Oil & Natural Gas Technology

DOE Award No.: DE-FE0024296

Quarterly Research Performance Progress Report

(Period ending: 03/30/2016)

Methods to Enhance Wellbore Cement Integrity with Microbially-Induced Calcite Precipitation (MICP)

Project Period: October 1, 2014 – September 30, 2018

Submitted by:
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Office of Fossil Energy

ACCOMPLISHMENTS

Goal

The goal of this project is to develop improved methods for sealing compromised wellbore cement in leaking gas wells, thereby reducing the risk of unwanted upward gas migration. To achieve this goal an integrated workplan of laboratory testing, simulation modeling and field testing is underway. Laboratory testing and simulation modeling are being conducted at the Center for Biofilm Engineering (CBE) at Montana State University (MSU) and field testing will take place at the 1498 m (4915 foot) deep Alabama Power Company well located at the Gorgas Power plant in Walker County, Alabama (Gorgas #1 well). This project will develop technologies for sealing compromised wellbore cement using the process known as microbially induced calcite precipitation (MICP). The project has two main objectives:

Objective 1: Prepare for and conduct an initial MICP field test aimed at characterizing a region of compromised well cement in the Gorgas which is suitable for MICP sealing. The location for MICP sealing has now been chosen to be in the interval of 310.0 -310.9 m (1017-1020 feet) below ground surface (bgs). The first MICP sealing test is scheduled for late April 2016.

Objective 2: After thorough analysis of the results from the first field test, conduct a second MICP test using improved MICP injection methods. The second field test will target compromised wellbore cement located above the underground coal seam at an as yet undetermined location.

After each test at Gorgas the following methods will be employed to assess effectiveness of the MICP seal: Pressure falloff testing, sustained natural gas flow rate testing at the well head, USIT (ultrasonic imaging tool) logging to assess the cement bond log, and side wall coring. Successful demonstration of improving wellbore integrity and sealing gas leaks from poor cement bond regions will result in a reduction in the pressure falloff, reduction in the sustained gas flow rate at the well head, noticeable differences in the USIT data in the targeted biomineralization regions, and demonstration of MICP byproducts ($CaCO_3$) in the treated regions on side wall cores.

The project milestones are shown below in Table 1. This table was updated to reflect the change in milestone dates per the one year no-cost time extension that went into effect October 1, 2015.

Table 1. Project Milestones

Related Task	Milestone Number	Milestone Title	Planned Completion Date	Revised Completion Date	Verification Method
1.0	1	Update Management Plan	11/30/2014	NA	Project Management Plan
1.0	2	Kickoff Meeting	11/06/2014	NA	Presentation
2.1	3	Complete construction and testing of wellbore-cement analog testing system.	3/31/2015	NA	Quarterly Report

		Expected result is a system which facilitates biomineralization sealing in annular spaces representative of field conditions.			
3.2	4	Complete first wellbore cement remediation field test. Expected results include obtaining side wall cores and pressure testing to evaluate the extent of biomineralization sealing.	9/30/2015	9/30/2016	Quarterly Report
4.1	5	Complete analysis of field data from first field test. Expected result is a data set which will enhance the design of the second field test.	3/31/2016	3/31/2017	Quarterly Report
4.1	6	Complete design of injection protocol for second field test.	9/30/2016	9/30/2017	Quarterly Report
5.2	7	Complete second field test. Expected results include obtaining side wall cores and pressure testing to evaluate the extent of biomineralization sealing.	3/31/2017	3/31/2018	Quarterly Report
6.0	8	Complete analysis of laboratory, simulation modeling and field data. The expected result will be a comprehensive evaluation of MICP	9/30/2017	9/30/2018	Quarterly Report

		sealing technology for well cement repair.			

Accomplishments under the goals

Project Planning
During this reporting multiple teleconference calls have been conducted including Jim Kirksey of Schlumberger (SLB), Robin Gerlach, Lee Spangler, Al Cunningham and Adie Phillips (MSU), and Randy Hiebert of Montana Emergent Technologies (MET). Issues discussed have mainly centered on planning for our team to visit the Gorgas well site April 11-20, 2016 to conduct the wellbore cement remediation test.

