Measurements of the capillary trapping of super-critical carbon dioxide in Berea sandstone

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[1] We measure primary drainage capillary pressure and the relationship between initial and residual non-wetting phase saturation for a supercritical carbon dioxide (CO₂)brine system in Berea sandstone. We use the semipermeable disk (porous-plate) coreflood method. Brine and CO₂ were equilibrated prior to injection to ensure immiscible displacement. A maximum CO₂ saturation of 85% was measured for an applied capillary pressure of 296 kPa. After injection of brine the CO₂ saturation dropped to 35%; this is less than the maximum trapped saturation of 48% measured in an equivalent *n*-decane (oil)-brine experiment. The dimensionless capillary pressure is the same to within experimental error for supercritical CO₂-brine, *n*-decane-brine and a mercury-air system. CO₂ is the non-wetting phase and significant quantities can be trapped by capillary forces. We discuss the implications for CO₂ storage. Citation: Pentland, C. H., R. El-Maghraby, S. Iglauer, and M. J. Blunt (2011), Measurements of the capillary trapping of super-critical carbon dioxide in Berea sandstone, Geophys. Res. Lett., 38, L06401, doi:10.1029/ 2011GL046683.

1. Introduction

[2] Saline aquifers have been identified as suitable storage locations for anthropogenic CO₂ emissions collected from fossil-fuel burning power stations and other industrial sites, because of their large capacities and wide geographical spread [Lackner, 2003]. Storage would likely occur at depths greater than 800 m where the formation temperature and pressure render the CO₂ in a dense supercritical phase. The CO₂ is trapped by dissolution, mineralisation, structural and stratigraphic confinement, and capillary trapping. Capillary trapping is a rapid and effective mechanism to ensure safe storage. It occurs when brine displaces and traps the CO₂ as discontinuous droplets at the trailing edge of a rising CO₂ plume, or when engineered through brine injection [*Ide* et al., 2007; Juanes et al., 2006; Kumar et al., 2005; Qi et al., 2009; Saadatpoor et al., 2009]. Where the resident formation brine has become saturated with CO2 - and the CO₂ is saturated with water – there will be a region within the aquifer where immiscible displacement occurs. Simulation and analytical studies imply that this region covers the majority of the plume [Hesse et al., 2008; Qi et al., 2009].

[3] There are two key properties that influence the subsurface behaviour of CO₂: the primary drainage capillary pressure; and hysteresis in the CO₂ saturation – specifically,

the amount of CO_2 that is trapped as a function of initial CO_2 saturation – the trapping curve. The primary drainage capillary pressure determines the threshold pressure to enter the rock that controls the ability of low permeability layers to stop the upwards migration of CO_2 . The trapping curve will determine how much CO_2 can be rendered immobile by capillary forces which in turn controls how far the CO_2 migrates and the effectiveness of brine injection as a design for safe storage [Oi et al., 2009].

[4] If supercritical CO₂ were the non-wetting phase in the presence of aquifer brines, then there would be significant trapping of CO₂, allowing safe storage [Hesse et al., 2008; Oi et al., 2009]. In contrast, if the CO₂ were wetting or neutrally-wet, there would be little capillary trapping within the aquifer, making safe CO2 storage problematic. For instance, Spiteri et al. [2008] showed using pore-scale modelling that the residual saturation can be almost 0.5 in Berea sandstone when the trapped phase is non-wetting, but that this saturation drops rapidly to 0.25 when the contact angle becomes greater than 90° and falls further if the residual phase is wetting (contact angles much larger than 90°). Capillary pressure and the trapping curve have been measured before on oil-water systems with applications to hydrocarbon recovery, where the oil is the non-wetting phase (see Pentland et al. [2010] for a review of the literature). However, only isolated measurements of trapped CO₂ saturation have been reported [*Plug and Bruining*, 2007; Perrin and Benson, 2010; Bennion and Bachu, 2008; Okabe et al., 2010; Shi et al., 2010; Suekane et al., 2008, 2009]. The CO₂ trapping curve has not been measured and there has been no comparison with equivalent strongly non-wetting systems.

