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## Development of swelling strain in smectite clays through exposure to carbon dioxide



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#### ABSTRACT

Smectites are common constituents of claystones, mudstones and shales and are often present in the caprocks and faults that seal potential CO<sub>2</sub> storage reservoirs at depths up to 2 or even 3 km. Whilst the marked swelling behaviour of smectites caused by hydration through interlayer sorption of water molecules has been extensively studied, less is known regarding possible interactions of the mineral structure with supercritical CO<sub>2</sub>. To explore the possibility of such effects, and to assess any implications for CO<sub>2</sub> storage, we performed unconfined volumetric strain (dilatometry) measurements on compacted pellets of montmorillonite, a common smectite frequently present in smectite-bearing shales and fault gouges. This was done by exposing SWy-1 and Na-SWy-2 type smectite samples (Wyoming) to CO<sub>2</sub> in a high pressure optical cell. We used this cell to assess the strain response to exposure to  $CO_2$  at the P-T conditions expected in relatively shallow carbon dioxide storage reservoirs, i.e. at a temperature of 45 °C and CO<sub>2</sub> pressures up to 15 MPa. Samples were heat-treated prior to exposure to CO<sub>2</sub> to obtain initial hydration states ( $d_{001}$ -spacing), determined independently using X-ray diffraction methods. Our results show that SWy-1 and Na-SWy-2 montmorillonites swell almost instantaneously (in a few seconds) to an equilibrium state when exposed to (supercritical) CO<sub>2</sub> at the conditions investigated, with both materials exhibiting closely similar behaviour. Swelling strain increases systematically with CO<sub>2</sub> pressure in the range 1-7 MPa, but only slightly at higher pressures. The SWy-1 data show maximum swelling strain for an initial  $d_{001}$  spacing of 11.1 Å, reaching 2.46  $\pm$  0.45% at a CO<sub>2</sub> pressure of 15 MPa. For initial *d*-spacings down to the fully dehydrated state of 9.8 Å, swelling decreases systematically with decreasing d-spacing at all pressures investigated. Only minor swelling ( $0.55 \pm 0.47\%$  at  $10 \text{ MPa CO}_2$  pressure) is found for an initial d<sub>001</sub> spacing of 9.8 Å. For smectite-bearing caprocks and faults sealing carbon sequestration reservoirs, our results imply that CO<sub>2</sub> penetration can be expected to cause swelling of a few percent in the affected zone, closing small fractures or joints and thus reducing bulk permeability.

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#### 1. Introduction

Long-term storage of carbon dioxide in deep geological formations is regarded as a key option for reducing anthropogenic greenhouse gas emissions into the atmosphere (e.g. Bachu, 2003; Lackner, 2003). One of the main knowledge gaps in this field relates to the processes occurring when supercritical CO<sub>2</sub> migrates slowly into the low-permeability pore network present in caprocks and faults. In particular, there is a need to evaluate the long-term effects of CO<sub>2</sub> on caprock and fault sealing integrity (e.g. Hawkes et al., 2005). Many potential storage reservoirs are sealed by claystone or shale caprocks, the generally low permeability of which is predominantly caused by the presence of clay minerals. Smectites (or swelling clays) are common constituents of such rocks and are often present in faults sealing potential storage reservoirs, at least at depths up to about  $\sim$ 2–3 or 3.5 km, i.e. temperatures up to 100 °C (Hower et al., 1976). Beyond this, smectites generally start to transform to the non-swelling clay mineral illite.

The crystal structure of smectites is characterized by octahedral silicate sheets (O) sandwiched between tetrahedral sheets (T) to form TOT layers separated by cation-bearing interlayers (Fig. 1).