Gorgas Field Test Plan
Based on these data and observations from the December 2015 sidewall coring and pressure testing, it was the collective opinion of CBE researchers and SLB that the well cement in the 310.0 – 310.9 m (1017-1020 feet) bgs interval offers a good candidate for sealing with MICP technology. Accordingly, in April 2016 we will conduct the MICP sealing test at this interval (see Figure 1).

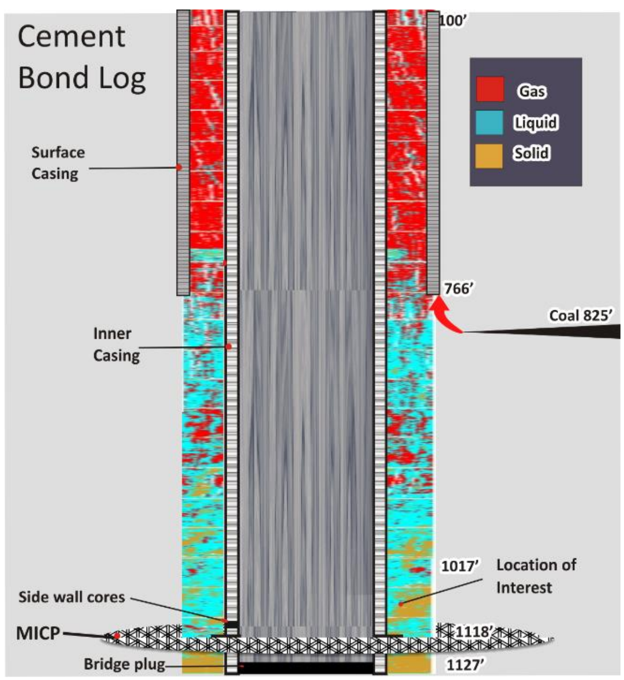

Figure 1. Schematic showing the cement bond log for the Gorgas well based on December 2015 imaging. The target zone for the April 2016 MICP cement sealing test will be the interval 310.0 – 310.9 m (1017-1020 feet) bgs as shown above.

April 2016 DRAFT Work Plan. (entries in red represent items still in the planning stage)

On **Friday, April 8, 2016,** MSU, MET and Schlumberger (SLB) will move on-site, receive rental equipment and chemicals, and begin cultivating microbes. MSU, SLB, and MET will assemble and test all pumps, surface tubing, sampling equipment, mobile chemical testing laboratory, and microbial laboratory. SLB will workover the pump skid and receive the chemicals.

MSU will immediately begin cultivating microbial inoculum culture (*Sporosarcina pasteurii*) for injection. This process will likely take 2-4 days. Two separate coolers of frozen stocks of culture on dry ice will be shipped overnight to the Jasper hotel when a MSU representative is available on-site to receive them. One 50 ml (1.70 ounces) centrifuge tube of BHI+urea and a BHI+urea plate shipped in a separate box will be inoculated just prior to shipment as a back-up in case the frozen stocks do not grow or are somehow compromised. Sterile BHI+ Urea medium will be prepared in 250 ml (8.45) plastic screw top flasks and inoculated with the frozen stock. These flasks will be used to maintain continuous overnight cultures of *S. pasteurii* which can be used to inoculate larger carboys that will be used to fill the bailer for subsurface inoculation. To fill one bailer full of *S. pasteurii* culture 11.34 L (~3 gallons) of culture is required. The carboy growth medium will be prepared by mixing (as aseptically as possibly) YE- (5 g/L Yeast Extract) ingredients to DI (deionized) water (Walmart or Winn Dixie) into a sterile (or disinfected carboy) prior to inoculation with 150 ml (5.07 ounces) of the overnight flask. From lab studies, it has been observed that a 10^7 cfu/ml density of cells can be achieved in 24 hours in 10L (2.64 ounces) of CMM- stirring on a stir plate at room temperature. Since it is uncertain whether the temperature at the site will inhibit or slow growth, seed heating mats will be placed into the cooler with the carboys and stir plates to warm the culture. Finally, a contingency plan for contamination of a carboy should be considered. If contamination overtakes the *S. pasteurii* culture the culture should be discarded and the carboy disinfected with a 1% bleach solution and rinsed with filtered water prior to re-inoculation from an overnight 150 ml (5.07 ounces) flask (or a new carboy should be used). The *S. pasteurii* inoculum will be tested for viability. Cultures will be monitored to assess contamination by plating on BHI+Urea agar plates. If the colony types appear different, if the plate does not smell of ammonia, then it can be determined that contamination has occurred. Unfortunately, the plate assessment method requires at least overnight for growth so to directly assess contamination, a sample of the inoculum from the carboy will be measured for pH, conductivity, and urea or ammonium concentration. Table 2 lists the inoculation schedule.

