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The Development of a Leak Remediation Technology for Potential Non-Wellbore Related Leaks from CO_2 Storage Sites.

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Abstract

In order to reduce global atmospheric greenhouse gas emissions, many pilot-scale and commercial-scale Carbon Capture and Storage (CCS) projects are under development or are operating commercially. There are two main recognized potential leakage mechanisms which may allow CO_2 to leak out of the intended storage complex, migrate into overlying aquifers and eventually seep back to the near-surface and atmosphere, with potentially negative impacts upon natural resources and/or the environment. The primary potential leakage pathway is via wellbores, which may provide a direct connection between the storage formation and the surface. Should a well integrity related leak occur, established remediation techniques may be employed to mitigate and/or remediate further leakage. A second potential leakage pathway includes fluid migration through geological faults, fractures and high permeability zones within a caprock. Not only is it more difficult to constrain and characterize these leaks, there is currently no routine method available to intercept and repair solution leakage via such pathways. This experimental study was conducted to assess the injection of chemical solutions capable of physically and chemically interacting with a CO_2-containing brine to form a geochemically stable blocking agent capable of preventing further fluid leakage. A number of potential blocking agents were evaluated, and experiments were carried out under quasi formation conditions (*i.e.* elevated pressure and temperature) using a combination of simulated 2D caprock micromodels and 3D geological porous medium core floods. Experiments succeeded in determining the behaviour of the blocking agent, CO_2 saturated brine behaviour, reaction front location, the concentration and amount of blocking agent required and an indicative timescale for remediation. Using experimental parameters as bounding conditions, numerical simulations using PetraSim (TOUGH2 and TOUGHReact) were used to assess the upscaling requirements of the blocking process in preparation for large-scale laboratory tests and a field demonstration.

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1. Introduction

The large-scale geological storage of CO_2 in deep subsurface formations is being investigated and developed in order to reduce the global impact of greenhouse gas emissions to the atmosphere. Once injected, CO_2 is expected to remain within the geological storage complex for long periods (*i.e.* thousands of years). However, there are two main potential leakage mechanisms which may allow CO_2 rich fluids to leak from the intended storage formation, migrate into overlying aquifers and eventually seep back to the biosphere and atmosphere; potentially negating the financial investment and benefits of injection and sequestration [1]. The primary potential leakage route is via wellbore leakage which forms a direct physical connection between the storage formation and the surface [2]. However, the experience of oil and gas operators and service contractors with wellbore processes and workover practices, along with the more accessible wellbore infrastructure, allow the resolution of many engineering problems related to well integrity [3, 4]. A second potential CO_2 leakage pathway consists of cross-formational geological leakage pathways including geological planar structures (*e.g.* faults, joints and/or fracture zones) as well as caprocks which contain variable hydraulic conductivity (*e.g.* facies changes or sand channels within a shale caprock) (Figure 1).
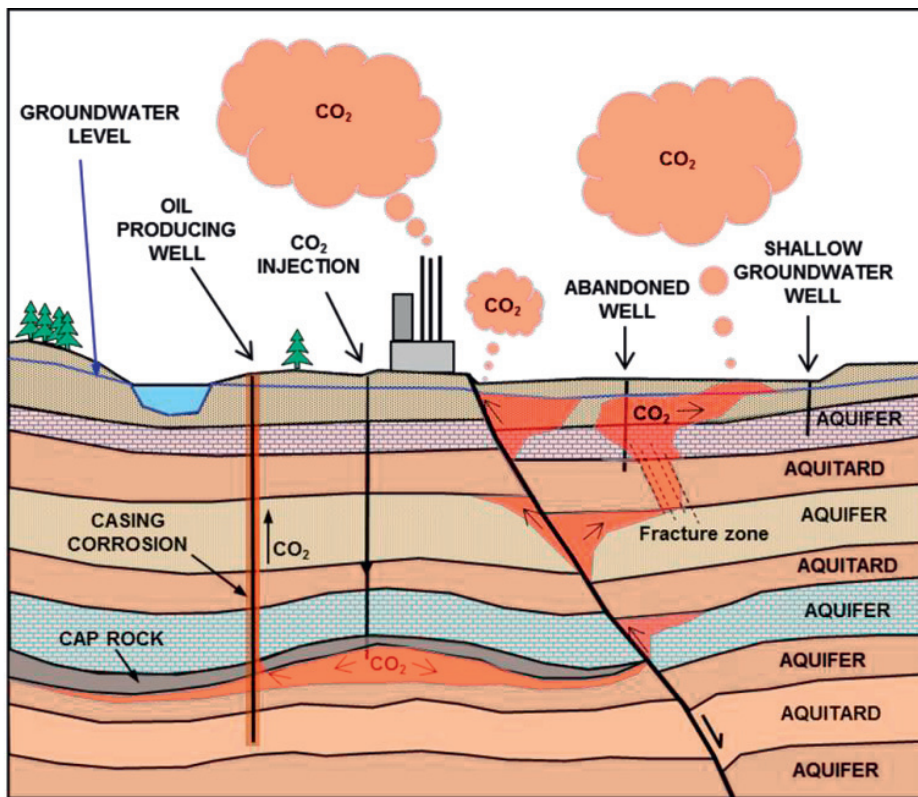

