THE IMPACT OF RESERVOIR CONDITIONS ON WETTING AND MULTIPHASE FLOW PROPERTIES MEASUREMENTS FOR CO_2-BRINE-ROCK SYSTEM DURING PRIMARY DRAINAGE

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ABSTRACT
The wettability of CO_2-brine-rock systems will have a major impact on the management of safe carbon sequestration in subsurface geological formations. The wetting properties of a system controls the mobility and trapping efficiency of CO_2 during injection processes for carbon storage as well as for enhanced oil recovery operations. Recent contact angle measurement studies have reported significantly different wetting behaviour with regard to pressure, temperature and water salinity. We report the results of an experimental investigation into the wetting properties of CO_2 and brine solutions in a single Berea sandstone utilising measurements of the multiphase flow properties under a wide range of reservoir conditions with pressures (5 to 20 MPa), temperatures (25 to 50 °C) and ionic strengths (0 to 5 M kg-1 NaCl). Primary drainage capillary pressure curves were measured in a horizontal core flooding apparatus using the semi-dynamic method to investigate the wetting properties during CO_2 injection process. The observations were made using a reservoir condition core-flooding laboratory that included high precision pumps, temperature control, the ability to recirculate fluids for weeks at a time and in situ saturation monitoring with x-ray CT scanner. The wetted parts of the flow-loop are made of anti-corrosive material that can handle co-circulation of CO_2 and brine at reservoir conditions. Measurements in the Berea sample were made using CO_2-brine and N_2-water. The capillarity of the system, scaled by the interfacial tension, were equivalent to the N_2-water system. Thus reservoir conditions did not have a significant impact on the capillary strength of the CO_2-brine system through a variation in wetting. The capillarity is consistent with general characteristics of drainage in strongly water-wet rocks. The fluid distributions were observed using x-ray computed tomography and the spatial saturations were investigated and were found to be invariant with different reservoir conditions in homogeneous samples.

INTRODUCTION
While recent contact angle measurements have shown CO_2 to generally act as a non-wetting phase in siliciclastic rocks, some observations report significantly different

wetting behaviour, if not entirely contradictory results, with regard to pressure, temperature and water salinity of CO_2-brine-rock systems. Additionally, there is a wide range of reported contact angles for this system, from strongly to weakly water-wet. In the case of some minerals, intermediate wet contact angles have been observed. Using contact angle measurements on smooth crystal surfaces, [1] found that the CO_2-brine system was strongly water-wet on quartz and calcite with no impact on pressure and a small shift with salinity up to 3.5M NaCl. [2] have also observed the system to be water-wet on quartz with little impact of both pressure and salinity from atmospheric to 10 MPa and 0.01-1M NaCl solutions. In the case of mica, however, the system shifted from water wet to intermediate wet with increasing CO_2 pressure (> 10 MPa). Water wettability was reduced by 25° with salinity change from 0.1 to 1 M NaCl. However, the results at 0.01 M NaCl didn't obey the trend. [3] have found that contact angles on silica increased up to 17.6° ± 2.0° as a result of reactions with supercritical CO_2. The observed increase occurred primarily within the pressure range 7−10 MPa, but remain nearly constant at pressure greater than 10 MPa. They also observed that the contact angle increased with ionic strength nearly linearly with a net increase of 19.6° ± 2.1° at 5.0 M NaCl. Pressure had only a minor influence on the ionic strength effect. [4] have observed the system to be water-wet on quartz with contact angle <30° for both low and high temperature and pressure (T=30° P=7 MPa vs. T=50° P=20 MPa). However, they observed that the contact angle decrease with ionic strength for both conditions. In microfluidic experiments with silica micromodels, [5] observed contact angles ranging from water-wet to mixed wet for the supercritical CO_2-brine system as the salinity is increased from 0.01-5M NaCl.

In this study, we make several primary drainage (CO_2 displacing brine) capillary pressure measurements of at different reservoir conditions to investigate the effective wettability during CO_2 injection process that can last for decades in CCS projects. This process is a very important if not the most critical stage in CCS projects for safe CO_2 injection. A solid and clear understanding of the wettability of the system is essential for proper reservoir management and simulation of multiphase flow (CO_2 plume) during CO_2 injection. Figure 1 show that capillarity and multiphase flow effects may or may not be important depending on the rock properties of the storage target and thus it can be important to understand these properties in some detail. All of those graphs, however, were scaled from the mercury-air system assuming that the CO_2-brine system is strongly water wet. This assumption presumes maximum capillarity in the system. If the system is in fact less strongly water wet this manifests as a weakening of capillarity. As the system becomes less wet with respect to a given phase, it matters less and less what the particular pore structure looks like, the multiphase flow effects will be largely nullified.