Table 2. Inoculation and growth schedule (Note: repeat day 1, 2 to day 8 as necessary)

Action	Day -1	Day 0	Day 1		Day 2	
	(Mid Day)	(Mid Day)	(Mid Day)	(Evening)	(Mid Day)	(Evening)
Start from Stock	Frozen stock into 150 ml in 250 flask	Frozen stock into 150 ml in 250 flask	Frozen stock into 150 ml in 250 flask		Frozen stock into 150 ml in 250 flask	
Transfer		Transfer 150 ml to 12L YE-		Transfer 150 ml to 12L YE-		Transfer 150 ml to 12L YE-

Inoculate with bailer			Inoculate with 11.3L	Inoculate with 11.3L	Inoculate with 11.3L	Inoculate with 11.3L

MSU/SLB will procure treated river water from the Gorgas plant to use as injection water. This water will be stored in two 550 gal tanks which can be re-filled as needed. Salt (adjusted to a final formation concentration of 24 g/L) will be added to the water. The salt will be delivered on site with the urea and calcium on Friday, April 8, 2016. MSU/MET will run surface tests to confirm that treated river water, plus urea-nutrient amendment mixture, plus calcium will result in calcium carbonate precipitation at acceptable rates.

On **Monday, April 11th, and Tuesday April 12th, 2016,** Schlumberger will move on-site and mobilize equipment including slickline truck and rig crew. SLB will run 2 7/8 inch tubing (referred to here after as "tubing"). The packer will be attached to tubing but not engaged at this point.

SLB will fill the bottom portion of the 9 5/8 inch well with sodium chloride-river water solution of approximately 1.2 specific gravity. This fluid will be pumped into place, between the existing core points and the bottom plug (approximately 20 feet). By filling the sump with fluid of higher density than the other injection fluids, injection fluids will be encouraged to flow into the formation instead sinking through the low density fluid. Once the high density fluid has been placed, the packer will be set (APPENDIX A). All subsequently added biomineralization fluids and microbial growth media components will be delivered using a 3.0 gallon delivery bailer.

On **Tuesday April 12th, 2016,** the fracture will be inoculated by injecting 3.0 gallons of overnight grown inoculum (amended with 5 g/L YE and 24 g/L urea) through the bailer followed by ~6 gallons of river water (with 24 g/l NaCl). The brine will be injected through the tubing at 0.5 gpm to dilute the inoculum/growth solution and push it into the fracture. Periodically, the bailer will be exchanged to a sampling type Kuster sampler and the delivery bailer will be decontaminated with a 1% bleach solution followed by rinsing with river water. Each bailer of inoculum or concentrated growth/calcium solution should be sampled and at a minimum pH and conductivity should be recorded. Additional analysis on bailer or down hole fluids are possible and include ammonium (Hach Method), Urea (Tecan Method), and Calcium (Hach Digital Titrator). Samples will also be filtered into micro or 15 ml (0.50 ounce) centrifuge tubes to ship back to MSU for analysis. Particularly important for inoculation is to assess the optical density (OD) and perform a population assay (phosphate-buffered saline (PBS) dilution and plate onto BHI+Urea) to confirm the number of cells being added.

