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Wellbore leakage mitigation using engineered biomineralization

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Abstract

Research on microbially induced calcite precipitation (MICP) is reported. MICP may serve to reduce near-wellbore permeability, reduce CO_2–related corrosion, and lower the risk of unwanted migration of CO_2 or other fluids. MICP research on the lab scale has demonstrated the ability to seal sandstone cores using injection strategies engineered to control precipitation. Experimentation was also aimed at transitioning MICP strategies for field implementation. MICP was evaluated in the field in a hydraulically fractured sandstone formation at a Walker County, Alabama well. The field experiment resulted in greatly reduced injectivity indicating that the fractured formation was plugged after MICP treatment.

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Keywords: carbon storage; biomineralization; well remediation; well bore integrity; carbon sequestration storage security; MICP

1. Introduction

1.1. Wellbore Leakage Mitigation

A major risk to geologic carbon sequestration storage security is leakage through abandoned wells, especially in depleted oil and gas reservoirs where large numbers of wellbores are often present. When compromised, the near-wellbore environment can become a key leakage pathway for CO_2 and other fluids to migrate to the surface or into functional aquifers above. The environmental implications of leakage include potential damage to drinking water aquifers or release of greenhouse gases to the atmosphere.

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Leakage pathways can form due to corrosive fluids (CO_2) acting to reduce well cement integrity, fractures in the surrounding formation, or regions of poor cement bonding during well completion [1-4].

The Energy Research Institute (ERI) at Montana State University (MSU) is directing research on the use of microbial biofilms which enzymatically hydrolyze urea, resulting in the precipitation of crystalline calcium carbonate. This process is referred to as microbially induced calcite precipitation (MICP). Use of ureolytic, biofilm-forming bacteria allows control over the distribution of the catalyst that induces calcite formation so precipitation occurs along a length of the leakage pathway instead of solely at the inlet. This method has the potential to reduce near-well bore permeability, coat cement to reduce CO_2–related corrosion, and lower the risk of unwanted migration of CO_2 or other fluids.

1.2. MICP Overview

Microbes alter the chemistry of their surrounding environments, which in some cases can result in supersaturation and precipitation of minerals such as calcium carbonate. One mechanism that has been researched extensively is the microbial enzyme urease, which catalyzes the hydrolysis of urea to produce carbonate species and in the presence of calcium results in calcite ($CaCO_3$) precipitation. MICP has been investigated for a wide range of engineered applications [5] including amending or improving construction materials [6-8], cementing porous media [9-13], and environmental remediation including the potential to reduce subsurface leakage in the context of geologically sequestered carbon dioxide [14-19]. In MICP, the urease reaction influences chemical conditions (saturation state) through: increasing dissolved inorganic carbon (DIC) concentration and increasing pH, effectively increasing the carbonate alkalinity [15, 20-22] (Eq.1).

$$CO(NH_2)_2 + 2H_2O \longrightarrow 2NH_4^+ + CO_3^{2-} \quad (1)$$

The resulting precipitation of $CaCO_3$ depends on the saturation state of the mineral and the presence of nucleation sites as described in detail elsewhere [5]. The urease enzyme can be found in a wide variety of microorganisms [23]. The MICP studies presented here utilized *Sporosarcina pasteurii*, ATCC 11859, formally *Bacillus pasteurii* [24]. MICP has the potential to deposit calcite in preferential flow paths in porous media and in the near-well bore environment to mitigate leakage potential. The primary advantage of the MICP technology is that aqueous solutions with low viscosity are used to promote precipitation that can reduce permeability and seal unwanted flow paths in the near wellbore environment.

2. **MICP Research: lab and field scale**

Laboratory-based investigations have focused on engineering the MICP process to seal both porous media matrix and fractures under field-relevant conditions. This multi-scale research program combines two novel core testing systems (including one capable of accommodating 75 cm diameter cores for testing at the meso-scale) and a computational simulation model to investigate biomineralization under both radial and axial flow conditions and at temperatures and pressures which permit CO_2 to exist in the supercritical state. The laboratory experiments have focused on understanding processes and developing injection strategies toward moving to the field scale. MICP-related research has been directed by MSU's ERI and performed in the laboratories of the Center for Biofilm Engineering (CBE).

