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Energy Procedia 4 (2011) 4969–4976

**Energy
Procedia**

www.elsevier.com/locate/procedia

GHGT-10

Immiscible Displacements and Capillary Trapping in CO₂ Storage

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Abstract

We measure the residual non-wetting phase saturation of super-critical carbon dioxide in a Berea sandstone core. We use the porous plate method while a stirred reactor ensures equilibrium between the carbon dioxide and brine. We also measure carbon dioxide-brine contact angles on the porous plate to understand wetting behavior in the experiment. The application of the work is for carbon dioxide storage in aquifers, where capillary trapping is a rapid and effective mechanism to render the injected fluid immobile. The experiment was performed at temperature and pressure representative of potential subsurface storage formations. The measured residual saturation is 37% which is lower than the measured residual for an oil-brine system on a similar core (48%), but higher than measured by other authors for super-critical CO₂ in Berea sandstone. We suggest that super-critical CO₂ is still non-wetting in sandstones with considerable trapping and discuss the implications for CO₂ storage in aquifers.

© 2011 Published by Elsevier Ltd. Open access under [CC BY-NC-ND license](https://creativecommons.org/licenses/by-nc-nd/4.0/).*Keywords:* Capillary trapping; Residual saturation; Trapping capacity; Porous plate; Contact angle

1. Introduction

Saline aquifers have been identified as suitable storage formations for injected carbon dioxide (CO₂) due to their wide geographical spread and large storage capacities [1-3]. When CO₂ is injected into a saline aquifer its movement is controlled by a number of parameters including: the solubility of CO₂ in the formation brine; the phase densities and viscosities; the petrophysical properties of the aquifer; and the injection rate and duration. Mass transfer between CO₂ and brine will occur at the leading edge of the injected CO₂ plume as it comes into contact with fresh formation brine. Behind the leading edge the CO₂ and brine in the pore space are mutually saturated and immiscible displacements dominate. As the CO₂ plume rises upwards in the formation – due to the density contrast with brine – there will be secondary imbibition at the trailing edge of the plume [4,5]. The re-imbibition of brine into the pore space will trap CO₂ as a residual discontinuous phase held in place by local capillary forces – this process is called capillary trapping.

Capillary trapping has been extensively studied due to its importance for hydrocarbon recovery and contaminant remediation. It is proposed that capillary trapping can also play an important role in CO₂ storage – as it is a fast and secure method of storing CO₂ in porous media. In this context it is desirable to

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maximize the trapped non-wetting phase (CO₂) within the porous media. One way of achieving this is to engineer the process, i.e. by the injection of additional brine into the formation after CO₂ injection [6,7].

Capillary trapping can be characterized by measuring the form of the initial versus residual non-wetting phase saturation curve – the trapping curve. This has been extensively measured for gas-water and oil-water sand/sandstone systems [8-19]. A number of studies have proposed empirical equations to generate the trapping curve if the end point of the curve is known – that is the maximum residual saturation (S_r^{\max}) for a given maximum initial saturation (S_i^{\max}). The advantage being that only one measurement is required instead of several, saving considerable time. Recent research [19] shows that the Land, [18] and Spiteri *et al.*, [20] equations provide the best match to measurements on consolidated sandstone samples. The Aissaoui, [21] equation provides the best match to unconsolidated sand pack data [16].

To date the trapping curve has not been measured for CO₂-brine fluid systems. Research has primarily focused on relative permeability [22-24], the influence of sub core scale heterogeneity [24-26], mineral-CO₂ interactions [27-29], and the measurement of residual saturations [30-32]. This paper complements the data on residual saturation – specifically S_r^{\max} – with a new CO₂-brine coreflood experiment performed at flow rates representative of storage conditions.

If S_r^{\max} and porosity (ϕ) are known then the capillary trapping capacity (C_{trap}) of a given rock can be determined as:

$$C_{\text{trap}} = \phi S_r^{\max} \quad (1)$$

The influence of porosity on C_{trap} has been studied previously for oil-water systems [17].