On **Wednesday April 13th, 2016,** after a period of overnight attachment a sampling bailer will be used first thing to collect a sample and assess ureolytic activity, which if deemed unsatisfactory will be followed by re-inoculation of a bailer full of cells amended with fresh urea and yeast extract. A six gallon brine rinse will be performed to minimize mixing zone contact of cells and calcium after which bailers of concentrated calcium/growth solution (Table 3) will be injected. Brine will be pumped down the tubing at a rate of 0.5 gpm to dilute and push the reagents into the fracture from the mixing zone. A 30 minute shut in/batch period will follow and if time allows, three more bailers of concentrated calcium/growth solution will be added to the formation followed by ~6 gallons of river water pumped down the tubing at 0.5 gpm to dilute and push the reagents from the mixing zone into the fracture. During each shut in period the pressure will be monitored to assess the fall off pressure or improvement in wellbore integrity. Down-hole sampling will occur periodically as deemed necessary.

Table 3. Concentrated calcium/growth solution.

Ingredients	Original (g/L)	3X Conc. (g/L)
$CaCl_2$	41.3	123.9
NH_4Cl	1	3.0
Urea	24	72.0
Yeast Extract	1	3.0
NaCl	24	24.0

At mid- day, six gallons of rinse will occur to rinse the mixing zone of calcium prior to re-inoculation to prevent suspension clumping. Following the rinse, the fracture will be re-inoculated with 1-2 bailer(s) full of amended culture and pushed into the fracture with six gallons of river water. A one hour reaction period will be allowed followed by a brine rinse and additional calcium pulses. At the end of the day, one bailer of inoculum with fresh urea and yeast extract amendment will be added and the well will be shut in overnight. Refer to Table 4 for complete injection scenario details. This procedure will be repeated over the course of the next few days until evidence of formation/fracture sealing is observed.

Table 4. Injection scenario details (note: all times listed are only estimates)

Day Zero and Before	Fill sump with 1.2 SG NaCl solution	
	Start growing inoculum	
First day	Condition with growth solution (1 bailer)	11:00 AM
	Inoculate (1 bailer)	12:00 PM
	Rinse –brine	1:45 PM
	Calcium (5 bailers)	2:00 pm to 6:00 PM
	Rinse- water	6:55 PM
	Re-inoculate (1 bailer)	7:10 PM
Second day	Sample mixing zone	7:00 AM
	Rinse –brine	7:15 AM
	Calcium (5 bailers)	7:30 AM- 11:30 AM
	Rinse –brine	12:30 PM
	Re-inoculate (1 bailer)	1:00 PM
	Rinse –brine	1:30 PM
	Calcium (5 bailers)	2:00 pm to 6:00 PM
	Rinse –brine	6:30 PM
	Re-inoculate (1 bailer)	7:10 PM
Third-Eight Day	Keep going with injection if no perm reduction, or shut in followed by falloff testing and logging	

Criteria to determine test completion: Monitor pressure/flow relationship to remain below fracturing pressure. At this time, assume the maximum allowable pressure is 900 psi. We will continue to lower the injection flow rate until the lower limit of pump is reached (about 0.15 gpm). After each injection, a mechanical integrity test (MIT) for 10 minutes will be performed where the wellbore pressure will be monitored. Test results will be compared with the initial pressure fall-off test(s). At the end of the experiment, the USIT log will be run to assess changes before and after the MICP treatment.

Supporting Laboratory Tests

During this reporting period additional laboratory analog testing has also been completed. The MICP test system shown below in Figure 2 was developed and tested by our collaborators at MET. As the Figure 2 schematic shows, this reactor consists of a carbon steel plate overlaying a plate structure composed of fine cement. The cement plate structure has raised borders so as to create a gap between steel and cement, thereby simulating a wellbore delamination between casing a surrounding cement. This 61 cm (24 inch) long reactor system is referred to as the "fracture fixture" reactor. The steel-cement gap consists of four 15.2 cm (6 inch) sections arranged in series with gap widths of 4 mm, 2 mm, 0.2mm, and 4 mm (0.157, 0.079, 0.0079, and 0.157 inch) respectively (see Figure 3). This gap arrangements simulates a delamination of variable width along the outside of the well casing. The minimum gap width of 0.2 mm (0.0079 inch) was chosen based on analysis of the December 2015 pump testing data at Gorgas.