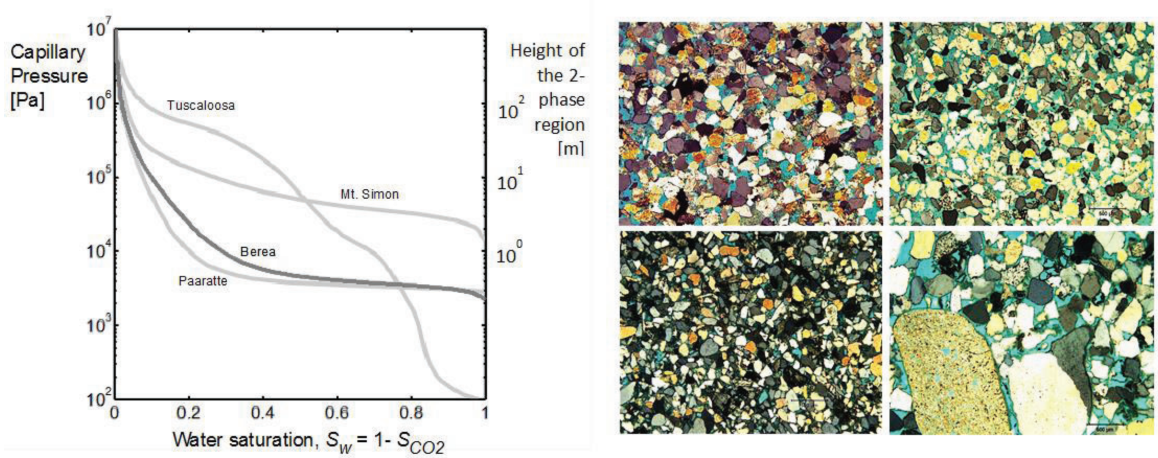

Figure 1. Reproduced from [6] showing the impact of capillarity on the height of 2-phase region. On the left, capillary pressure functions reproduced from plotted from observations with mercury-air displacement (MICP) scaled to interfacial tension values relevant to the CO_2-brine system. The Pc was converted to height to demonstrate the height of the 2-phase region in a CO_2 plume for different rock types. On the right, thin sections under cross-polarized light to compare grain size and distribution of the four sandstone rock types showing in the Pc curves graph. Blue die is used to visualize the pores. Clockwise from upper left: Berea, Paaratte, Tuscaloosa, Mt. Simon.

Figure 2 shows two end member examples reproduced from the model developed in [7, 8] of a buoyant CO_2 plume migrating upwards and along an impermeable boundary. Weak capillarity results in a very narrow range in the plume over which the saturation varies, corresponding to a relatively compact and fast moving plume. On the other hand, strong capillarity results in a much larger range over which the saturation varies, and an overall larger plume volume for the same mass of fluid and thus a more slowly migrating system. Once the injection phase is completed, imbibition plays a major role in CO_2 residual trapping. The impact of different reservoir conditions on residual trapping and hysteresis is investigated on the same Berea sample and conditions and discussed in details in earlier work [9].

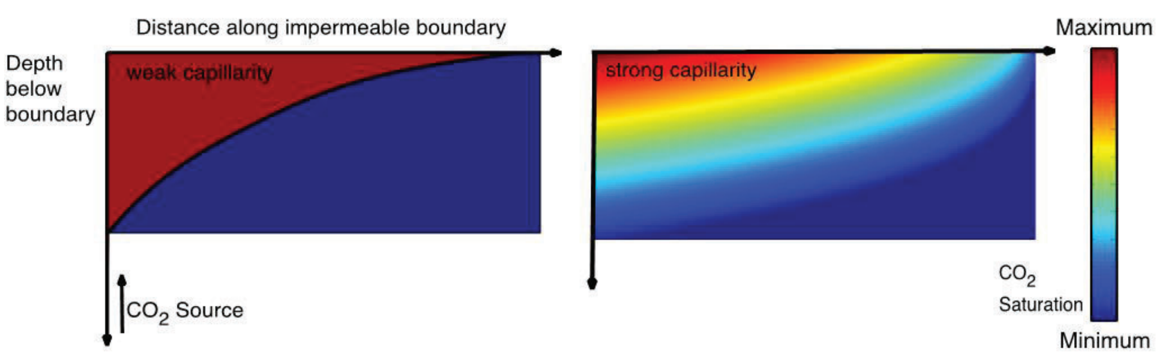

Figure 2. Two end member examples reproduced from the model developed in [7, 8] of a buoyant CO_2 plume migrating upwards and along an impermeable boundary.