System wettability is a key control on the ability to trap CO₂ as a residual immobile phase as it determines fluid distribution on the pore scale. If CO₂ were to wet the rock surface the amount of trapping would be very limited – however there is little evidence from measurements that this is the case. Water-CO₂ contact angles have been measured on various substrates [33-35]. The results for untreated silicate minerals show that water will be the wetting phase in these systems (defined as $\theta < 90^\circ$ [36] where θ is measured through the denser brine phase).

An immiscible displacement coreflood experiment was performed to measure S_r^{\max} on a Berea sandstone core-plug. The results are analyzed in terms of C_{trap} and compared with the Berea CO₂-brine data of Suekane *et al.*, [30]. The system wettability is inferred from coreflood results and the solubility of CO₂ in brine is measured. In addition contact angle measurements are performed on a silicate mineral substrate.

2. Experimental Conditions and Equipment

Experimental conditions representative of saline aquifers ($T = 343$ K; Pore pressure = 9 MPa) were considered. The cylindrical Berea core-plug used was 0.0384 m in diameter and 0.0754 m in length. The wetting phase in the experiment was 5wt.-%-sodium chloride, 1wt.-%-potassium chloride synthetic brine. Table 1 summarizes the petrophysical properties of the Berea core-plug.

The importance of immiscible displacements to CO₂-brine capillary trapping has been highlighted. In order to perform such laboratory displacement experiments special care must be taken in the equilibration of the CO₂ and brine phases. If the phases are not mutually saturated then there will be mass transfer across the phase interface leading to saturation changes in the pore space which are not controlled by capillary forces. In order to achieve equilibrium between the CO₂ and brine at the experimental conditions a coreflood experiment was designed and built around an equilibration reactor (1200 mL C276 autoclave with gas entrainment stirrer – Parr Instruments Co., IL, USA).

Table 1: Petrophysical properties of the Berea core-plug used in the CO₂-water coreflood experiments.

Porosity	Brine Permeability (m ²)	Formation Factor
0.222	4.4×10^{-13}	14

The coreflood displacement method used to achieve initial and residual CO₂ saturations is of paramount importance. Steady and unsteady state CO₂-brine displacement methods have been reported elsewhere in the literature [24-26,30-32], however these methods are unable to achieve a homogenous saturation profile along the length of the core-plug due to the influence of capillary end effects. In addition a study of the literature indicates that high initial CO₂ saturations (> 80%) are rarely achieved with these methods. A modified porous plate method was used in this study. A hydrophilic ceramic disk (15bar, Aluminium Silicate, Weatherford Laboratories, Stavanger, Norway) was placed immediately downstream of the core-plug. The porous plate retains the non-wetting CO₂ phase within the core-plug due to the capillary entry pressure constraints of the porous plate's small diameter pores. During CO₂ injection equilibrium corresponds to a uniform pressure distribution along the core-plug length in each of the phases. The difference in the equilibrium pressure of the two phases corresponds to the capillary pressure (P_c). At equilibrium the phase saturations are assumed to be uniform along the length of the core-plug. No additional CO₂ enters the core-plug and no brine is produced. The porous plate method has been used previously to measure CO₂-water capillary pressure curves for unconsolidated sand [37]. We modified the porous plate to shorten the duration of waterflooding. A hole was drilled through the centre of the porous plate through which a 3.175×10^{-3} m (1/8") tube could pass. This tube was used during initial saturation and waterflooding to avoid large pressure drops across the porous plate. The tube was connected to a valve which was closed during primary drainage to prevent CO₂ production. A fluoroelastomer (Viton®) o-ring provided a seal between tube and porous plate to prevent bypassing.

The core-plug was housed in a custom Hassler type core holder. All flowlines and valves were C276 alloy metal to mitigate the risk of corrosion. The equilibration reactor was housed in an oil bath and the core holder in an air bath – both maintained at the experimental temperature. Experimental pressure and displacement rates were controlled by high precision syringe pumps (Teledyne ISCO 500D & 1000D, Lincoln, NE, USA). Super-critical CO₂ (scCO₂) was generated inside a custom 1000 mL piston accumulator (Phoenix Instruments, Splendora, TX, USA) controlled by a syringe pump. The coreflood effluent was received in an identical piston accumulator, again controlled by a syringe pump. This effluent accumulator controlled the system back pressure. Figure 1 shows the arrangement of the experimental equipment.