The goal of the laboratory testing with this reactor was to successfully plug the 0.2 mm (0.0079 inch) gap using MICP to precipitate calcium carbonate. MICP sealing tests resulted in the calcium carbonate distribution (for each of the four gap widths) as shown in Figure 3. The laboratory MICP sealing tests using the fracture fixture reactor consisted of establishing an initial flow rate through the (clean) reactor, adding *Sporosarcina pasteurii* inoculum, urea and calcium using the same procedure planned for the Gorgas field test, then monitoring the reduction in flow rate and corresponding pressure increase as calcium carbonate deposition progressed. After three days of operation, a three order of magnitude reduction in reactor permeability was observed, due mainly to the mineral deposition occurring in the 0.2 mm (0.0079 inch) gap (Figure 4 shows a close up photograph of the calcium carbonate deposed in the 0.2 mm (0.0079 inch) gap). This outcome was considered very successful as a laboratory analog for the up-coming wellbore cement field test at Gorgas.

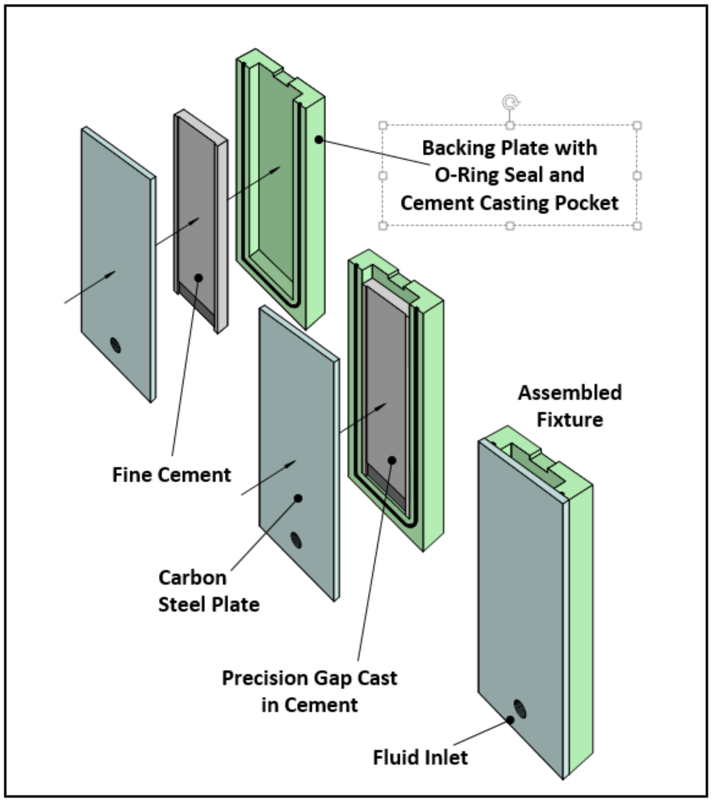

Figure 2. Fracture fixture reactor system simulating a delamination gap between wellbore cement and well casing. MICP testing was done to seal the gap between steel and cement with calcium carbonate.

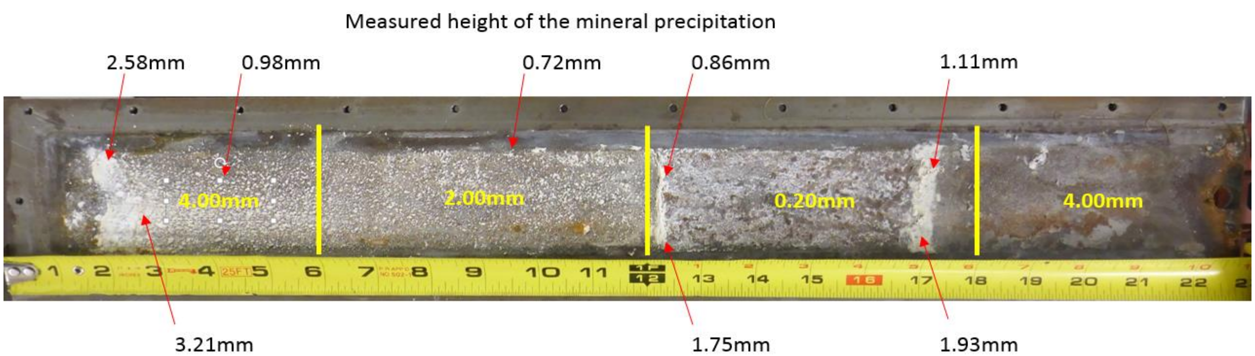

Figure 3. Calcium carbonate distribution along the Fracture Fixture reactor after MICP testing