Figure 1. Schematic figure showing potential leakage pathways from a geological CO_2 storage site (Modified from IPPC, [5]).

This study focused upon the non-wellbore leakage scenario and evaluated the potential for injecting a chemically reactive chemical solution into the leakage zone to react with CO_2-bearing solutions, form a solid precipitate and effectively seal the fluid leakage pathway for long periods of time.

1.1. CO$_2$ Leak Remediation Approaches

In the event that supercritical CO_2 and/or CO_2-bearing brine leakage is detected outside of the intended geological storage complex, there are several general options suggested to mitigate (*i.e.* reduction and minimization of the short term effects of leakage) and/or remediate (*i.e.* long term engineered solution to prevent further leakage or impacts) further fluid leakage. A review of engineering options for the remediation of leakage from CO_2 storage reservoirs is provided by Kuuskraa and Godec [6]. Leak mitigation approaches typically involve proposed short-term engineering interventions to reduce and/or stabilize the flow of CO_2-rich solutions from the storage formation. These include: the manipulation of formation fluid pressure [7], physical extraction of CO_2-rich fluids, the creation of *in situ* hydraulic barriers via intentional water injection [8] and the intentional dilution of leaked fluids [9]. Remediation approaches may involve the physical blocking of the fluid leakage pathway via a number of physico-chemical processes [10, 11, 12] and/or microbiological treatments [13, 14, 15]. These latter options are intended to remediate fluid leakage by the permanent closure and sealing of fluid flow pathways. Of these approaches, remediation options are considered to be the most beneficial because, ideally, no further engineering intervention would be required. Aside from leak mitigation and remediation, there are a number of existing technical approaches designed to control fluid flow behaviour within geological formations. These approaches are typically applied in the field of Enhanced Oil Recovery (EOR), where chemical floods and foams are implemented *in situ* in order to enhance fluid flow rates, or to minimize unwanted fluid migration which may reduce the efficiency of hydrocarbon recovery [16]. By extension, some of these methods may be adopted (and adapted) to prevent and/or reduce the leakage of fluids, in this case supercritical CO_2 and/or CO_2-rich brine.

In order to effectively seal a supercritical CO_2 and/or CO_2-rich brine leakage pathway using a chemical solution injection, the chemical blocking agent may act in one of the following ways; (i) chemically react directly with supercritical CO_2 and/or dissolved CO_2 species to form a pore-blocking precipitate, (ii) chemically react in response to increased salinity to form a precipitate, and (iii) cause precipitation as a result of chemical instability under the expected low solution pH conditions within a CO_2-rich environment, or (iv) provide a source of nutrients to encourage microbial biofilm development and associated bioclogging and/or mineral precipitation. Of particular interest in this study were simulations which use amorphous silica and sodium silicate as injection solutions to block pore space and to solidify unconsolidated sediments. The application and numerical simulation of this method has been reported by Hamderi [17]. A summary of approaches and the blocking agents considered is provided in Table 1.

