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Design of a meso-scale high pressure vessel for the laboratory examination of biogeochemical subsurface processes

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ABSTRACT

A meso-scale high-pressure vessel for testing subsurface relevant processes under simulated *in situ* pressures was designed and constructed. This system is capable of providing pressures up to 96 bar and capable of housing porous media samples such as rock cores up to 74 cm in diameter and up to 50 cm high. A valved switchboard allows for fluids to be pumped into and extracted from the vessel and to be sampled in a spatially resolved manner. The switchboard assembly also allows for the monitoring of fluid chemistry in real time, such as pH and conductivity of either the injected or effluent fluids. The vessel can be equipped with an optional heating jacket to control temperatures. The system can be used to investigate a wide range of subsurface relevant processes, including those related to a variety of petroleum industry interests such as fracture sealing for improving the security of geologic carbon sequestration or enhancing wellbore integrity.

As an example, this paper describes the use of the vessel to study ureolysis-driven calcium carbonate precipitation to reduce the permeability of a hydraulically fractured core under relevant subsurface pressure (45 bar). The core was inoculated with *Sporosarcina pasteurii* and biofilm growth was promoted in the fracture, followed by injection of calcium- and urea-containing growth reagents to promote saturation conditions favorable for ureolysis-driven $CaCO_3$ precipitation. This process is referred to herein as microbially-induced calcium carbonate precipitation (MICP). MICP treatment reduced the permeability in the mineralized fracture more than two orders of magnitude. This single high pressure experiment suggests that MICP can be used to reduce permeability in fractures under relevant subsurface conditions. This study also suggests that the high pressure vessel is suitable for testing a range of biogeochemical processes in meso-scale fractured porous media samples under pressure. The high pressure test system could also be well suited for studying microbially-enhanced methane production from coal, wellbore and cement integrity challenges with corrosive fluids, proppant and hydraulic fracturing fluid investigations, enhanced oil recovery, microbially-induced corrosion, or biofouling among many other industry-related biogeochemical processes.

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1. Introduction

1.1. Meso-scale investigation of biogeochemical processes

Laboratory studies of geochemical and biogeochemical processes are often limited to the small core scale and may not incorporate reasonable three dimensional and geologic heterogeneity (Yale et al., 2010b). Field tests are expensive, laborious and often field opportunities are limited. Therefore, to prepare for field scale experiments or technology deployment it is important to study *in situ* processes at intermediate scales. Intermediate or

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meso-scale experiments (defined here as ~1/2 m to 1 m scale) can more closely simulate real-world environments by incorporating larger scale phenomena that are not captured with typical lab scale experiments (McCallum et al., 2007) and may provide knowledge valuable in the transition to the field scale (DeJong et al., 2010).

Aside from commercially available core analysis equipment, such as those available from Core Laboratories (Texas, USA), high pressure vessels have been designed and constructed for the study of methane hydrates (Eaton et al., 2007; McCallum et al., 2007; Fitzgerald et al., 2012), sampling and analysis of deep sea microbiology (Bianchi et al., 1999) and a large-scale high pressure vessel for purposes of petroleum industry-related studies (Yale et al., 2010a, 2010b). Additional high pressure research equipment is necessary to advance the understanding of biogeochemical processes such as (1) how microbes and microbial activity is affected by pressure and temperature conditions in the subsurface (Abe et al., 1999; Bartlett, 2002; Spilimbergo et al., 2002; Martin et al., 2013), (2) how chemical reactions are enhanced or inhibited by high pressure and (3) how pressure may impact porous media characteristics such as porosity and permeability (Fatt, 1953; Ali et al., 1987).

To contribute to the equipment available, a meso-scale high-pressure vessel (described herein) was constructed and used to study biogeochemical processes in porous media samples under relevant subsurface pressure conditions. The pressure vessel described in this manuscript can be most closely compared to the Yale et al. (2010a, 2010b) LARGE system. The vessel described here has a lower maximum pressure rating (96 bar compared to 144 bar) and sample size capacity (74 cm diameter compared to 210 cm) than the LARGE system. This vessel is similar to the LARGE system as it allows for the study of porous media samples under radial flow and three dimensional conditions.