Figure 4. Calcium carbonate distribution within the 0.20 mm gap.

Opportunities for training and professional development

Dr. Adrienne Phillips was a Ph.D. student in Environmental Engineering when this proposal was written in June 2014. She was subsequently hired as an Assistant Professor in Environmental Engineering at Montana State University. As a Co-PI on this project and with years of biomineralization laboratory and field project management, she was the likely candidate to step in as a temporary PI during Al Cunningham's five month break in service July–November, 2015. Therefore, this project is affording Adie the opportunity for professional development by serving as a Principal Investigator. It is the opinion of Lee Spangler, Project and Al Cunningham that, given Adie's high level of accomplishment during fall 2015, together with her leadership in organizing and conducting the successful December 2015 field test, it is clear that she is eminently qualified and will continue serving as project PI on a permanent basis. Adie is currently leading the effort to mobilize and conduct the MICP wellbore sealing field test at the Gorgas facility scheduled for April 11-20, 2016. Dr. Cunningham has now completed his mandatory five month service break, has re-joined the project, and will continue to serve as a co-principal investigator.

Eric Troyer, a Chemical and Biological Engineering graduate from MSU (Dec. 2015), has worked on the team for the last three years during his undergraduate education. His undergraduate research efforts included screening tirelessly sources of chemicals that can promote precipitation economically (he determined urea fertilizer and calcium ice melting products worked well). He is now a Research Engineer on the team and will be much of the effort for the field experiment in April 2016. He will be attending graduate school at UC Berkley in the fall and was recently awarded a fellowship with the National Science Foundation Graduate Research Fellowship Program.

Disseminating results to communities of interest

Project results will be disseminated in a timely fashion through publications, conference participation, etc. During this reporting period the following manuscript was accepted and printed by *Environmental Science and Technology* Journal:

Adrienne J. Phillips, Alfred B. Cunningham, Robin Gerlach, Randy Hiebert, Chaichi Hwang, Bart Lomans, Joe Westridge, Cesar Mantilla, Jim Kirskey, Richard Esposito, Lee Spangler, 2016. Fracture Sealing with Microbially-Induced Calcium Carbonate Precipitation: A Field Study. Publication Date (Web): February 25, 2016, **DOI:** 10.1021/acs.est.5b05559

Also Dr. Adrienne Phillips presented "Biomineralization Sealing Technology- A Technology Developed in Montana", presented to the Montana Energy Conference, March 30, 2016, Billings Montana.

Planned activities during the next reporting period

The major activity planned for next reporting is to conduct the MICP wellbore sealing test at the Gorgas facility scheduled for April 11-20, 2016.

Products

Publications

Adrienne J. Phillips, Alfred B. Cunningham, Robin Gerlach, Randy Hiebert, Chaichi Hwang, Bart Lomans, Joe Westridge, Cesar Mantilla, Jim Kirskey, Richard Esposito, Lee Spangler, 2016. Fracture Sealing with Microbially-Induced Calcium Carbonate Precipitation: A Field Study. *Environmental Science and Technology*. Publication Date (Web): February 25, 2016, DOI: 10.1021/acs.est.5b05559

Conference Presentations

Phillips, A.J., A. Cunningham, R. Gerlach, and Spangler, L. Biomineralization Sealing Technology- A Technology Developed in Montana, Presented to the Montana Energy Conference, March 30, 2016, Billings Montana.