In assessing the important attributes of candidate blocking agent solutions, it is important to consider not only the effectiveness of the chemical as a blocking agent, but also aspects of implementation, upscaling, cost of materials and the required duration of fluid injection. The ideal candidate solution would be inexpensive, easy to obtain, stable enough to store and transport, easy and safe to handle, react rapidly in the subsurface to form an insoluble (or sparingly soluble) precipitate, form a hydraulic barrier resistant to CO_2 and mildly acidic solutions (*i.e.* carbonic acid) and produce a barrier which will last on a time scale of centuries to millenia. Of the chemicals, solutions and approaches summarized in Table 1, and following preliminary batch tests, the use of sodium silicate was considered to have the greatest potential to satisfy the blocking agent criteria, and so was used as the blocking agent in 2D visualization and subsequent core floods.

Table 1. Summary of the methods and mechanisms evaluated in assessing the most useful chemical blocking agents for experimental simulations.

Method and chemical compound		Relative Cost**	Feasibility of blocking agent formation	CO_2 targeted application	Chemical stability under reservoir conditions	Permanence of precipitated agent	Relative probability of success
Solid precipitate	Calcium Carbonate	Low	High	Yes, reacts with CO_2	Yes	Very likely	High
	Magnesium carbonate	Low	High	Yes, reacts with CO_2	Yes	Very likely	High
	Antisolvent	High	Low	Yes, reacts with CO_2	Not known	Not known	Uncertain
Gel formation	Time dependent gelling polymer	High	High	No, time controlled	Yes	May be degraded	Moderate
	pH dependent gelling polymer	High	High	Reservoir conditions may lead to premature gelling	Yes	May be degraded or reversible	Moderate
	Viscosifier	High	Moderate	Yes, reacts with CO_2 but mixing may be an issue	Yes	Not known	Low
	Sodium Silica/ Colloidal silica	Medium	High	Reservoir conditions may lead to premature gelling	Yes	May be degraded or reversible	Moderate
Microbial Biofilm		Wide range*	Moderate	May target CO_2 location	Yes	Degrades	Low

2. Materials and Methods

All experimental and numerical simulations were carried out using the same bounding conditions for CO_2-rich brine chemical composition, fluid pressure, fluid temperature, range of fluid flow rate and simulation duration. Visualization experiments were conducted using the techniques and methods developed by Javadpour and Fisher [18]. The purpose of the laboratory testing was to investigate the effectiveness of precipitated sodium silicate as a blocking agent within small- and medium-scale porous media under reservoir conditions, and to establish bounding conditions for the numerical simulations.

2.1. Geological Porous Media, Solutions and Equipment

Materials and equipment included simulated formation water, sodium silicate solution, a two dimensional micromodel visualization cell and core flood apparatus. The chemical composition of simulated formation water was sodium chloride (8.1%), potassium chloride (0.1%), calcium chloride (1.3%), magnesium chloride (1.0 %), sodium bicarbonate (0.007%). These chemicals were dissolved in reverse osmosis water (15 MΩ) and equilibrated at 850 p.s.i. and 40°C. This composition was used for all experiments and is referred to herein as "standard brine". Sodium silicate solution, acquired from National Silicates, consisted of a 3:2 ratio sodium silicate:water solution. Percentage composition of Na_2O and SiO_2 was analyzed at 8.9 % and 28.6 %, respectively. A customized

