted in the DOE Fellows website http://fellows.fiu.edu under the "DOE Fellows Internship Reports" tab. The DOE Fellows Class of 2015 was identified and recruited, and a list was sent to DOE. The DOE Fellow Induction Ceremony (milestone 2015-P4-M3) was held on November 5, 2015.

FIU Year 6 Milestones and Deliverables for Project 4

Milestone/ Deliverable	Description	Due Date	Status	OSTI
2015-P4-M1	Draft Summer Internships Reports	10/16/15	Complete	
Deliverable	Deliver Summer 2015 interns reports to DOE	10/30/15 Reforecast	Complete 11/30/15	OSTI
Deliverable	List of identified/recruited DOE Fellow (Class of 2015)	10/30/15	Complete	
2015-P4-M2	Selection of new DOE Fellows – Fall 2015	10/30/15	Complete	
2015-P4-M3	Conduct Induction Ceremony – Class of 2015	11/05/15	Complete	
2015-P4-M4	Submit student poster abstracts to Waste Management Symposium 2016	01/16/16	On Target	
Deliverable	Update Technical Fact Sheet	30 days after end of project	On Target	

Work Plan for Next Quarter

- Continue research by DOE Fellows in the DOE-EM applied research projects under the cooperative agreement and research topics identified as part of their summer 2015 internships.
- Submit final technical paper to WM16. Prepare presentation and present at the conference.
- Coordinate travel for DOE Fellows to attend Waste Management 2016 Symposium.
- DOE Fellows to develop posters and present at student poster session at WM16.
- Begin Spring 2016 campaign to recruit DOE Fellows into the program.
- Begin coordination of internship placements for summer 2016 at DOE sites, national laboratories, DOE HQ, and DOE contractor locations.