The specific design of the vessel was driven by tight cost and laboratory height constraints, while meeting the experimental requirements. The vessel alone was fabricated for under $70,000. Overall constructed system cost including switchboard, fittings, pumps, instrumentation and other accessories was approximately $100,000. The system is equipped with high pressure capable pH and conductivity monitoring instrumentation which makes the vessel well suited for studying biogeochemical processes at elevated pressures. Since the vessel is housed in a university environment it is accessible for collaborations. The vessel was shown in this manuscript to be successfully employed to investigate the use of microbially-induced calcite precipitation (MICP) to seal a fracture in a 29 in (74 cm) diameter sandstone core.

1.2. Microbially-induced calcium carbonate precipitation (MICP)

Microbially-induced calcium carbonate precipitation (MICP), particularly ureolysis-driven MICP, has been studied extensively for a wide range of engineering applications (Phillips et al., 2013a) including enhanced oil recovery (Ferris et al., 1996), improving construction materials (De Muynck et al., 2010; Achal et al., 2011; Dhami et al., 2012), consolidating porous media (Whiffin et al., 2007; DeJong et al., 2011; Stabnikov et al., 2011; Tobler et al., 2012), remediating environmental contaminants (Mitchell and Ferris, 2005; Mitchell and Ferris, 2006; Fujita et al., 2008; Okwadha and Li, 2011; Achal et al., 2012; Lauchnor et al., 2013) and enhancing the storage security of geologically sequestered CO_2 (Dupraz et al., 2009; Mitchell et al., 2010; Mitchell et al., 2013; Phillips et al., 2013b). Ureolysis-driven MICP involves microbes, particularly in an attached form, also known as biofilm, to promote the precipitation of calcium carbonate. The microbes produce the enzyme urease which catalyzes the hydrolysis of urea to form carbonate and ammonium. In the presence of calcium, the hydrolysis of urea can create saturation conditions favorable for the precipitation of calcium carbonate (Stocks-Fischer et al., 1999; Hammes and Verstraete, 2002; Ferris et al., 2003).

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

$$Ca^{2+} + CO_3^{2-} \leftrightarrow CaCO_3\ (s) \quad (2)$$

Ureolysis-driven MICP was used previously to reduce permeability in a hydraulically fractured Boyles Sandstone core at ambient pressure (Phillips et al., 2013b). It was also shown that ureolysis-driven MICP can occur at elevated pressures such as those encountered at geologic CO_2 sequestration or hydraulic fracturing sites (Cunningham et al., 2013; Mitchell et al., 2013). At the time of this study, MICP treatment of porous media and fractured rock was deployed in several field scale experiments (Fujita et al., 2008; van Paassen et al., 2010; Burbank et al., 2011; Cuthbert et al., 2013). The vessel described in this paper could be useful to researchers seeking to add the dimensions of increased scale, temperature and pressure to their experiments prior to field deployment. Meso-scale experiments performed under relevant subsurface conditions and on a near-wellbore scale allow for the testing of injection strategies, monitoring of the population of microbes, and gathering of data to assist in the transition from the laboratory scale to the field-scale.

1.3. Motivation for investigation

The purpose of this paper is to describe a new high pressure test vessel capable of the examination of biogeochemical processes at the meso-scale under subsurface relevant pressures. Another motivation of the research presented in this paper was to assess the application of ureolytic biomineralization under meso-scale and relevant subsurface pressure and temperature in a laboratory experiment to prepare for a planned field scale experiment. One question related to field relevance which motivated the conditions of the laboratory experiment was whether MCIP processes change significantly at pressures related to field deployment. A field test was planned to use MICP in a fractured formation at the depth of approximately 1120 ft accessed through a well drilled through the Fayette Sandstone formation located at the William Crawford Gorgas Electric Generating Plant near Parrish, Alabama, USA. To illustrate the vessel capabilities and prepare for the field deployment, an experiment was carried out under elevated pressure conditions to study the permeability reduction in a hydraulically fractured sandstone core due to ureolysis-induced calcium carbonate precipitation.

2. Materials and methods

2.1. Vessel design and construction

The pressure vessel was designed to inject and extract pressurized aqueous solutions, supercritical carbon-dioxide, or other fluids including gases into meso-scale porous media samples (such as rock cores) of up to 74 cm diameter and 50 cm height under reservoir relevant pressure and temperature conditions. In order to keep the vessel fabrication cost and weight low the vessel was specified for a maximum allowable working pressure (MAWP) of 96 bar at 43 °C. Since the super-critical point for carbon-dioxide resides at 74 bar and 31.5 °C, this means a differential injection pressure of up to 22 bar over the CO_2 critical pressure can be safely applied. The vessel was designed, fabricated and tested according to ASME standards by Alaskan Copper Works in Seattle, Washington.