Micromodel was designed in-house at Alberta Innovates – Technology Futures (AITF) and constructed via hydrofluoric acid etching of borosilicate glass at the University of Calgary (Figure 2).
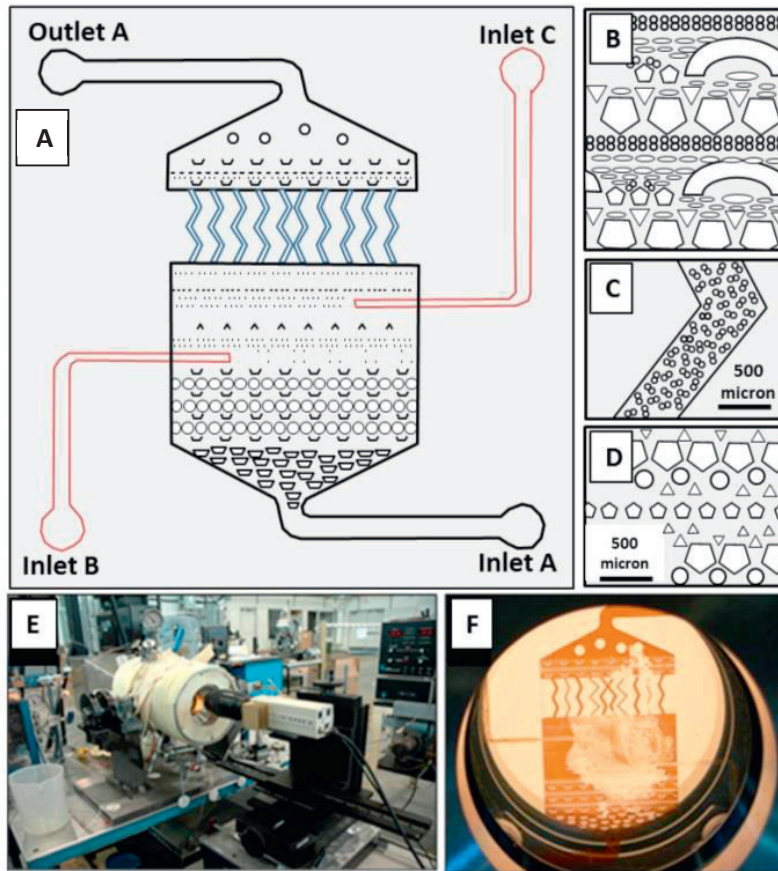

Figure 2. Schematic of the visualization cell used for simulated caprock leakage remediation experiments. (A) Inlet A allowed brine to be pumped through the model, and extracted from Outlet A (*i.e.* upward fluid flow direction). Inlets B and C were both used to inject blocking agent chemicals. (B-D) Enlarged views of the micromodel structure illustrating setup of the high pressure chamber, ancillary equipment and camera used to capture time series images of the experiment (E). (F) Real-time images of the micromodel flood, under reservoir conditions, with sodium silicate blocking agent co-injected into brine flowing through a simulated caprock fracture system.

Similar micromodels have been successfully used to study a wide range of pore-scale processes within the oil and gas industry including water floods, gas floods, surfactant and polymer floods, foam, gel-foam studies and in microbially enhanced oil recovery studies [19]. In this case, the micromodel was designed to simulate a caprock leakage scenario, with length scale typical of a 2D linear fracture zone through a caprock. The design was not intended to simulate single fracture flow, but rather to simulate a zone of increased hydraulic conductivity and permeability within a heterogeneous aquitard which included variations in porosity and some linear features. Core flood apparatus was constructed and commissioned as part of the current study. Berea Sandstone cores (660 mD and 1530 mD), were acquired and cut to 15 cm lengths to fit inside pre-existing 5 cm diameter Buta-N rubber core sleeves. Fluid injection pressure was maintained at 850 p.s.i. (5860 kPa), with a maximum working confining pressure of 1400 p.s.i (9652 kPa).

2.2. Laboratory Test Procedures

Preliminary benchtop chemical reactivity tests were carried out to assess the ratio of silicate solution to brine required to precipitate a solid material. Following a series of solution titration tests with brine and sodium silicate, it was empirically determined that a 0.44 % silicate solution provided sufficient reaction and precipitation to use for the 2D visualization experiments. This concentration equated to a 50:50 $NaSiO_3$:brine ratio. A similar range of sodium silicate concentrations were used in core floods. Lower concentrations of $NaSiO_3$ were used (*i.e.* 0.22 %) in order to assess the effect of lower silicate concentration upon precipitate formation behaviour. Reaction at 0.22 % still occurred, but took approximately 24 hours to fully precipitate.