Contact angle measurements were performed on a separate apparatus which consisted of a view cell (Sitec, model 740.2093-5, Zurich, Switzerland) connected to a custom designed high pressure apparatus (Eurotechnica GmbH, model PD-E700 LL, Bargteheide, Germany) with operating pressure and temperature range up to 70 MPa and 473 K respectively. A detailed description of the apparatus and operating procedures is given elsewhere [38].

3. Experimental Procedure

Contact Angle Measurement. Before performing saturation measurements the contact angle of the CO₂-water-porous plate system was investigated. This was done to develop confidence in the ability of the porous plate to prevent CO₂ production during primary drainage and to investigate wettability in silicate mineral systems. A segment of porous plate was placed inside a view-cell which was filled with de-ionized water and heated to 354.5 K. The water was pressurized and CO₂ introduced onto the surface of the porous plate via a capillary tube from a screw injector. The porous plate was placed at an angle of 21° from the

horizontal to investigate advancing and receding contact angles (raindrop effect – Bear, [36]). Contact angles were measured for pressures between 26.6 MPa and 48.9 MPa.

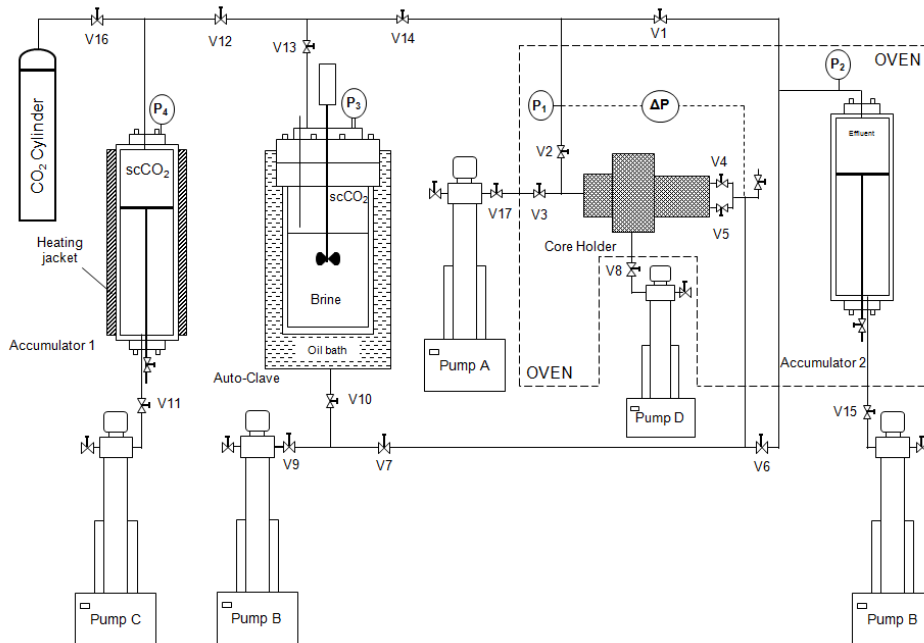


Figure 1: Schematic diagram of the experimental setup for measuring capillary trapping in scCO₂-brine-sandstone systems.

Phase Equilibration. The equilibration reactor was half filled with de-aired brine and half filled with scCO₂. The top of the autoclave was connected to a reservoir of scCO₂ maintained at constant pressure by a syringe pump. The reactor's internal gas entrainment stirrer was then rotated at 200 r.p.m. to mix the phases. Brine was sampled from a bottom drain valve and the solubility of CO₂ in brine measured. Equilibrium was reached when no more CO₂ entered the reactor from the reservoir with continued rotation of the stirrer shaft. The equilibrated brine was stored in the reactor at constant pressure and temperature until required in the experiment.