2.1. Bench-scale sandstone core experiments

Previous research has focused on using MICP to reduce permeability in 2.54 cm (1 inch) diameter sandstone cores [25-27]. The Hassler-type core holder system shown in Figure 1 has been used to investigate MICP (and subsequent permeability reduction) under pressures up to 75.8 bar (1100 psi) in Berea sandstone cores, by injecting the ureolytic bacterium *S. pasteurii*, promoting biofilm growth and alternating pulsed injections of growth medium and mineralization (containing calcium and urea) solution with periods of batch reaction times in between injections.

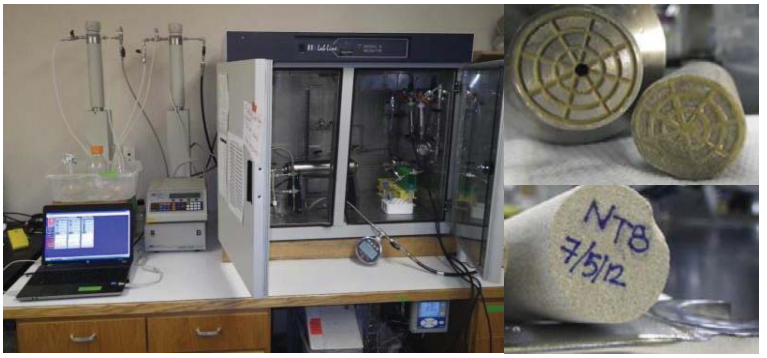

Figure 1. High pressure test system with Hassler sleeve core holder houses 2.54 cm (1 inch) diameter samples up to 15.24 cm (6 inches) long at pressures to 136 bar (2000 psi) (left). After MICP treatment, significant precipitation was observed on the core inlet (not shown) and outlet (right top) as compared to an unmineralized core (right bottom).

An example of Berea sandstone permeability reduction due to MICP is shown in Figure 2 [27]. This experiment demonstrates that the MICP process was unaffected when the system was pressurized to 75.8 bar (1100 psi).

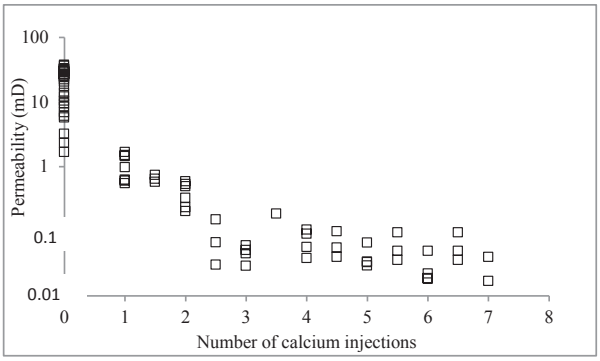

Figure 2. Permeability reduction in Berea Sandstone 2.54 cm diameter core over time after injection of *S. pasteurii* and with alternating pulsed injections of calcium- and urea- containing solution and growth medium. The initial permeability was approximately 40 mD and at the end was measured as 0.02 mD, showing a greater than three order of magnitude reduction. The core was tested at a pressure of 75.8 bar (1100 psi).

Pore throat size distributions in the Berea core material (before and after biomineralization) were examined using mercury intrusion porosimetry [28] to determine the pore sizes most impacted by MICP process. Results showed that most of the reduction in pore throat diameters via MICP occurred in pore throats with the size range of 6-16 μm, resulting in an increase in the number of pore throat diameters less than 1 μm [27]. Since the microbe itself is in the size range of 2-5 μm, the MICP process is only limited by the ability of the microbe to be transported into the pore throat or fracture. MICP may be able to access smaller pore spaces than cement-based technologies, because of the higher viscosity of cement/water mixtures (for example 5-7 cP for ultrafine cement slurries with high water to cement ratios), compared to aqueous solutions used in MICP treatment (1.0-1.3 cP) [26, 29].

