Oil & Natural Gas Technology

DOE Award No.: DE-FE0024296

Quarterly Research Performance Progress Report

(Period ending: 09/30/2015)

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 at Montana State University and field testing will take place at the 1498 m (4915') 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 sealing a poor well cement bond in the Gorgas well approximately located 820 feet (249 meters) below ground surface (bgs).

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 approximately 960 feet (293 meters) bgs at Gorgas.

Note. *The proposed elevations of the planned field tests at Gorgas well (i.e. 820 and 960 feet bgs) are no longer considered the most suitable locations for the experiment. After side wall coring and re-running the cement bond log, it is anticipated that the 1017' (~310m) bgs region will be the most suitable elevation for the first test. Additional side wall coring and site characterization will occur in late October 2015.*

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. Expected result is a system which facilitates biomineralization sealing in annular spaces representative of field conditions.	3/31/2015	NA	Quarterly Report
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 sealing technology for well cement repair.	9/30/2017	9/30/2018	Quarterly Report

Accomplishments under the goals

Major activities completed through this reporting period include: (1) Completing analysis of side-wall core and mud type material retrieved from the Gorgas well and completing analysis of the new cement bond logs, (2) completing development of wellbore cement analog testing system, and (3) performing a wellbore analog MICP experiment to assess potential field injection strategies.

Project Planning

During this reporting period, multiple teleconference calls have been conducted including Jim Kirksey (Schlumberger Carbon Services), Robin Gerlach, Lee Spangler, and Adie Phillips Montana State University). Issues discussed have mainly centered on planning for Jim Kirksey to visit the Gorgas well site and conduct additional side wall coring and pump testing.

Analyzing Side Wall Cores and Cement Bond Logs from the Gorgas Well

In late May, Jim Kirksey visited the Gorgas well and conducted sidewall coring and additional well logging (isolation scanning).

SIDE WALL CORE ANALYSIS

The side wall coring was successful in recovering a cement core plug and a piece of casing material at 1112 feet (340 m) below ground surface (bgs) and some mud type material from a potential channel behind the casing at 1108 feet (337 m) bgs. The core and mud type material was analyzed at our MSU laboratories. The cement portion was scanned at 100kV without a filter using X-ray Micro Computed Tomography (Micro-CT) (Sky Scan 1173, Bruker USA, Wisconsin). Additionally, the channel was observed with a Zeiss stereoscope equipped with a fluorescent imaging attachment. In the Micro-CT analysis of the side wall core from 1112’ (340 m) a channel was observed and auto fluorescent mineral was observed in the region of the channel (Figure 1).

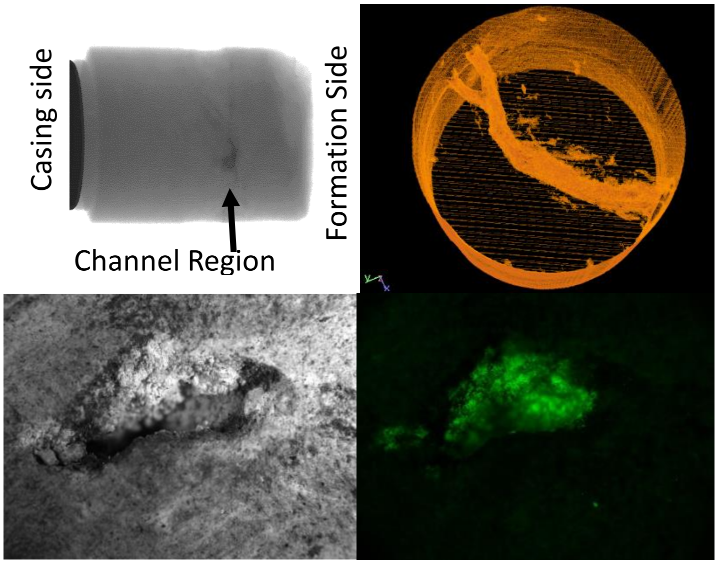


Figure 1. A channel was detected in the cement core as evidenced by the CT scan. Auto fluorescent mineral was detected in the flow channel suggesting calcite may have formed in the channel after the April 2014 MICP fracture sealing experiment that was performed as part of the DOE project DE- FE0009599. This finding is promising for the current project as it suggests that calcite can form in a cement channel environment in the field.