Initial Saturation. The core-plug pore space was initially vacuumed for 15 minutes with the experimental confining pressure applied (11.72 MPa). Fresh (unsaturated with CO₂) de-aired brine was then injected into the core-plug from right to left (Figure 1). The fresh brine was initially introduced at ambient conditions then pressurized to 9 MPa. Five pore volumes (PVs) of fresh brine were injected through the core-plug. Flooding was then switched to the equilibrated brine for a further five PVs of injection. The flowlines connected to both valves 4 and 5 were used during injection. Valve 4 was connected to the flowline terminating at the outside face of the porous plate. Valve 5 was connected to the flowline that passed through the centre of the porous plate terminating at the face of the core-plug. Brine production was from the flowline connected to valve 2.

Primary Drainage. During primary drainage CO₂ was injected from left to right in the core-plug. CO₂ was introduced via the valve 2 flowline. At the outlet face the valve 5 flowline was closed to prevent bypassing of the porous plate. As CO₂ entered the core-plug the displaced brine was produced through the porous plate and out of the core holder via the valve 4 flowline. The injection of CO₂ was controlled by operating the inlet and outlet syringe pumps in constant pressure mode. The difference in pump pressures was 0.28

MPa at equilibrium – corresponding to the applied P_c . Equilibrium was reached when the production of brine and injection of CO_2 ceased. The brine produced was considered equal in volume to the CO_2 injected. The initial CO_2 saturation was calculated based on this volume measurement. The duration of primary drainage was 20 hours.

Waterflooding. The core-plug was waterflooded to residual CO_2 saturation with equilibrated brine. Waterflooding took place from right to left in the same manner as initial saturation. Five PVs of equilibrated brine were injected at a capillary number of 4.2×10^{-7} .

Residual Saturation Measurement. The residual CO_2 saturation was measured by isothermal expansion of the core-plug pore space, as per Suekane *et al.*, [30]. The pore space was initially isolated from the experimental system before connection to an empty syringe pump (via valve 3). The pump was maintained at the experimental temperature and the internal volume increased while the pressure was monitored. Knowing the pore volume, dead volume, volume of expansion, pressure, CO_2 density and CO_2 solubility in brine the CO_2 saturation was calculated.

4. Results

The contact angle measurements are summarized in Table 2. In addition Figure 2 shows the CO_2 -water-porous plate contact angle at the maximum pressure (48.9 MPa). The contact angles on the two sides of the drop were found to be almost identical (within 1-2 degrees). Smaller contact angles at lower pressures were possibly an effect of small contact force (CO_2 drop just reached the substrate); whereas at higher pressures (39.5 – 48.9 MPa) good contact was achieved and thus contact angles remained stable at 40° . Another explanation for the observed change in contact angle with pressure is the increase of CO_2 solubility in brine and subsequent shift in the reactions $\text{CO}_2 + \text{H}_2\text{O} \leftrightarrow \text{H}_2\text{CO}_3 \leftrightarrow \text{H}^+ + \text{HCO}_3^- \leftrightarrow 2 \text{H}^+ + \text{CO}_3^{2-}$ to the right side. The drop in the pH value and an increase in HCO_3^- and CO_3^{2-} concentrations may have changed the surface chemistry of the substrate and the electric double-layer on it, influencing the spreading of CO_2 . This effect requires further study.

The solubility of CO_2 in brine was measured as 0.0136 mole fraction \pm 0.0020 mole fraction ($32.4 \text{ kg m}^{-3} \pm 4.8 \text{ kg m}^{-3}$) for the experimental conditions investigated. This is in the range of solubilities measured on similar systems [39-43]. The solubility was measured by the same isothermal expansion procedure used in pore space saturation measurement. The coreflood experiment gave a maximum residual CO_2 saturation of 37% (\pm 2%) for a corresponding initial CO_2 saturation of 90% (\pm 5%). Applying equation 1 to these results yielded a C_{trap} of 8.2%. These results are summarized and compared to those of Suekane *et al.*, [30] in Table 3.

Table 2: Contact angle measurements at 354.5 K between the de-ionized water and scCO_2 phase interface on a porous plate (aluminium silicate). Contact angles are reported for the denser brine phase.