[5] The receding contact angle (CO₂ displacing brine) affects the primary drainage capillary pressure and caprock invasion, while the advancing angle (brine displacing CO₂) governs capillary trapping. A recent review of the literature [Naylor et al., 2010] concluded that for primary drainage, super-critical CO₂ remains the non-wetting phase with contact angles up to 49°. For brine displacing CO₂, however, there is disagreement over the typical wettability. *Plug* and Bruining [2007] measured the drainage and imbibition capillary pressures for super-critical CO₂ and suggested that during water displacement, CO₂ could become the wetting phase. Dickson et al. [2006] and Yang et al. [2008] found values of the advancing contact angle close to 90° on limestone and chemically treated glass surfaces, again implying neutral wettability with little trapping. However, several other authors [Chiquet et al., 2007; Espinoza and Santamarina, 2010; Tonnet et al., 2010; Shah et al., 2008] found lower contact angles on quartz, mica and caprock surfaces: although the contact angle is a function of temperature and pressure, at super-critical conditions CO₂ remained

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Table 1. Estimated Phase Properties at 343 K and 9 MPa (Unless Stated) From the Literature

	CO_2	Brine (CO ₂ Saturated)	n-Decane
Density, ρ (kg m ⁻³)	$208^{a} \\ 2.1 \times 10^{-5} \text{ d}$	$1025^{\rm b}$	700 ^c
Viscosity, μ (Pa s)		5.1 × 10^{-4} e	5.5 × 10 ⁻⁴ f

^aSpan and Wagner [1996].

the non-wetting phase. We address this ambiguity by measuring directly the physical quantities of interest: namely the amount of capillary trapping for the full range of initial saturation – the trapping curve – and the primary drainage capillary pressure on a Berea sandstone core, representative of aquifer rocks. We use the semi-permeable disk (porous plate) technique to control the initial saturation and a stirred reactor to ensure equilibrium between the CO₂ and brine prior to injection. Our results suggest that CO₂ is the non-wetting phase and that significant trapping is possible.

2. Experiments

[6] The experiments were performed on two consolidated Berea sandstone samples. One sample was used for CO₂brine experiments and the other for oil-brine experiments. Measured properties were the same for both samples (porosity = 0.22; brine permeability = 4.6×10^{-13} m²; diameter = 0.0385 m; length = 0.0753 m). A modified semipermeable disk (porous plate) method [Hassler and Brunner, 1945; McCullough et al., 1944] was used to vary the capillary pressure and hence saturation within the sample. A hydrophilic semi-permeable disk (aluminium silicate, Weatherford Laboratories, Stavanger, Norway) was placed at one end of the rock sample. The non-wetting phase was injected into the opposite face of the rock sample and its production was prevented by the high capillary entry pressure of the disk on the outlet face. The resident brine could pass freely through the disk. In this manner the brine pressure was held almost constant throughout the system while the non-wetting phase pressure was increased. The difference between the phase pressures was equal to the capillary pressure at hydrodynamic equilibrium.

2.1. Experimental Conditions

[7] Experiments were performed at conditions representative of storage formations – 343 K (70°C) and 9 MPa pore pressure. The wetting phase was aqueous 5wt.%-sodium chloride, 1wt.%-potassium chloride synthetic brine which was saturated with CO_2 in the CO_2 -brine experiments using a stirred reactor. In the oil-brine experiments the non-wetting phase was n-decane. The phase properties at these conditions are estimated from the literature and shown in Table 1.

2.2. Experimental Equipment

[8] The rock sample and semi-permeable disk were housed within a pressure cell. A radial confining pressure (2.76 MPa above pore pressure) was applied. The semi-permeable disk was modified by drilling a hole in the centre,

through which a tube could pass. This tube was used during waterflooding to avoid large pressure drops across the disk. The tube was connected to a valve which was closed during primary drainage to prevent non-wetting phase production. A fluoroelastomer o-ring provided a seal between tube and disk to prevent bypassing of the non-wetting phase. All flowlines and most valves were C276 alloy metal to mitigate the risk of corrosion. Experimental pressure and displacement rates were controlled by high precision syringe pumps (Teledyne ISCO 500D & 1000D, Lincoln, NE, USA). One pump was used to pressurise the non-wetting phase to experimental conditions for injection into the sample. An identical pump received the effluent from the sample during each flooding sequence. In the CO₂-brine experiments the phases were equilibrated prior to use inside a stirred reactor (1200mL C276 autoclave with gas entrainment stirrer – Parr Instruments Co., IL, USA).