The mud type material was retrieved from the 337.7m (1108') location which in the laboratory was dried in a sterile petri dish on the benchtop prior to the collected particles analyzed by X-Ray Powder Diffraction Spectrometer (XRD) (Scintag X-GEN 4000 XRD). The particles were scanned from 20.0 to 65.0 degrees at 1 deg/min and DMSNT analysis software (Scintag) was used to characterize the mineralogy of the samples. Calcite (data not shown) was detected in the material which may also serve as evidence that biomineralization was promoted not only in the hydraulic fracture but also in a channel behind the casing. These are positive results that suggest the new wellbore cement integrity experiment has potential for success. Additional positive laboratory experiments have been performed and are discussed in the "*Development of wellbore cement analog testing systems"* section below.

CEMENT BOND LOGS

After Schlumberger reviewed the new cement bond logs, the assessment was unfavorable for performing an experiment at elevations higher up in the casing as the void spaces might be too large for effective biomineralization sealing. From their analysis it is thought there is a channel in the cement at 1017' (~310m) below ground between the casing and the shale formation (Figure 2). Additional field characterization is planned for late October 2015. The purpose of the additional field work is to drill another set of side wall cores in the 1017' (~310m) region and set a tubing string and packer to inject brine to assess flow-pressure relationships. These flow-pressure relationships will be used to assess the channel configuration and design and plan for the first field test.

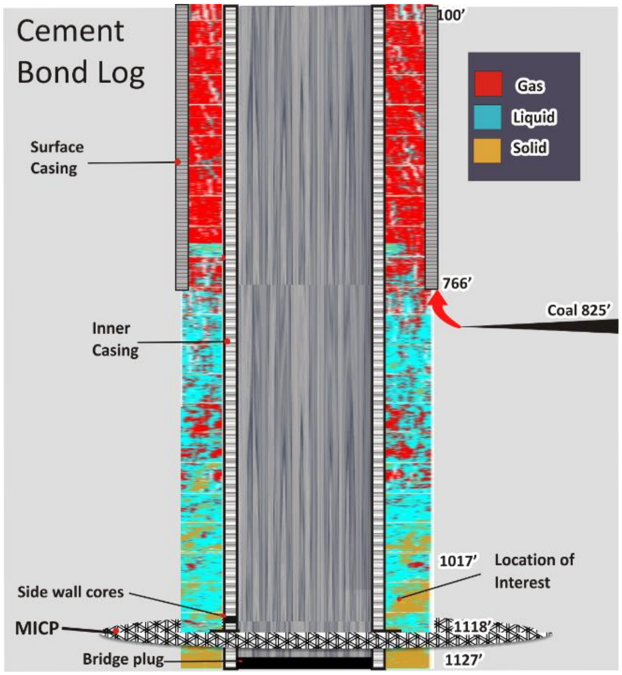


Figure 2. The new cement bond logs suggest that the region at 1017' (~310m) bgs is favorable for an MICP sealing experiment as it is thought a channel exists in the cement behind the casing. The April 2014 MICP fracture sealing experiment that was performed as part of the DOE project DE-FE0009599 was performed at 1118' (340.8m) bgs.

Development of wellbore cement analog testing systems. Several reactor systems have been developed or are in development for the testing of MICP in the near wellbore environment. *These systems represent successful completion of Milestone #3 "Complete construction and testing of wellbore-cement analog testing system. Expected result is a system which facilitates biomineralization sealing in annular spaces representative of field conditions".* Refinement of injection strategies is ongoing in preparation for the first field experiment.