Solid solution substitutions in the silicate sheet structure cause a negative charge deficiency, which attracts these charge-balancing cations, and associated water molecules, into the interlayer region. The cation exchange capacity (CEC) is defined as the total number of balancing ions that the structure attracts at a given pH. Uptake of water molecules into the interlayer region causes the structure to expand or shrink, depending on factors such as temperature, pressure, water activity and clay composition (Mooney et al., 1952;



**Fig. 1.** Layered structure and basal unit cell dimension of smectite clay minerals, showing an octahedral silicate sheet enclosed between two tetrahedral silicate sheets, plus the cation-filled and generally hydrated interlayer region. The basal spacing is defined as the  $d_{001}$  distance, which increases with higher hydration states (after Wersin et al., 2004).

Harward and Brindley, 1965; Bishop et al., 1994). This expansion is referred to as "clay swelling" and is widely believed to occur in a stepwise manner (Norrish, 1954), giving rise to specific values of the interlayer spacing or  $d_{001}$  spacing, corresponding to uptake of 1, 2 or 3 layers of water. It produces swelling strains of up to 80%, which can generate significant deformation and damage to structures built in smectite-rich soils (e.g. Anderson et al., 2010), as well as causing borehole and formation damage in smectite-rich hydrocarbon reservoirs and caprocks (e.g. Civan, 2007).

Whereas the water uptake and swelling properties of smectite clays have been studied extensively, few studies have been directed at possible interactions with (supercritical) CO<sub>2</sub> under conditions typical for geological storage. However, several consequences including interlayer shrinkage (due to dehydration) and swelling (surface adsorption and/or uptake of CO<sub>2</sub> into the interlayer region) of smectites are conceivable, which could have important implications for caprock and fault integrity. Recently, Giesting et al. (2012a) used a high pressure environmental chamber (HPEC) to conduct transmission (THPEC) and reflection (RHPEC) mode in situ XRD experiments, studying smectite-CO<sub>2</sub> interactions by investigating shifts in basal spacing  $(d_{001})$  reflections. It was shown that Na-exchanged montmorillonite (Na-SWy-2) can swell by up to 9% when exposed to high purity CO<sub>2</sub>, depending on the initial hydration state (or  $d_{001}$  spacing) of the interlayers. The swelling effect was observed for initial hydration states located in the range  $\sim 10$  Å  $\leq d_{001} \leq \sim 12$  Å. Almost no (*d*-spacing change) was observed for zero interlayer water  $(d_{001} \sim 9.8 \text{ \AA})$  or for the single water (1 W) layer configuration ( $d_{001} \sim 12.3 - 12.5$  Å) upon exposure to CO<sub>2</sub>. Using similar methods and initial conditions, CO<sub>2</sub>-induced swelling was also observed recently in Ca-exchanged STx-1 montmorillonite under similar initial conditions by Schaef et al. (2012) and by Giesting et al. (2012b) in SWy-2 samples. In addition, neutron diffraction experiments on sub-1W Na montmorillonite (i.e. with less than one water layer) have provided direct evidence for CO<sub>2</sub>-induced interlayer (i.e. *d*-spacing) swelling of the order of 4% (Rother et al., 2013). Besides expansion for initial hydration states in the range of  $\sim 10$  Å  $\leq d_{001} \leq \sim 12$  Å, CO<sub>2</sub>-induced shrinkage due to interlayer dehydration was also reported by Schaef et al. (2012), but only for initial hydration states  $\geq 2 W$  ( $d_{001} \geq 15 \text{ Å}$ ). A suite of similar experiments has been reported by Ilton et al. (2012) for Na-exchanged smectite (Na-STx1), but using watersaturated and not dry CO<sub>2</sub>. These authors observed expansion for low hydration states (0-2W), pointing to (inseparable) effects of water and/or CO<sub>2</sub> uptake, compared with interlayer shrinkage for samples with an initial hydration state of 2 or more interlayer water layers, suggesting dehydration. Alongside this work, using the same STx-1 sample as Schaef et al. (2012), Loring et al. (2012) found direct intercalation of CO<sub>2</sub> in the interlayer space. This was done using a combination of X-ray diffraction, nuclear magnetic Download English Version:

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