2D visualization experiments were carried out in order to visually assess the flow of CO_2-rich brine via simulated leakage through geological storage caprock. The location of the chemical reaction front as a function of blocking agent composition, injection rate, CO_2-brine leakage rate and overall duration of injection were all assessed. The primary aim was to establish the amount of silicate material required to block the porous medium and the overall time required for that blockage to occur. A series of preliminary visualization experiments were carried out in order to commission the micromodel, to assess fluid flow through the simulated fracture zone and porous medium and to visualize the reaction and formation of a hydraulic barrier via two separate injection ports. Co-injection was also considered in order to enhance the blocking agent:brine ratio. A summary of the core flood apparatus is provided in Figure 3 which is a schematic of the overall system with ports and instrumentation. Constant monitoring the DP across the core, inlet/outlet flow-rates, and the DP across the injection ports was used to determine experimental conditions and to determine an experimental end-point. This system is effectively automated, with pressure and temperature logged automatically. Solution samples were taken manually and solutions prepared for subsequent physical and chemical analysis. The results of solution analysis are not presented here.

Brine and blocking agent solutions were prepared using reverse osmosis H_2O which had been boiled and purged with N_2 gas to ensure an O_2 / CO_2-free solution. The solution was then stored in a sealed glovebag under a N_2 atmosphere. One litre of brine solution was placed into a 2 litre pressure rated piston cylinder and saturated with commercial grade carbon dioxide (CO_2) at 850 psi and 21°C. The cylinder was rocked periodically over 2 days and pressure was maintained at 850 psi via a hydraulic pump. After saturation, the cylinder was placed upright and excess CO_2 was vented while maintaining constant pressure, leaving a CO_2 saturated brine, as confirmed by chemical analysis. Sodium silicate solutions were placed into separate 500 mL pressure-rated piston cylinders, pressurized to attain the desired experimental conditions, and stored at ambient room temperature (*i.e.* 21°C +/- 2°C).
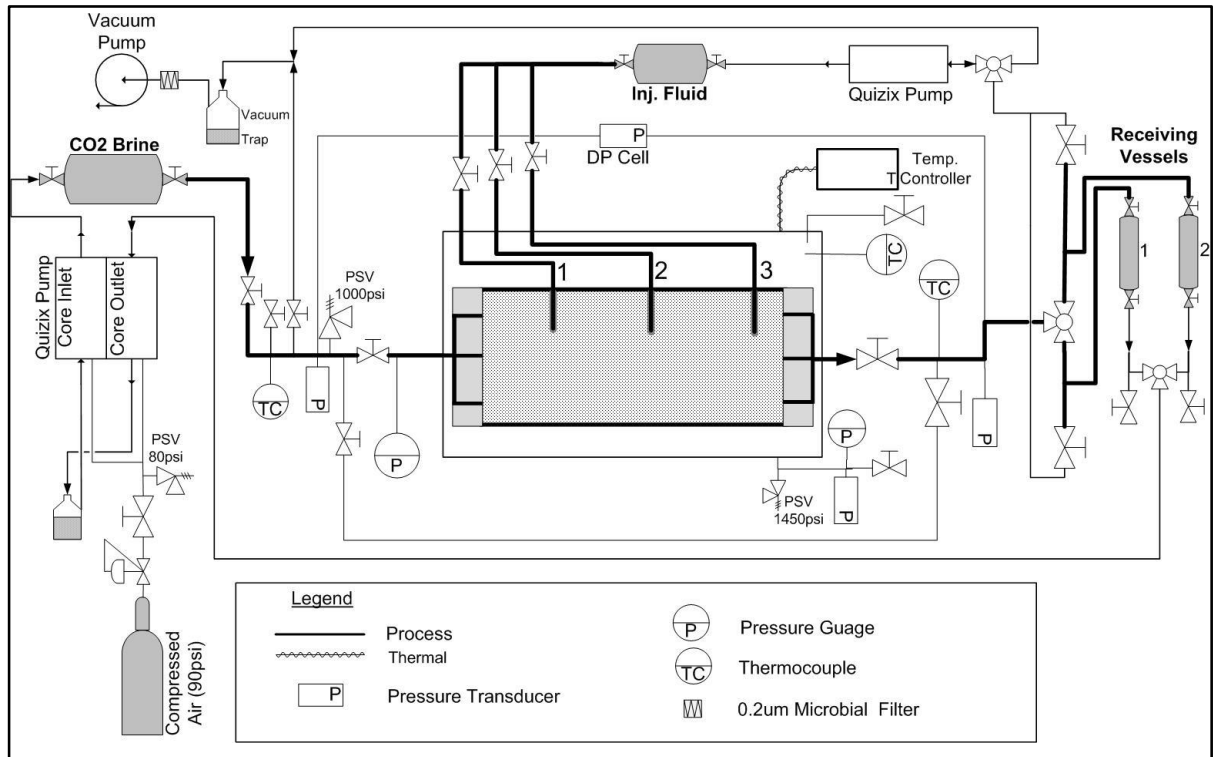
Figure 3. Schematic of the core flood experimental setup. The sandstone test core is contained within stainless steel core holder and the relative locations of the three blocking agent injection ports are shown as ports 1, 2 and 3.