2.2. Meso-Scale Laboratory Experiments

MICP research has also been performed under high pressure in a fractured 74 cm diameter Boyles sandstone sample [26, 30]. In the experiment, MICP was promoted in the fractured sandstone under 45 bar confining pressure. The experiment was performed in a novel high pressure test vessel designed to house porous media samples up to 76 cm in diameter and 50 cm tall[25, 30]. The purpose of this experiment was to observe whether MICP treatment could seal fractures under high pressure and radial flow conditions with the presence of a confining fluid (brine) similar to what might be faced in a field application. An additional objective of the experiment was to assess MICP treatment using a more economic urea source (urea fertilizer) in the growth and mineralization media. Over the course of the pulsed injection strategy experiment, the permeability was reduced and the fracture strength was increased. In addition, ureolysis was detected indicating the use of fertilizer was not inhibitory to the MICP process. It was noted

however that plugging of the fracture was delayed as compared to previous MICP fracture sealing experiments under ambient conditions [26, 30]. Although the reason for delayed sealing could not be determined (overburden fluid dilution of the reactants or some negative impact of the pressure conditions on *the bacteria*) the lesson allowed researchers to be prepared for the field by ordering excess chemicals to have on hand during the field experiment. In addition to the laboratory work in preparation for the field, collaborators at the University of Stuttgart have developed a reactive transport model [31], that has been integrated and calibrated with previous laboratory experimental data. This model was used to help design the field test by simulating the proposed injection strategy to predict the permeability reduction by MICP prior to field deployment.

2.3. Economics of Chemicals for Field Application

Additional experiments were performed to ensure economically viable field-grade chemicals instead of expensive lab-grade chemicals could promote MICP in the field environment. For three chemical components, economic alternatives were evaluated in MICP screening tests to narrow down field-ready solutions that were economically favorable over laboratory-grade chemicals and still promoted growth and MICP. The base medium that has been studied frequently in the laboratory is comprised of 3 g/L nutrient broth, 10 g/L ammonium chloride (NH_4Cl), 20 g/L urea ($CO(NH_2)_2$) and 49 g/L calcium chloride dihydrate ($CaCl_2$-$2H_2O$) (Table 1, Medium 1). To improve the economic feasibility, three components of the medium were tested, the urea source, the calcium source, and the carbon/nutrient source. The alternate chemicals assessed were:

- urea fertilizer (Potash brand) (Medium 3 and 4),
- $CaCl_2$ ice melting products (Extreme Ice Melt or Peladow Premier Snow and Ice Melter) (Medium 3), and
- either yeast extract (Acros) and molasses (Grandma's) (Medium 3) or a less expensive laboratory-grade nutrient broth (Research Products International (RPI)) (Medium 4).

In addition, since the formation fluids in the Gorgas well (the test site for the field experiment described in section 2.4) were sampled and contained 24 g/L total dissolved solids (TDS), all of the solutions evaluated in this experiment were amended with 24 g/L NaCl (Morton) in anticipation of field injection to match injected fluid TDS to formation TDS. To assess the precipitation kinetics or growth, 100mL of the various media were inoculated with *S. pasteurii* grown from frozen stock. The cultures were put on a rotating shaker for 1 hour during the precipitation kinetics experiment and 24 hours during the growth experiment and sampled over time to determine extent of ureolysis, calcium precipitation, and growth. A portion of the sample was filtered and used to measure the urea concentration with a modified Jung assay and calcium concentration with ion chromatography as previously described [26, 27, 32]. The unfiltered portion of the samples was used to measure the change in the bacterial cell concentration as a change in the optical density at 600 nm (OD_{600}).

The calcium precipitation experiments suggested that ice melt and urea fertilizer were suitable replacements for the lab grade sources previously used in MICP (Figure 3). $CaCO_3$ precipitated and the dissolved calcium concentration was reduced from 0.33 M to 0.25 M in all samples with different calcium sources over the course of one hour. It was determined that either the Extreme Ice Melt or the Peladow $CaCl_2$ ice melting products would be a suitable and economically favorable alternative to the laboratory-grade $CaCl_2$. Although the data is not shown, urea hydrolysis was not impacted by the less expensive sources of calcium or the urea fertilizer.

The results from the growth experiments suggested that *S. pasteurii* grew to a similar cell density using 1g/L yeast extract with 3g/L molasses or the less expensive nutrient broth (RPI) as nutrient sources in comparison to laboratory-grade nutrient sources (Table 1). The OD_{600} increased indicating bacterial growth in all of the different media, although the highest growth over 24 h was achieved in the brain heart infusion broth based medium #2 (Figure 4). However, the high cost of BHI was prohibitive to its use in larger scale experiments. Additionally, it was determined that sources of NH_4Cl that contained anti-caking agents were inhibitory to the growth of *S. pasteurii* (data not shown), so care was taken to ensure the NH_4Cl used in the field experiment did not contain an anti-caking agent.