2.3. Experimental Procedure

- [9] The CO₂-brine experimental procedure is given below; it provides one unique data point comprising initial saturation, corresponding drainage capillary pressure and residual saturation after waterflooding. After depressurisation of the pore space to measure the residual CO₂ saturation steps 2–5 were repeated to vary the initial saturation, each time producing an additional data point.:
- [10] Phases equilibrated in the stirred reactor at experimental conditions.
- [11] Rock sample and semi-permeable disk saturated with brine.
- [12] CO_2 injected into rock sample at a predetermined capillary pressure P_c (primary drainage) to initial saturation S_c
 - [13] Rock sample waterflooded to residual saturation $S_{\rm r}$.
- [14] S_r measured by an isothermal depressurisation of the pore space.
- [15] The phases were initially equilibrated by agitation in the stirred reactor. Equilibrium was reached when no more CO₂ entered the rector from an attached pump held at constant pressure. The solubility of CO₂ in brine was measured to be a mole fraction of 0.0136 ± 0.0020 for the experimental conditions investigated. This is in the range of solubilities measured on similar systems [Koschel et al., 2006]. The sample was saturated by introducing un-equilibrated brine into the pore space under vacuum. Once the pore space was pressurised injection was switched to equilibrated brine and five pore volumes were pumped though the sample. During immiscible CO₂ injection, hydrodynamic equilibrium corresponded to a uniform pressure distribution along the sample length in each of the phases. The difference in the equilibrium pressure of the two phases corresponded to P_c . At equilibrium the phase saturations were assumed to be uniform along the length of the sample. No additional CO₂ entered the sample and no brine was produced. P_c was accurately controlled by the inlet and outlet syringe pumps. The difference in pump pressures was varied up to 296 kPa at equilibrium. This is equivalent to a CO₂ column height of 37 m in a formation at our experimental conditions. This $P_{\rm c}$ was sufficient to de-saturate the sample to irreducible brine saturation. The duration of primary drainage was inversely related to the applied $P_{\rm c}$. The longest drainage time was 166 hours and the shortest was 14 hours. The sample was flooded to residual CO₂ saturation by immiscibly displacing

^bBando et al. [2003] assuming an unsaturated brine density of 1020 kg m⁻³ [Mao and Duan, 2008].

^cBanipal et al. [1991] and Lee and Ellington [1965].

^dFenghour et al. [1998].

^eBando et al. [2003]; 3.0wt.% NaCl aqueous solution at 10.0 MPa, 333 K.

^fLee and Ellington [1965]; 9.48 MPa, 344K.

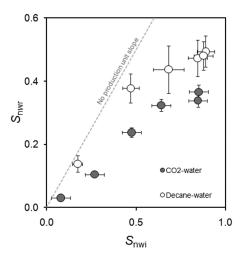


Figure 1. Saturation hysteresis data – the trapping curve – measured on Berea sandstone.

and trapping CO_2 with equilibrated brine. Five pore volumes were injected at a capillary number of 4.1×10^{-7} ; the maximum capillary number during drainage was in the range $0.5\text{--}3 \times 10^{-10}$. It was not possible to measure imbibition capillary pressures with our apparatus. The initial CO_2 saturation was determined from a volume balance measurement. The volume of brine produced during primary drainage was considered equal to the volume of CO_2 injected. Knowing the dead volume and pore volume S_i was calculated. S_r was measured by isothermal depressurisation of the CO_2 within the sample pore space immediately after waterflooding [Suekane et al., 2008]. The same procedure was followed in the oil-brine experiments except S_i and S_r were determined by mass and volume balance measurements and phase pre-equilibration was unnecessary – further details are given elsewhere [Pentland et al., 2010].