Experimentation was performed at a fluid pressure of 850 p.s.i. and a temperature of 21 °C (*i.e.* ambient) with a bulk core fluid flow rate of 2.0 ml/minute. Pre-testing of the core was performed to optimize overall system configuration. Reverse Osmosis (RO) water, at room temperature (*i.e.* 21°C +/- 2°C) and 150 psi, was used to flush the system and measure core characteristics such as pore volume (PV) and permeability (k). To measure the pore-volume, the apparatus was pre-evacuated. Quizix pumps were used to fill the system prior to the inlet valve to 150 p.s.i. and the pumps stopped to re-zero the volumes used to quantify the PV. After the inlet valve was opened, pumps were restarted and water filled the core pore volume and also void volume associated to the core sleeve and tubing. The pumps filled the core holder until the fluid pressure reached 150 psi. At constant back-pressure, maintained by a receiving PA and varied flow-rates of 0.1, 1, 5, and 10 ml/min, the differential pressure (DP) was measured across the core to determine core permeability. After testing, the overall pressure was brought up to operating conditions and re-tested for leaks and fluid flow. After commissioning the system, brine was flushed through the system to remove all ultrapure water or other contaminants. Brine was flushed through the system at 10mL/min for a total volume equivalent of up to 4 pore volumes.

Injection fluid (*i.e.* sodium silicate solution diluted with brine) was injected fluid primarily to port 1 (closest to brine inlet, see Figure 3), then switched to the next port along the column when pore space blockage was observed (via an injection port pressure increase). Effluent generated from the core holder / injection fluid was collected under operational pressure and temperature conditions via two 80 mL stainless steel piston accumulators (PA). Each PA was set at a constant pressure setting (850 p.s.i.) and acted as a receiving vessel for the effluent. Brine and injection fluids were set to constant flow rates as required by the desired injection silicate:brine ratios. When a PA contained one pore volume of effluent, the experiment was paused momentarily and the full PA unit was removed and replaced with an empty PA, and the experiment was resumed. During stoppages, the brine and injection fluids were isolated from the core, and the effluent piston cylinder acted as a back-pressure regulator, to prevent any changes in the system and maintain conditions to allow for reaction to continue in a safe manner.

All effluent samples collected were depressurized, the liquid and gas components quantified, and the effluent analyzed for solution pH and electrical conductivity prior to sub-sampling for further chemical analysis. Analytical data were recorded and combined using Data Acquisition (DAQ) software data to define the experimental conditions at which the samples were taken.

Along with core effluent samples, brine and injection fluids were sampled and analyzed as procedural blanks. Electrical conductivity and pH were measured at the time of sampling to allow analytical data correction for the degassing of CO_2 from solution prior to chemical analysis. Visual inspection of the core post-experiment was carried out immediately to assess the injection ports and condition of the core. Cores were sent for CT Scans to non-invasively image the distribution of precipitate formation surrounding the ports and/or within the cores. CT scan data are not reported here.