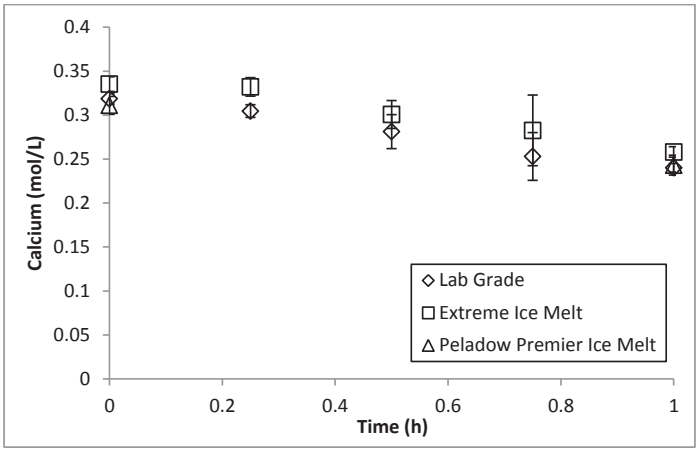

Figure 3. The calcium concentration in the bulk fluid decreased over time due to precipitation of calcium carbonate associated with MICP with both Extreme Ice Melt and Peladow calcium sources in two different solutions containing urea fertilizer. The error bars indicate the standard deviation of triplicate experiments.

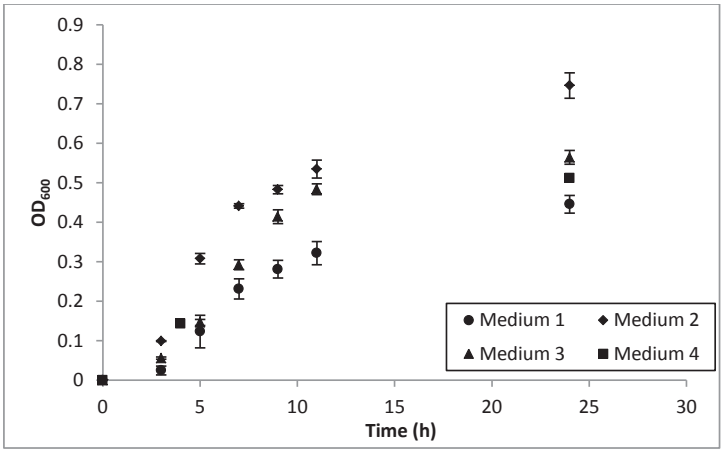

Figure 4: Growth of *S. pasteurii* over 24 hours on multiple nutrient sources with the most increase seen with 37g/L BHI (Medium 2). The growth of *S. pasteurii* over time was not hindered using alternate chemical sources. Each curve represents the different Medium listed in Table 1 and the error bars represent the standard deviation associated with triplicate experiments.

Table 1. Components of the different chemical combinations tested for the field experiment.

Components	Medium 1	Medium 2	Medium 3		Medium 4
Nutrient Sources	Nutrient Broth (Difco)	Brain Heart Infusion (Difco)	Yeast Extract and Molasses		Nutrient Broth (RPI)
Urea	Fisher Scientific	Fisher Scientific	Potash Urea Fertilizer		Potash Urea Fertilizer
Calcium	BDH		Extreme Ice Melt	Peladow Ice Melt	Peladow Ice Melt
Price ($/L) (Base Medium)	$2.34	$9.88	$0.08	$0.08	$0.28
With Calcium	$3.35		$0.13	$0.15	$0.35

The final chemical sources used in the field experiment described below were the Peladow ice melting product (Occidental Chemical Corp.), ammonium chloride without anti-caking agent (BASF), urea fertilizer (Potash), nutrient broth (RPI), and NaCl (Mix-N-Fine, Cargill). While both the yeast extract with molasses and the RPI nutrient broth provided economically beneficial sources of carbon for the bacteria, the RPI broth was used because it was similar in composition to the nutrient broth that was tested

extensively in the laboratory. By testing alternate chemical sources for the ability to achieve successful MICP with those chemicals, the price of the base field test solutions were significantly reduced ($0.28 per liter) as compared to the standard solutions used in the laboratory to promote MICP ($2.34 per liter).