The measurements of this work uses X-ray CT imaging in a state of the art core flooding laboratory designed to operate at high temperature, pressure, and concentrated brines.

This is the first study looking systematically at the impacts of reservoir conditions on the effective wettability in the CO_2-brine-sandstone system in a single rock sample. We also make measurements using N_2 and water to assess the ability to use analogue fluids to represent CO_2 and brine systems and developed a method to quantify shifts in effective wetting properties with changing reservoir conditions. We find no significant impact on wetting during primary drainage within the range of reservoir and flow conditions relevant to CO_2 storage, consistent with traditional multiphase flow theory but despite observations by others suggesting that wetting properties and multiphase flow in this system are sensitive to pressure, temperature and brine salinity [10]. In this paper, we report some of the results showing the impact of pressure on effective wettability and on spatial saturations distributions.

MATERIALS AND EXPERIMENTAL CONDITIONS AND SETUP

The Rock Sample and Fluids

A single homogeneous 20.32 cm long, 3.81 cm diameter Berea sandstone core sample was used for the entire tests performed in this study. Berea sandstone is an outcrop rock from the United States which is widely used in studies as a benchmark and for its utility in making comparisons with other studies. The sample was fired at 700 ^oC for 4 hours to prevent fines migration by stabilizing swelling clays that otherwise would cause permeability changes during core flooding tests. The faces of the core were machined flat to ensure good contact with the end-caps. The sample was vacuum dried in air at 70 ^oC overnight before each test. The absolute permeability of the sample to water was 212 mD with an average porosity of 21% as measured by x-ray CT scanning by following a protocol described in [11]. The skeletal density of the sample is 2.601 g/cm^3 measured with Helium pycnometer (AccuPyc 1330, Micromeritics) at 24 ^oC and 20 psi on a sub-sample (~ 0.8 cm^3) that was cut from a section adjacent to the inlet face of the core sample. Carbon dioxide and Nitrogen were used as the non-wetting phases in the core-flooding experiments both with 99.9% purity (BOC Industrial Gases, UK). The wetting phase fluids used were deionized water or brine. The brine solutions were made of deionized water and NaCl with total salt molality ranging from 0 to 5 mol kg^{-1}. In this paper, we report the capillary pressure experimental results investigating the impact of pore pressure on wetting properties and CT x-ray images. The fluids interfacial tension values as well as density and viscosity ratios for the experimental conditions are summarized in Table 1. Experiment N. 4, as described in the table was the only experiment where the wetting phase solution was deionized water doped with 5 wt% Sodium Iodide. This was necessary to compare and quantify the saturation distribution for the high density CO_2 experiments as will be discussed in a later section.

Table 1. Experimental conditions and fluids properties of experimental results investigating the impact of pore pressure on wetting properties and CT x-ray images

Experiment number	Non-wetting fluid	Wetting fluid	*Pressure* [Mpa]	*Temperature* [C]	σ^{ab} [mN/m]	μ_w/μ_{nw} [cd]	ρ_w/ρ_{nw} [cd]
1	gCO_2	H_2O	5	50	47	32	9.5
2	$scCO2$	H_2O	10	50	36	19	2.6
3	$scCO_2$	H_2O	20	50	30	8	1.3
4	$scCO_2$	H_2O with 5wt% NaI	20	50	-	8	1.3
5	N_2	H_2O	10	25	67	45	9

[a] CO_2 IFT from correlation developed in [12], [b] N_2 IFT from [13], [c] CO_2 densities and viscosities from NIST chemistry web book [14], [d] Water and brine densities and viscosities from [15]

Core-flooding Experimental Setup

The experimental work was conducted using a state of the art multi-scale imaging laboratory (core and pore scale) recently developed at Imperial College London designed to characterise reactive transport and multiphase flow, with and without chemical reaction for CO_2-brine systems in both sandstone and carbonate rocks at reservoir conditions [16]. The experimental setup is designed to replicate in-situ conditions of up to 120 ^oC and 30 MPa. Details of the experimental apparatus are reported in [11].

METHODOLOGY

Core-flooding was performed for capillary pressure measurements. Total injection flow rates varied from 0.5 up to 50 mL min^{-1}. The dimensionless capillary number, $N_c = V\mu/\sigma$, was used to ensure that local capillary equilibrium conditions applied during all of the experimental flow rates. Where V [m/s] and μ [Pa s] are the CO_2 superficial velocity and dynamic viscosity, respectively, σ [N/m] is the interfacial tension between CO_2 and brine. Capillary numbers around 10^{-4}-10^{-5} indicate that viscous and capillary forces are equivalent for the reservoir rock [17]. In this study, the capillary numbers for the highest flow rates applied ranged between 10^{-7} to 10^{-6} and the pore-scale fluid distribution was thus controlled by capillary forces.