Pressure (MPa)	θ (degrees)
26.6	10 ± 5
29.7	32 ± 2
32.0	30 ± 2
39.5	39 ± 2
47.0	40 ± 2
48.9	40 ± 3



Figure 2: A drop of scCO_2 generated from a capillary tube is brought into contact with a porous plate substrate angled at 21° . The bulk phase is de-ionized water ($P = 48.9 \text{ MPa}$).

The CO₂-brine S_r^{\max} can be compared with the oil-brine S_r^{\max} of 48% reported previously [19] – table 4. Both experiments were performed at the same conditions of temperature and pressure and on core-plugs from the same batch of Berea sandstone.

Table 3: Summary of coreflood results from this study and a comparison with literature capillary trapping data.

Study	Sample	ϕ	T (K)	P (MPa)	S_i	S_r	C_{trap}
Suekane <i>et al.</i> , 2008	Berea	0.198	308 - 323	7.6 – 10.0	-	0.25 - 0.28	0.050 - 0.055
This study	Berea	0.222	343	9.0	0.90	0.37	0.082

Table 4: Summary of oil-brine and scCO₂-brine residual saturation coreflood data.

Study	Sample	ϕ	Phases	IFT (mN m ⁻¹)	S_i	S_r
Pentland <i>et al.</i> , 2010	Berea	0.221	<i>n</i> -decane – brine	48.3 ^a	0.88	0.48
This study	Berea	0.222	CO ₂ – brine	32.5 ^b	0.90	0.37

^a Zeppieri *et al.*, 2001; linearly extrapolated to 343K.

^b Chalbaud *et al.*, 2009; experimental data for 345.1 K, 9.56 MPa and 0.87 m NaCl salinity.

5. Conclusions

Laboratory measurements of contact angle and capillary trapping were presented for a CO₂-brine system. Capillary trapping was measured by performing immiscible displacement sequences in a water-wet Berea sandstone using the porous plate method. Contact angles were measured on an aluminium silicate substrate which was found to be water-wet. For Berea sandstone the maximum residual CO₂ saturation was shown to be higher than literature measurements indicating that up to 8.2% of the gross rock volume can contain CO₂ trapped on the pore scale by capillary forces. This is over 50% higher than previous measurements [30]. Lower residual saturations in previous studies may have been a result of the experimental procedure (unsteady state versus porous plate) where lower initial CO₂ saturations would be expected.

The residual saturation measured in this study indicates that CO₂ was the non-wetting phase in the system. If the CO₂ had completely wet the sandstone then lower residual saturations would have been expected as the CO₂ drained from the sample through wetting layers during waterflooding. The contact angles measured in this study add weight to this theory – CO₂ was clearly shown to be the non-wetting phase in the presence of water on an aluminium silicate substrate.

The CO₂-brine residual saturation measured in this study was compared to a literature measurement of oil-brine residual saturation for the same rock type [19]. The lower CO₂-brine residual saturation could be attributed to the lower CO₂-brine interfacial tension and associated changes in contact angle. For a given waterflood capillary pressure more CO₂ than oil would be produced from the same rock sample. However, the reduction in residual saturation compared to analogue oil-brine systems is modest and implies that super-critical CO₂ still behaves as a strongly non-wetting phase in clean sandstones.

Future work will focus on investigating system wettability further and measuring the shape of the trapping curve for CO₂-brine systems and different rock types.

Nomenclature

- C_{trap} = capillary trapping capacity
 P_c = applied capillary pressure, Pa
 S_i^{max} = maximum initial (post-primary drainage) saturation
 S_r^{max} = maximum residual (post-secondary imbibition) saturation
 S_i = initial (post-primary drainage) non-wetting phase saturation
 S_r = residual (post-imbibition) non-wetting phase saturation
 θ = macroscopic intrinsic contact angle, °
 ϕ = porosity

Acknowledgments

We would like to thank Shell under the Grand Challenge on Clean Fossil Fuels for supporting this work. The authors gratefully acknowledge Alexander Toth and Graham Nash for providing technical laboratory support and advice.

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