2.3. TOUGHReact Simulation of Blocking Agents

The geochemical simulation code TOUGHReact [20] with the PetraSim graphical interface was used to simulate the extent of blocking agent reaction and the time required to form an effective blockage under reservoir conditions. Experimental core flood parameters were used as bounding conditions to model the effect of sodium silicate injection into a simulated sandstone reservoir. The initial fluid composition in the simulation was the same as used for the fluid which saturated the core flood, except in the injection ports. To avoid precipitation in, and the blockage of, the injection ports, each port was initially assumed to contain very dilute water. Once injection was initiated, this dilute fluid was displaced by sodium silicate solution. If an injection port was not in use for the simulation, then the fluid was assumed to be the same composition as the initial fluid which saturated the core. The fluid injection rates and compositions were set to the same values as core flood experimental conditions. A 24,000 cell orthogonal mesh was established, with x:y:z cell dimensions of 0.5 cm x 0.5 cm x 0.8 cm, respectively. The outer edges of the grid (i.e. corners) were set as non-reactive and non-flow, analogous to the cylindrical experimental core. Side-wall injection ports were simulated at 4.0 cm, 9.1 cm and 14.1 cm along the edges of the core from the primary brine inlet. Mesh density around the simulated injection ports was increased by a factor of 2 in order to replicate the injection port dimensions, and also to capture rapid chemical reactions close to the injection location(s). The grid blocks immediately around the injection port areas were assigned a permeability two orders of magnitude lower than the surrounding matrix. This minimized fluid flow through the sides of the injection port and simulated the effect of the experimental stainless steel injection tubes within the experimental cores.

3. Results

3.1. 2D Visualization Experiments

Visualization experiments carried out using the 2D Micromodel apparatus successfully demonstrated the precipitation of sodium silicate within the model, as well as some advantages and limitations of blocking agent co-injection and the placement of simulated injection wells within a geological storage reservoir. Precipitation occurred across the model, and fluid flow through the simulated caprock fracture zone resulted in the sealing of "fractures" via the direct precipitation of sodium silicate. Time series images of the process revealed the progressive blocking of simulated porous medium and fractures, with subsequent fluid flow around the blockage, and eventual sealing of the simulated leak zone. This result suggests that an *in situ* application of the blocking agent would be possible.

3.2. 3D Core Flood Experiments

Core floods proved to be successful in blocking the geological porous medium by sodium silicate injection. The reaction proved to be rapid, and in some instances blocked the injection port almost immediately. The most successful injection application resulted from the use of 50 % diluted sodium silicate solution.

Given a bulk fluid flow rate of 2.0 ml / min across the Micromodel, an injection rate of 1.0 ml / min of 0.44 wt % sodium silicate solution allowed the precipitation of sufficient sodium silicate to cause blockage and prevent further fluid flow across the model. This was quantified using pressure measurements taken across the core apparatus at various locations (Figure 4).
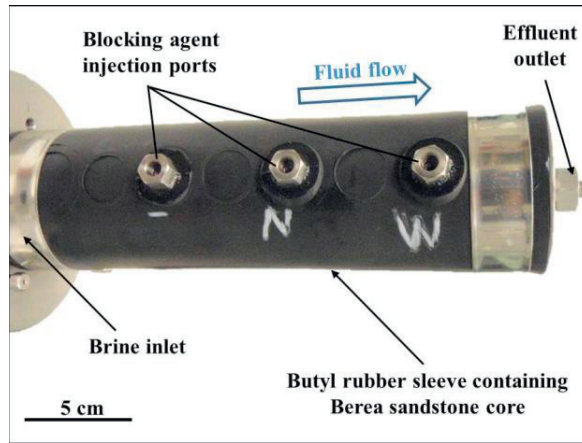

Figure 4. Photograph of the core holder with side mounted injection ports. This assemblage fits inside a larger stainless steel sleeve containing pressurized fluids.