<u>One inch diameter cement/steel cores.</u> As previously reported data has been collected on 1" (2.54 cm) diameter laboratory wellbore cement debonding analog systems (Figure 3). The purpose of this experiment was to assess the ability of microbially-induced calcite precipitation (MICP) to reduce permeability in a defined 100 µm gap in a cement casing analog system. A cement core cylinder was constructed with Class H oil field well cement with a steel rod placed in the center of the core to mimic a cement/casing interface. A thin piece of metal stripping was slid between the rod and the cement core outer cylinder to hold the rod in place and create a constant gap (100 µm) (Figure 3). After the baseline initial permeabilty was calculated, the core was inoculated with a *S. pasteurii* culture (adjusted to an optical density of 0.4 or approximately $1x10^8$ cfu/ml). An attachment period of three hours and forty minutes was allowed before CMM- (3 g/L nutrient broth, 10 g/L NH_4Cl, 20 g/L urea) growth solution was injected for two hours to promote biofilm formation. The injected solution was then swtiched to CMM+ (CMM- with 50 g/L $CaCl_2$-$2H_20$) which was injected for the rest of the experiment (223 total hours). Over the course of the 223 hour experiment, the permeability of the gap was reduced by five orders of magnitude.

<u>Larger scale well-bore cement analog testing system.</u> As reported last quarter, a larger wellbore analog reactor system was constructed. The system consists of a 4 inch (10.16 cm) diameter outside casing and a 2.5 inch (6.35 cm) diameter inner PVC delivery pipe. This results in a 0.44 inch (1.18 cm) gap into which well cement can be placed. Flow of biomineralization fluids from

the inner PVC pipe into the cement region is accomplished via 4 x 5/16 inch (0.794 cm) diameter injection ports. The system is adequate for forthcoming experimental needs and it can be easily modified if needed. The columns are 1.0 foot (30.4 cm) in height (Figure 3).

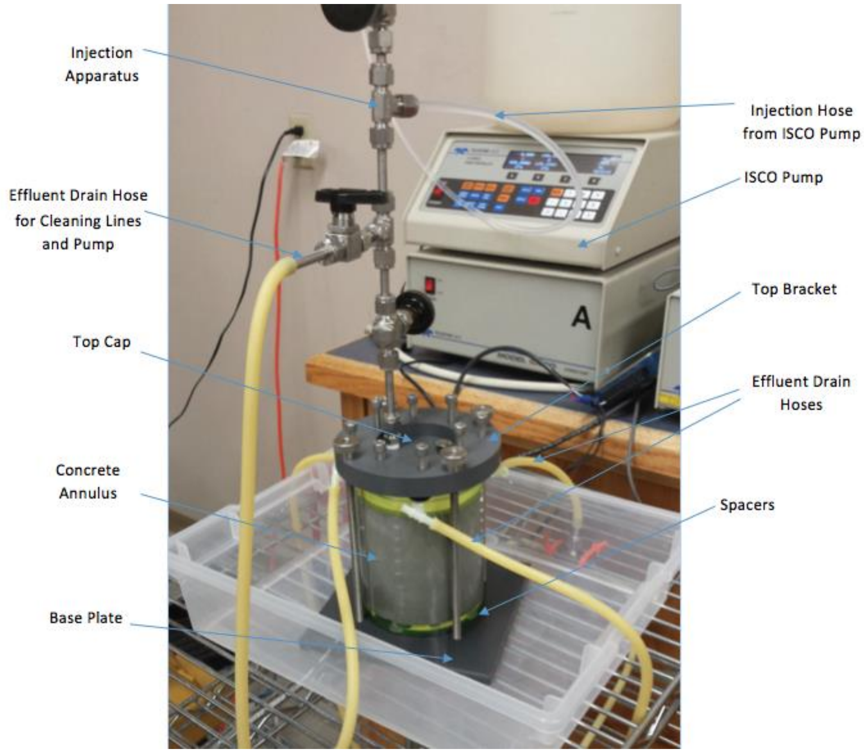


Figure 3. Wellbore-cement analog system composed of two annular PVC conduits with a 0.44 inch (1.18 cm) gap filed with cement. Cement can be fractured or the debonding aberration spaces adjusted in dimension if necessary. The bright green fluid is a fluorescein dye tracer injected to visualize flow paths in the cement annulus.

MICP Wellbore Annulus Sealing Experimental Procedure

An experiment was recently performed in the wellbore analog reactor where MICP promoting substrates were injected to seal a 250 µm gap between the cement and the outer casing.

