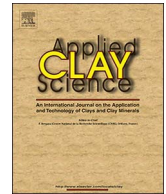




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Research paper

Influence of supercritical CO₂ on bentonite properties

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ABSTRACT

Wet bentonite was used to simulate a caprock for geologic CO₂ sequestration. Its properties were characterized after reacting with supercritical (SC) CO₂ alone and in the presence of salt solutions. High-pressure reaction tests were conducted to investigate the interaction of SC CO₂, bentonite, and water. Wet bentonite mixed with 1 N NaCl solution and then reacted with SC CO₂ resulted in noticeable changes in volume and bentonite mineral components compared with unreacted bentonite, due to SC CO₂ induced dehydration. Plagioclase, K-feldspar, montmorillonite, and pyrite were dissolved but Ca-bearing minerals, i.e., calcite, dolomite, and gypsum, and halite were precipitated. In contrast, wet bentonite mixed with the salt solution reacted with SC CO₂ in the presence of the salt solution resulted in less distinct changes in volume and mineral components, likely due to the dissolution of SC CO₂ in the salt solution. The fewer changes in mineral components in the bentonite reacted with SC CO₂ in the presence of the salt solution were attributed to the greater quantity of water, causing CO₂ dissolution. These results suggest implicitly that caprock in the immediate vicinity of an injection well may significantly deteriorate due to the interaction between caprock and dry SC CO₂, and caprock far from the injection well may deteriorate less because the SC CO₂ may react in the presence of large quantities of water.

1. Introduction

Geologic CO₂ sequestration (GCS) is an effective option for reducing CO₂ in the atmosphere. Among various reservoir types for storing CO₂, deep saline formations have the largest CO₂ storage capacity (Metz et al., 2005). For GCS, CO₂ is injected into a saline aquifer (i.e., permeable rock) with an impermeable caprock (e.g., shale) serving as a seal for permanently storing CO₂. During CO₂ storage, CO₂ can be dissolved and mineralized into stable form (e.g., carbonate minerals) within several thousand years (Lindeberg and Bergmo, 2003; Metz et al., 2005).

Natural hydrocarbon reservoirs are commonly sealed with caprocks having very low permeability, such as shales and evaporates. These rocks are considered potential caprocks for geological storage of sequestered CO₂. Shales are generally composed of fine-grained quartz, feldspar, carbonates, and clay minerals (e.g., illite, kaolinite, chlorite, and smectite) (Gueguen and Palciauskas, 1994). The mechanical and transport properties and sealing capacity of shales can be affected by the content of high swelling clays (Mitchell and Soga, 2005; Kaya and Fang, 2005; Espinoza and Santamarina, 2012).

As liquid CO₂ is injected into deep saline formations with overlying caprock, it becomes supercritical (SC) CO₂ in the reservoir. The SC CO₂ exhibits intermediate behavior between liquid and gas, and has a high

density, close to the liquid state, and lower viscosity, similar to the gas state (Lozowski, 2010). The interactions between SC CO₂, water, and caprock in the CO₂ storage area can potentially occur as three reaction systems: a SC CO₂/caprock reaction system in the immediate vicinity of the injection well; SC CO₂/water/caprock reaction system in the aquifer around the CO₂ injection well, where dissolved CO₂ can exist; and water/caprock reaction system far away from the CO₂ injection well.

The interactions between SC CO₂ and caprock, which are characterized by slow reaction rates, can affect the properties of the aluminosilicate minerals contained in the caprock. The interactions between SC CO₂ and potential caprock clay have been primarily studied using numerical modeling because of their very slow reaction rates and difficulties in experimentally simulating a deep geological environment (Botan et al., 2010; Romanov et al., 2010; Yang and Yang, 2010; Cygan et al., 2012; Rother et al., 2012; Krishnan et al., 2015; Leng and Rao, 2016). For example, Botan et al. (2010) reported that the CO₂ interlayer in Na-montmorillonite inhibits the diffusion of exchangeable cations. In addition, Yang and Yang (2010) conducted Gibbs Ensemble Monte Carlo simulations to investigate the swelling behavior and interlayer structure of Na-montmorillonite in SC CO₂ fluid. They showed that the interlayer spacing of Na-montmorillonite in the presence of SC CO₂ was larger than that of Na-montmorillonite in the absence of SC CO₂.

Many experimental studies have been conducted to investigate the

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interactions between SC CO₂ and potential caprock clay (Naveau et al., 2011; Shao et al., 2011; Espinoza and Santamarina, 2012; Giesting et al., 2012; Rother et al., 2012; Jong et al., 2014; Loring et al., 2014). These studies have suggested that the shrinkage or expansion of montmorillonite depends on the initial hydration state and relative humidity of SC CO₂. The types of cations in the montmorillonite interlayer also affect their swelling or shrinkage behavior. For example, Shao et al. (2011) conducted experimental tests to investigate the effect of salinity and water availability on the interactions between SC CO₂ and phlogopite, which is considered a potential clay mineral in caprock at GCS sites. Two reaction systems, SC CO₂/H₂O(liq)/phlogopite and SC CO₂/H₂O(g)/phlogopite, were evaluated in their study. They suggested that a thin water film adsorbed on the phlogopite surface formed dissolution pits and secondary mineral surface coatings that could change the caprock properties. In addition, Espinoza and Santamarina (2012) evaluated porosity changes and crack formation in Ca-montmorillonite in contact with 2 M NaCl solution and SC CO₂. They reported that the porosity of Ca-montmorillonite decreased more in SC CO₂ than in 2 M NaCl solution. Cracks formed in the Ca-montmorillonite in contact with SC CO₂ for 73.5 h due to changes in electrical and capillary forces in the Ca-montmorillonite particles caused by the SC CO₂.