3.3. TOUGHREACT Modelling Results

Fluid flow and chemical reactivity were successfully modelled using TOUGHREACT, and provided results comparable to the experimental core flood apparatus. Figure 5 shows typical results for a sodium silicate simulation contoured on an X-Z and a Y-Z plane through the centre of the core. The X-Z plane is a vertical section through the injection ports while the X-Y plane is perpendicular to it the orientation of the figures is shown in the upper right corner.
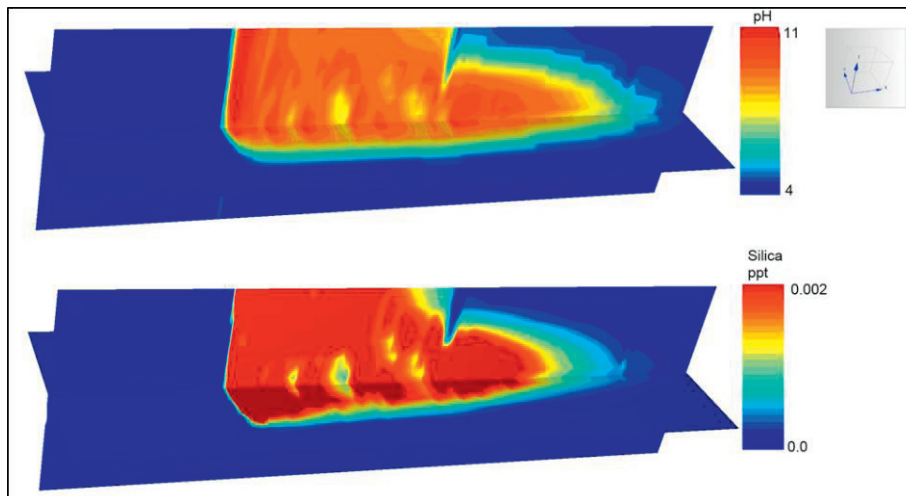

Figure 5. TOUGHREACT model output for Na-silicate injection at the first port along the simulated experimental column at time step of 1000 seconds (16.6 minutes). Silica precipitate (ppt) indicates precipitation up to 0.002 moles of silicate.

The experimental core flood used all injection ports sequentially. However, the simulation results shown in Figure 5 are for injection in the first port only (farthest left port in Figure 5). The top figure shows solution pH while the bottom shows the amount of precipitated silica. The contouring is stepped due to the grid size and is somewhat oscillatory due to both convergence issues and the gridding algorithm. Solution pH results are primarily due to the mixing of the acidic CO_2 flood (approximately pH 4.0) and the markedly alkaline sodium silicate solution (approximately pH 11). Thus there is a rapid change in pH at the interface between the two solutions. The rapid decrease in solution pH results in the precipitation of silica. Maximum predicted precipitation occurs slightly behind the neutralization front and is considered to be controlled by the flow rate and the nucleation / precipitation kinetics.

The physical effect of the injection ports on fluid flow can be seen in Figure 5. Injection port 1 can be identified in the X-Z plane of the pH graph as it contains the most alkaline fluids. No fluid mixing has occurred in this zone. Injection port 2 can be identified slightly to the right of centre in the X-Z plane for both graphs. Port 2 physically blocks flow, thus the fluids flow moves around it. As this port has not been used for this simulation, the port contains the acidic CO_2 flood fluid and appears as a blue narrow line. Injection port 3 is located at the front of the plume thus its effect on flow cannot be seen. The effects caused by the ports are not seen in the X-Y plane.

4. Discussion and Conclusions

The ability to numerically predict precipitation reactions at an experimental scale gives confidence that the experimental approach and numerical simulations are reasonable. The experiments undertaken used a single CO_2 saturated fluid (*i.e.* formation water) composition and using a series of Berea sandstone cores. Further work should involve the assessment of various fluid compositions, different core materials to confirm that the blocking agent is relatively independent of formation mineralogy. Furthermore, an evaluation of the current operational conformance methods by service companies should be evaluated in order to determine if they are compatible with this application.

2D experiments successfully demonstrated the timescale of precipitation (minutes), the distribution and behaviour of the chemical reaction front relative to the fluid injection ports and the optimum concentration of sodium silicate required for porous medium and fracture blockage. Three dimensional core floods confirmed the blocking agent injection rate was sufficient to allow precipitation and reaction to the point of blocking the porous medium. Numerical simulation of the core flood demonstrated that solution pH and fluid characteristics within the core, and similar distribution of precipitate within the model, provides a tool with which to plan larger scale experiments and field tests. It is concluded from the combination of 2D micromodel, 3D core flood experiments and numerical simulations that aqueous sodium silicate is a good candidate for injection as a blocking agent to remediate leakage from a geological CO_2 storage complex.

Acknowledgements

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