Cement Preparation

To prepare the cement sample cast, a class H cement mixture was mixed with water in a 4:10 water to cement by weight mixture and blended to ensure homogeneity of the slurry during pouring. Teflon material (10 mil (254 micron)) was attached to the inside of the outer casing of the wellbore analog reactor prior to pouring so that the cast could be removed and to induce a desired debonding type defect 250 micron in aperture. The cement was poured into the annular space between the inner and outer casing and it was allowed to cure for five days. The cement cast was then removed and immersed into water saturated with calcium hydroxide to cure for an additional 14 days at room temperature.

Microbial Inoculum Preparation

A volume of 100 µL of *S. pasteurii* (ATCC 11859) frozen stock culture was added to 100 mL of brain heart infusion amended with 2% by weight urea. The organisms were allowed to grow at 30°C on a shaker at 150 rpm overnight. The day following the beginning of the culture, the 100 mL culture was introduced to 900 mL of fresh YE- (2 g/L yeast extract, 20 g/L urea, 1 g/L NH_4Cl, 24 g/L NaCl) solution. The organisms were then placed on a stir plate and incubated for approximately 24 hours at room temperature. The bacterial suspension was amended with 24 g/L urea and 2 g/L yeast extract before being used as the inoculum. One ml of the 24-hour culture was removed prior to inoculation for dilution and plating.

Nutrient Solution and Calcium Solution Preparation

A yeast extract nutrient growth and calcium containing solution, YE+ and YE-, respectively were prepared. To prepare each solution, tap water was used as the base fluid and the ingredients were added according to their respective concentrations and allowed to dissolve on a stir plate. All solutions were made directly prior to their use to minimize degradation or contamination of the solution prior to injection. A sample from each solution was taken directly prior to injection for analysis. This procedure was followed to mimic an injection strategy that might be deployed in the field.

Reactor Inoculation

At the beginning of the experiment, the inoculum suspension amended with 24 g/L urea and 2 g/L yeast extract was injected at 15.1 mL/min (25 minute residence time) for 2.5 pore volumes of inoculum to allow for sufficient volume to flow through the reactor and feeder lines. After inoculation, the flow was stopped for 4 hours to allow cells to attach to the cement interface.

Biofilm Growth Phase

After allowing four hours for attachment, injection of the nutrient growth medium (YE-) was initiated. Flow was set to 15.4 mL/min for two hours. After two hours, the growth pulse was halted and the reactor was left to stand overnight.

Daily Pulse Schedule

Beginning on day two of the experiment, pulses of calcium were introduced to the reactor in single pore volume increments. These were injected at a flow rate of 15.4 mL/min followed by a 90 minute stationary phase until restriction of flow from permeability reduction. A total of 4 calcium solution pulses were injected per day. Following the stationary phase of the final calcium pulse of the day, the reactor was inoculated with one pore volume of microbial inoculum that was prepared as described above and amended with 24 g/L urea and 2 g/L yeast extract prior to injection. After each inoculation the reactor was left to stand overnight. When changing solutions either to or from YE+ (YE- recipe with the addition of 50 g/L $CaCl_2$-$2H_2O$), two reactor pore volumes of 24 g/L NaCl solution were flowed through the reactor to clear the reactor and the lines of residual substrates that could cause instantaneous precipitation and negatively affect injection of fluids.

Experimental Results

Permeability reduction

The flow rate and differential pressure across the reactor were recorded and the data was used to calculate the effective permeability of the fracture. A four order of magnitude decrease in effective permeability was observed over the course of the experiment. The vast majority of the permeability reduction was seen on days four and five after the 10th pulse of calcium solution.

Calcite precipitation

The permeability reduction mentioned above was observed as the MICP began to become very prevalent in the reactor towards the end of the second day and the beginning of the third day.

The calcite precipitation was seen primarily at the entrance region to the aperture and along channels at the cement interface (Figure 4). This precipitate was formed visibly within the first day of injection and developed over the course of the experiment.

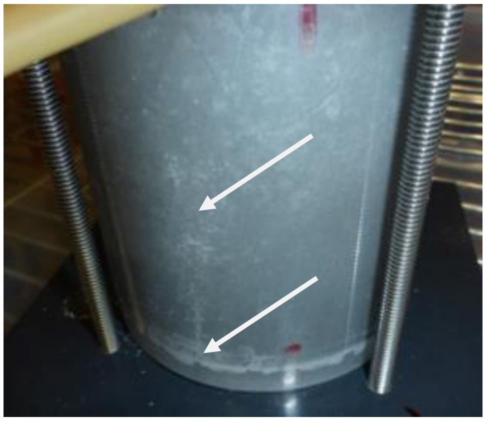


Figure 4. Formation of calcite within the created defect. The calcite was observed to form at the inlet region of the defect and also along preferential flow paths farther into the aperture as indicated by the arrows.