Other organizations involved as partners

Schlumberger (SLB) (formerly Schlumberger Carbon Services (SCS)). SLB is providing matching support for this project. SCS field workers, led by Jim Kirksey, will help identify and characterize the test locations in the Gorgas well, perform the packer initialization, injection of biomineralization fluids, pre- and post-experiment pressure tests, and well logging and coring. During this reporting period, Jim Kirksey and others from SLB conducted analysis of the side wall coring and logging at the Gorgas well and prepared equipment, subcontractors, and other details for the field experiment.

Southern Company (SC). SC is providing matching support for this project. Dr. Richard Esposito of SC, has identified and secured the 1493 m (4915 foot) deep well (Gorgas #1 well, Walker County, Alabama) to be used for our MICP field tests.

Montana Emergent Technologies (MET). MET attended meetings where discussion surrounded the current laboratory efforts and the field planning. MET will be mobilizing to the Gorgas #1 well, Walker County, Alabama to assist at the April 11-20, 2016 field test.

University of Alabama at Birmingham (UAB). Dr. Peter Walsh is in charge of the UAB Core Testing Laboratory. He will be conducting core testing activities throughout the duration of this project.

University of Stuttgart. Dr. Rainer Helmig, Director of the Institute for Modelling Hydraulic and Environmental Systems (IWS), and Johannes Hommel, Ph.D. Student, are project collaborators at the University of Stuttgart. They along with other colleagues have developed a reactive transport simulation model, referred to herein as the Stuttgart MICP model, that has been integrated with previous laboratory and field research. This model was successfully used to help design the Gorgas field test in April 2014, and will be used again for the design of both laboratory field tests for the current project.

IMPACT

While too soon to evaluate all of the direct impacts of this project, one positive impact is a recently awarded funding announcement. The proposal was recently funded by DOE in response to DE-FOA-0001240, AOI 3 “Advanced Materials and Methods for Mitigating Wellbore Leaks”. The proposal is entitled “Wellbore Leakage Mitigation using Advanced Mineral Precipitation Strategies” where we seek to expand the temperature and depth of the current microbially based solutions with enzymatic or other mineral precipitation strategies.

Impact will be addressed in future reports as appropriate.

Dollar amount of award budget spent in foreign country(ies)
February 11-18, 2016, Dr. Al Cunningham traveled to Stuttgart, Germany for research planning with the University of Stuttgart partners. The total cost of the trip was $2,111.85.

CHANGES/PROBLEMS

As of this reporting period there are no problems to report. As noted below, the project milestone deadlines have been revised due to the budget period 1 no cost extension,.

SPECIAL REPORTING REQUIREMENTS

At this time there are no special reporting requirements.

BUDGETARY INFORMATION

Table 5. Cost Plan Status

					NO COST EXTENSION				
Baseline Reporting Quarter	YEAR 1 Start:	10/1/2014	End:	9/30/2015	YEAR 2 Start:	10/1/2013	End:	9/30/2014	Total
	Q1	Q2	Q3	Q4	Q5	Q6	Q7	Q8	
Baseline Cost Plan (from SF424A)									
Federal Share	163,575	163,575	163,575	163,575					654,300
									-
Non-Federal Share	31,739	31,739	31,739	31,739					126,956
									-
Total Planned Shares	195,314	195,314	195,314	195,314	-	-	-	-	781,256
									-
Cumulative Shares	195,314	390,628	585,942	781,256					781,256
Actual Incurred Costs									
Federal Share	6,268	19,082	30,237	53,029	83,125	165,886			357,626
									-
Non-Federal Share			53,559	51,624	-	12,527			117,709
									-
Total Incurred Costs	6,268	19,082	83,796	104,652	83,125	178,413	-	-	475,336
									-
Cumulative Incurred Costs	6,268	25,350	109,146	213,798	296,923	475,336			475,336
Variance									
Federal Share	157,307	144,493	133,338	110,546	(83,125)	(165,886)	-	-	296,674
									-
Non-Federal Share	31,739	31,739	(21,820)	(19,885)	-	(12,527)	-	-	9,247
									-
Total Variance	189,046	176,232	111,518	90,662	(83,125)	(178,413)	-	-	305,920
Cumulative Variance	189,046	365,278	476,796	567,458	(296,923)	(475,336)	-	-	305,920

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