3. Results

[16] The capillary trapping curve $-S_r$ as a function of S_i is shown in Figure 1. A small but distinct difference exists between the magnitude of S_r in the CO_2 -brine and oil-brine systems – especially at high S_i .

[17] Figure 2 shows the dimensionless primary drainage capillary pressure – expressed as the Leverett-J Function $J[S_{\rm w}]$ [Leverett, 1941] – for each corresponding $S_{\rm i}$ in Figure 1. $J(S_{\rm w})$ is defined as [Rose and Bruce, 1949]:

$$J(S_{\rm W}) = \frac{P_c}{\sigma \cos \theta} \left(\frac{K}{\phi}\right)^{1/2} \tag{1}$$

where K and ϕ are the sample permeability and porosity respectively, σ the interfacial tension and θ the contact angle between the phases and the solid rock surface. Also shown in Figure 2 are mercury injection capillary pressure (MICP) measurements (Autopore IV 9520; Weatherford Laboratories, Stavanger, Norway) performed on the same Berea rock type at a commercial core laboratory. The interfacial tensions for the CO₂-brine, oil-brine and mercury-air systems are assumed to be 36.0 [Chalbaud et al., 2009; Chiquet et al., 2007], 48.3 [Zeppieri et al., 2001] and 485 mN m⁻¹ respec-

tively. The contact angle during primary drainage is assumed to be 130° for mercury-air and 0° for CO_2 -brine and oil-brine. There is good agreement between the mercury-air, oil-brine and CO_2 -brine datasets with the exception of the absence of an irreducible saturation in the MICP results. This is expected as the irreducible saturation is clay bound in the presence of brine [Omoregle, 1988]. The error bars on Figures 1 and 2 represent the standard deviation of the measured values; the greatest errors (as a proportion of the absolute value) were associated with the differential pressure at low capillary pressure. Each data point represents one complete experiment: primary drainage and brine flooding. We checked the reproducibility of the results by repeating several experiments with similar initial CO_2 or oil saturations; the results were similar to within the estimated measurement error.

4. Discussion and Conclusions

[18] Significant quantities of CO₂ (up to 35% of the pore space) are trapped in Berea sandstone by capillary forces after the re-injection of brine. This is achieved by applying a realistic *P*_c during primary drainage equivalent to a CO₂ column height of 37 m. When compared with oil-brine data there is a decrease in the quantity of capillary trapping. It is proposed that this is due to an increase in the effective imbibition (advancing) contact angle in the CO₂-brine system, consistent with measurements of contact angle on quartz surfaces (Berea sandstone is principally composed of quartz) at conditions similar to ours by *Chiquet et al.* [2007] and *Espinoza and Santamarina* [2010]. However, the system remains water-wet with a large degree of trapping possible. If the rock were neutrally or CO₂-wet,

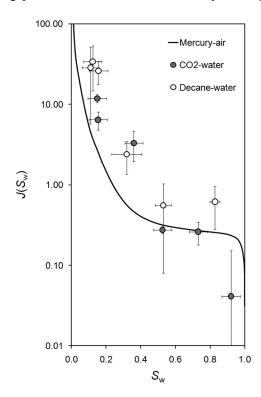


Figure 2. Primary drainage capillary pressure versus saturation data measured on Berea sandstone. The three sets of data (CO₂-brine; oil-brine; mercury-air) have been normalised using the Leverett-J Function (equation (1)).

lower residual saturations would have been expected, as CO₂ would no longer become stranded in the larger pore spaces [Spiteri et al., 2008].

[19] The dimensionless capillary pressure (the Leverett J function) is the same, to within experimental error, for oilbrine and CO₂-brine systems assuming, in both cases, that the contact angle during primary drainage is zero, except at the lowest capillary pressures. The discrepancy at low pressures (corresponding to low non-wetting phase saturations) could be caused by dynamic effects, pressure fluctuations or subtle differences in wettability. The assumption of low contact angles during primary drainage is in agreement with other studies, as reviewed by Naylor et al. [2010]. Future work will focus on investigating a wider range of pressure and temperature conditions and different rock samples and relating these to independent measurements of wettability and contact angle.

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