Positive results of this experiment were: (1) significant permeability reduction and (2) the observation of calcite precipitation in the wellbore annulus defect. Additional experiments are planned to refine the injection strategy in larger engineered defects (~500 micron to 2mm) at the cement interface. This will provide an opportunity to seal a larger gap. In addition, we are preparing two new reactor systems that will simulate more realistic field type conditions. The first reactor system has a controllable gap between cement and steel surfaces (as contrasted to the existing reactor's cement polycarbonate interface) (Figure 6a). The second reactor system mimics cement between a surface casing and inner casing and will allow testing for the ability of MICP to reduce gas flow (Figure 6b).

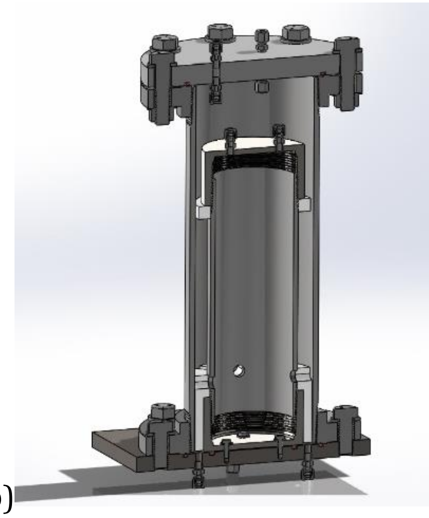
(a)  (b) 

Figure 6. Two new reactor systems in development to study MICP in the cement steel interface. (a) Composite fracture fixture reactor. A plastic mold is used to house a 6" long ¼" thick cement coupon on one side (left most) and a steel plate on the other side. The two pieces are flanged together and MICP promoting fluids can be injected from the bottom of the reactor and flow along the interface between the cement and steel. The reactor is designed so that microscopy and other analysis can occur to examine the surfaces of the cement and steel post MICP treatment. (b) Steel annulus reactor. A steel annulus reactor is in design to mimic a wellbore with a surface casing and an inner casing. Cement with predesigned fractures or aberrations will fill the space between the steel inner and outer casing. The reactor is designed with gas line connections such that after MICP treatment the reduction in gas flow (rather than just liquid flow as previously described) can be assessed.

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. Therefore, this project is affording Adie the opportunity for professional development by serving as a Principal Investigator during Al Cunningham's leave of absence. In addition, a Mechanical Engineering Master's student, Samuel Zanetti has recently been hired for a semester long internship to work on this project. His task is to prepare for field deployment by performing wellbore analog experiments, preparing field equipment, and developing methods to use ultrasound in the laboratory to detect cement bonds in our reactors. This internship will afford Mr. Zanetti interaction with industry (Schlumberger, Shell, Southern Company) enhancing his network, opportunities for learning, and future employment.

Disseminating results to communities of interest

Project results will be disseminated in a timely fashion through publications, conference participation, etc. During this reporting period, a manuscript entitled "Fracture Sealing with Microbially-Induced Calcium Carbonate Precipitation: A Field Study" is being prepared for submission to Environmental Science and Technology (ES&T). Dr. Phillips presented a paper entitled *"Biological influences in the subsurface: A method to seal fractures and reduce permeability with microbially-induced calcite precipitation"* at the ARMA (American Rock Mechanics/Geomechanics Symposium held in San Francisco, CA, 28 June –July 1, 2015. Dr. Robin Gerlach presented "*Field Test and Evaluation of Engineered Biomineralization Technology for Sealing Existing Wells"* at the U.S. Department of Energy, National Energy Technology Laboratory, Carbon Storage R&D Project Review Meeting on August 19, 2015.