However, experiments investigating the dehydration and shrinkage of clay materials in contact with SC CO₂ at various conditions (e.g., temperature, pressure, and water chemistry) have been rarely conducted. The objective of this study was to investigate the characteristics of bentonite when directly reacted with dry SC CO₂ and with SC CO₂ in the presence of salt solutions. Bentonite was used to simulate a caprock because bentonite is impermeable and primarily consists of clay minerals. Kinetic tests for SC CO₂, bentonite, and water interactions were conducted in a stainless steel autoclave reactor with two reaction systems, SC CO₂ and bentonite and SC CO₂, bentonite, and salt solutions, at 50 °C and 12 MPa to mimic the GCS environment. 1 N NaCl, 1 N CaCl₂, and 1 N MgCl₂ solutions and seawater were used to evaluate the effect of salts on bentonite properties in the presence of SC CO₂.

2. Materials and methods

2.1. Bentonite

The bentonite used in this study was obtained from a Geosynthetic Clay liner (GCL), which is sold commercially under the trade name Bentofix®. The mineralogy of the bentonite was analyzed using X-ray diffractometer (XRD) methods at Mineralogy-Inc., USA. The unreacted bentonite contained 74% montmorillonite, 12% plagioclase feldspar, 6% quartz, 5% K-feldspar, 1% cristobalite, and traces of halite and pyrite. The specific gravity (G_s) of the bentonite was 2.7. The chemical properties of the experimental seawater are shown in Table 1.

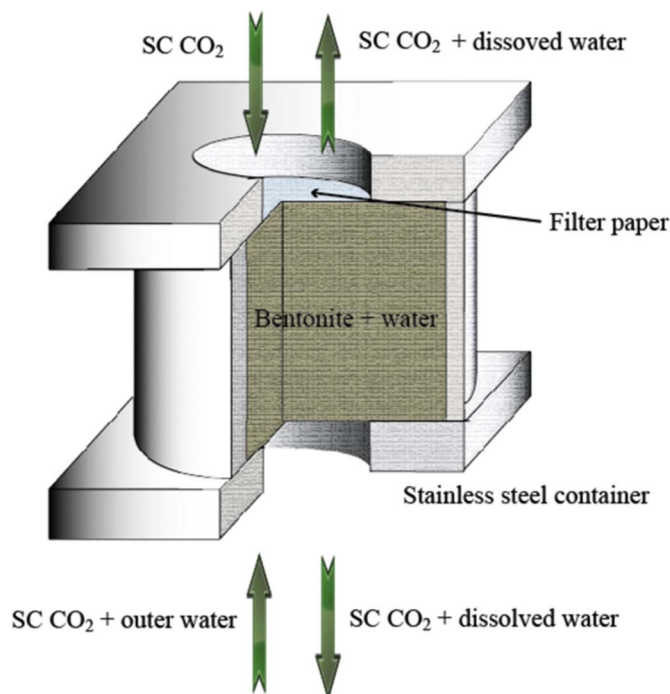
2.2. Vacuum-drying tests

The wet bentonite samples were prepared by mixing bentonite and 1 N NaCl, 1 N CaCl₂, and 1 N MgCl₂ solutions in a 2:1 weight ratio, and then placed in a stainless steel cell. The cell was cylindrical shape, with a 27.2 mm outer diameter, 23.2 mm inner diameter, 15 mm height, and 2 mm thickness. Fig. 1 shows a schematic of the stainless steel cell with the wet bentonite specimen. Filters, with a pore size of 0.45 μm from ADVENTEC®, were placed on the top and bottom of the wet bentonite

Table 1
Chemistry of seawater used in this study.

Material	Concentration (mg/L)					pH
	Na	K	Ca	Mg	Si	
Seawater	8719.0	291.9	323.2	983.0	0.3	8.0

Reaction of supercritical CO₂ with bentonite without water



Reaction of supercritical CO₂ with bentonite in the presence of water

Fig. 1. Schematic of the experimental stainless steel container containing the bentonite mixture.

specimen in the stainless steel cell. Therefore, SC CO₂ and water could penetrate into the bentonite, and the filter prevented the dispersion of bentonite particles. Before placing samples in the stainless steel cell, the mass of each stainless steel cell was measured to determine the subsequent changes in bentonite mass.

The cells containing the wet bentonite samples were placed in a vacuum desiccator. During testing, the diameter and height of the samples were measured using a vernier caliper at designated times (1, 3, 4, and 7 d). The volume and porosity of the bentonite samples were calculated using phase equations (Fredlund and Rahardjo, 1993). Reacted bentonite samples obtained from the vacuum drying tests were dried in an oven at 100 °C for 12 h. The oven dried bentonite samples were transferred right away from the oven to a 50 mL vial in the atmosphere condition. The 50 mL vial, which had the oven dried bentonite, was sealed and stored then in a vacuum desiccator for material characterization. All tests were conducted twice for replication under the same conditions.

2.3. SC CO₂ drying tests

A high-pressure environmental chamber (HPEC, 750 mL, Phosentech, Inc) was used to react the dry SC CO₂ with bentonite and investigate potential interactions in an aquifer saturated with CO₂ in deep geological CO₂ storage sites. Fig. 2 shows a schematic of the HPEC system. The bentonite specimen mixed with 1 N NaCl solution was placed in a stainless steel cell. The container with the wet bentonite specimen was placed in the HPECs for drying using SC CO₂ (i.e., SC CO₂ drying). The CO₂ was pressurized to 12 MPa using a syringe pump and introduced into the HPECs, which were controlled at 50 °C, right after the wet bentonite specimen was placed in the HPECs. The specific temperature and pressure were chosen to investigate interactions between SC CO₂ and caprock appropriate for geological conditions (Suto et al., 2007).

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