2.4. Field Fracture Sealing Experiment

In April 2014, a field experiment was performed using MICP to seal a hydraulically fractured Fayette Sandstone formation 341 m (1118 feet) below ground surface) in the Gorgas #1 well at the Southern Company Gorgas Power Plant in Walker County, Alabama. The zone of interest was first perforated and then isolated with a double packer system attached to a tubing string (Figure 5). The formation was hydraulically fractured and the pressure data suggested that a horizontal pancake-like fracture formed. *S. pasteurii* cultures as well as urea, calcium, and growth-media solutions were delivered to the fracture region by an injection bailer with periods of batch reaction time in between the deliveries. Calcium/urea solutions (24 total pulses) containing urea fertilizer, ice melt calcium chloride, and NH_4Cl without anti-caking agent and microbial suspensions (6 total) were delivered to the fracture over the course of four days. The injection of these MICP promoting solutions resulted in fracture plugging and reduced instant shut-in pressure decay suggesting improved wellbore integrity after MICP treatment (data not shown). Analysis of the data is ongoing.

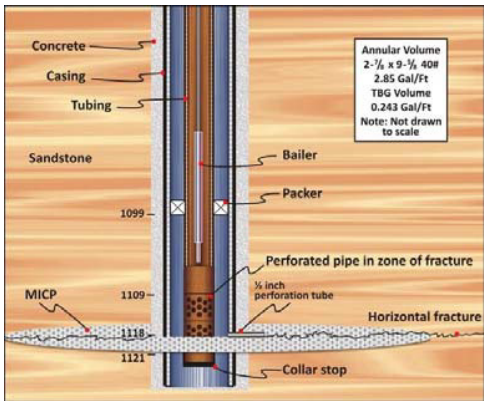

Figure 5. Schematic showing the down-hole elevations of key components at the Gorgas #1 well. The packer was set to isolate the perforated zone and the formation was hydraulically fractured at ~341 m (1118 feet) below ground surface (bgs). Perforated pipe was used below the packer so that when the bailer opened and delivered the contents the concentrated inoculum and growth or calcium containing solutions would be delivered at the elevation of the fracture. The concentrated solutions were then diluted and pushed into the fracture by brine pumped down the tubing string to promote MICP conditions in the fracture.

3. **Conclusions**

To prepare for a field experiment, MICP investigations at various spatial scales and pressures were performed to:

- Determine the impact of high pressure to permeability reduction in 2.54 cm diameter Berea cores and determine the aperture of fracture best impacted by MICP treatment,
- Assess fracture sealing under high pressure and confining fluid conditions in a fractured 74 cm Boyles sandstone core to set realistic field conditions,
- Verify that the use of the field-grade chemicals such as urea fertilizer and calcium ice melting products did not inhibit MICP

High pressure conditions were maintained in the Hassler-type core holder system while MICP by *S. pasteurii* was promoted. The result was the greater than 3 orders of magnitude reduction in permeability in a 2.54 cm diameter Berea sandstone core. The experiment provided information that MICP processes occurred under high pressure conditions and reduced the number of pore throat diameters in the range from 6 -16 μm and increased the number of pore throats less than 1 μm in diameter. The MICP process was also determined to provide fracture sealing in meso-scale fractured sandstone under subsurface relevant pressures and confining fluid conditions. The meso-scale experiment, while successful pointed the need to prepare for the field by assuring adequate supplies were on hand in case concentrations needed to be adjusted or injection strategies altered to achieve fracture sealing. In batch experiments, field-grade chemicals were assessed to enhance the economic viability of MICP fracture sealing technology on a larger scale. It was determined that urea fertilizer and calcium chloride ice melting products would achieve precipitation yet ammonium chloride amended with anti-caking agent was inhibitory to the microbial growth. As the research progressed from the laboratory to

the field, care was taken to order excess field-grade chemicals and avoid ammonium chloride with anti-caking agents. The field experiment resulted in meeting the goals of reducing permeability and improving wellbore integrity in 4 days. These results indicate MICP may become a viable option for wellbore leakage mitigation.

4. **Future Work**

Additional experimentation focuses on continuing to develop economically viable and commercially available MICP sealing technology. This work continues to assess the practicality of different chemical mixtures to promote MICP and develop injection strategies that improve MICP field-scale success. While a base assessment has been completed [33], work that determines the impact of the exposure of MICP minerals to supercritical CO_2 is also ongoing. A combination of modeling and experimentation will improve the MICP sealing technology in the subsurface to successfully reduce permeability and seal fractures to improve the storage security of geologically- stored carbon dioxide.

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