Planned activities during the next reporting period

During the next reporting period our project team will continue MICP seal testing on the 1-inch (2.54 cm) core analogs, the 4-inch (10.16 cm) well-bore cement system and the composite fracture fixture reactor. This testing will facilitate development and optimization of MICP injection protocols suitable for developing MICP sealing in de-bonded well cement. Analysis of the side wall coring materials retrieved from the new location of 1017' (~310m) at Gorgas will take place during the next reporting period. We will continue the project planning process by way of teleconferences with Schlumberger, Southern Company, University of Alabama at Birmingham, and Stuttgart University collaborators. Our project team will participate in the Web-based Quarterly Reporting conference which at the time of this report has not yet been scheduled.

PRODUCTS

The following are presentations from this reporting period:

Phillips, A., R. Gerlach, A.B. Cunningham, L.H. Spangler, R. Hiebert, J. Kirksey, R. Esposito, Biological influences in the subsurface: A method to seal fractures and reduce permeability with microbially-induced calcite precipitation, ARMA (American Rock Mechanics/Geomechanics Symposium, San Francisco, CA, June 28, 2015.

Gerlach, R., A.B. Cunningham, L.H. Spangler, Field Test and Evaluation of Engineered Biomineralization Technology for Sealing Existing Wells, U.S. Department of Energy, National Energy Technology Laboratory, Carbon Storage R&D Project Review Meeting, August 19, 2015.

Spangler, L.H., R. Gerlach, A. Cunningham, A. Phillips. Biofilm-Mediated Mineral Precipitation Technology – From the Microscale to the Field-Scale (Poster), Southampton, UK, September 29, 2015.

Other organizations involved as partners

Schlumberger Carbon Services (SCS). SCS 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, well perforation, injection of biomineralization fluids, pre- and post-experiment pressure tests and well logging and coring. During this reporting period, Jim Kirksey and others from SCS conducted analysis of the side wall coring and logging at the Gorgas well. They analyzed the cement bond log to determine a location (depth) for additional field characterization which is anticipated for late October 2015.

Southern Company (SC). SC is providing matching support for this project. Dr. Richard Esposito of SC, together with SCS, 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. During this reporting period, Dr. Esposito visited Montana State University to discuss sidewall coring and logging at the Gorgas well and the anticipated additional field characterization work.

University of Alabama at Birmingham (UAB). Dr. Peter Walsh is in command of the UAB Core Testing Laboratory. He will be conducting core testing activities throughout the duration of this project. Dr. Walsh also attended the December Project Planning meeting in Birmingham, Alabama.

University of Stuttgart. Dr. Rainer Helmig, Director of the Institute for Modelling Hydraulic and Environmental Systems (IWS), and Johannes Hommel, PhD 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. During this reporting period Johannes Hommel worked to develop the near wellbore domain to adapt the model from a fracture to a debonding in the wellbore cement steel interface.

IMPACT

Impact will be addressed in future reports as appropriate.

Dollar amount of award budget spent in foreign country(ies)

No project funds were spent in foreign countries this reporting period.

CHANGES/PROBLEMS

As of this reporting period, a change was made to the timeline of the project. Since the field characterization is not yet complete, budget period 1 has been extended and the project milestone deadlines have been revised.

SPECIAL REPORTING REQUIREMENTS

At this time there are no special reporting requirements.

BUDGETARY INFORMATION

Table 2. Cost Plan Status

Baseline Reporting Quarter	YEAR 1 Start:	10/1/2014	End:	9/30/2015
	Q1	Q2	Q3	Q4
Baseline Cost Plan (from SF424A)				
Federal Share	163,575	163,575	163,575	163,575
Non-Federal Share	31,739	31,739	31,739	31,739
Total Planned Shares	195,314	195,314	195,314	195,314
Cumulative Shares	195,314	390,628	585,942	781,256
Actual Incurred Costs				
Federal Share	6,268	19,082	30,237	53,029
Non-Federal Share			53,559	51,624
Total Incurred Costs	6,268	19,082	83,796	104,652
Cumulative Incurred Costs	6,268	25,350	109,146	213,798
Variance				
Federal Share	157,307	144,493	133,338	110,546
Non-Federal Share	31,739	31,739	(21,820)	(19,885)
Total Variance	189,046	176,232	111,518	90,662
Cumulative Variance	189,046	365,278	476,796	567,458

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