We used the semi-dynamic capillary pressure method to measure the capillary pressure in the whole core. The technique was developed by [18] based on a model proposed by [19] and has been recently applied to measurements in CO_2-brine systems by [20, 21]. The technique applied here is described in [11] but we do not fit MICP to measurements for effective contact angle estimation. We use a more practical reference know to be strongly water-wet to evaluate wetting strength. We do this by measuring the semi-dynamic Pc of N_2-water on the same core sample and use it as a benchmark. The technique was developed further in this work and the main modifications were (1) the use of a spacer at the downstream end of the core to control the outlet boundary condition and (2) the use of a numerical simulator to design the parameters for the test, e.g., the injection flow rate. Brooks-Corey curves were fit to the core-flooding capillary pressure measurements [22].

$$P_c = P_e \left(\frac{S_w - S_{w,irr}}{1 - S_{w,irr}}\right)^{-1/\lambda} \tag{1}$$

Where Pe [Pa] is the entry pressure (i.e. the minimum pressure required for the entry of CO_2 into the pores of the rock), S_w represent the water saturation, while $S_{w,irr}$ represent the irreducible water saturation, λ is a fitting parameter known as the pore size distribution index.

Experimental Procedures

The first step was to expel air from the flow loop by flowing CO_2 through all parts of the experimental setup at an elevated pressure and exhausting to the atmosphere. This was done at different stages by isolating different sections of the flow loop to insure that air is expelled entirely. Then the system was pressurized with CO_2 and heated to the experimental pressure and temperature. This step was required to obtain the CO_2 saturated core background scan. Next, water or brine, depending on the experimental fluid, was injected into the loop to displace and dissolve all of the CO_2 out of the core at experimental pressure and temperature. A scan of the brine saturated core was obtained for porosity measurements. Porosity and saturations calculations with full experimental procedures for absolute permeability, fluids equilibration and semi-dynamic capillary pressure are described in details in earlier work [11].

RESULTS

A stable pressure drop was achieved for each CO_2 injection flow rate applied for semi-dynamic Pc before taking x-ray scans to measure saturations. The horizontal and vertical error bars of the Pc measurements represent uncertainty caused by pressure fluctuations during X-ray imaging time and saturation uncertainty based on the range of saturations seen from 10 repeated scans, respectively. The measured Pc curves were fitted with Brooks-Corey model, Eq. (1), objectively using a MatLab code. A single value was assigned for λ as a single rock sample was used for the entire study.

The Impact of Reservoir Pressure on Wettability

To evaluate the effect of pressure on wettability three experiments were carried out at pressures of 5, 10 and 20 MPa pore pressure at 50^{o}C and 0 Mol kg^{-1}. The pressures range such that CO_2 phase is transitioning from gas to low density scCO_2 and finally to high density scCO_2. With increasing pressure, the interfacial tension between the fluids decreased from 47 to 30 mN/m, Table 1. This was reflected in the capillary pressure and observable in the measurements with a lower entry pressure as the interfacial tension decreases, Figure 3a. Each Pc curve was scaled by its respective interfacial tension to allow for seeing if there is a weakening of the capillarity of the system through a change in wettability, Figure 3b. The scaled data set collapse nicely suggesting no change in the wettability with regard to pressure.

Analogue Fluids

A core-flood was performed using N_2 and deionized water at lab temperature, 25^{o}C, and 10 MPa pore pressure for the purpose of comparison with a different fluids pair. The N_2-water system experiment is quicker and much easier than CO_2-brine experiments. Carbon dioxide-brine system is corrosive fluids pair and requires the entire wetted parts in the experimental apparatus to be anti-corrosion such as Hasteloy and Titanium. The solubility of CO_2 and N_2 at 25^{o}C and 10MPa are 31.75cm^3/g (cm^3 of gas at standard conditions per gram of water) [23] and 1.264cm^3/g [24] respectively, i.e., the solubility of N_2 is more than 25 times less than CO_2. Therefore, N_2 equilibrates with water much faster compared to CO_2. The thermophysical properties of N_2 at 25^{o}C and 10MPa were similar to CO_2 at 5MPa and 50^{o}C, as shown in Table 1. Nitrogen and CO_2 capillary pressure curves scaled by their respective IFT are similar with slight shifts with regard to wettability as discussed in previous sections. Therefore, it is possible that the capillary pressure for CO_2-brine in sandstone to be estimated using N_2-water fluids pair particularly where the experimental apparatus for handling CO_2-brine systems is not readily available.

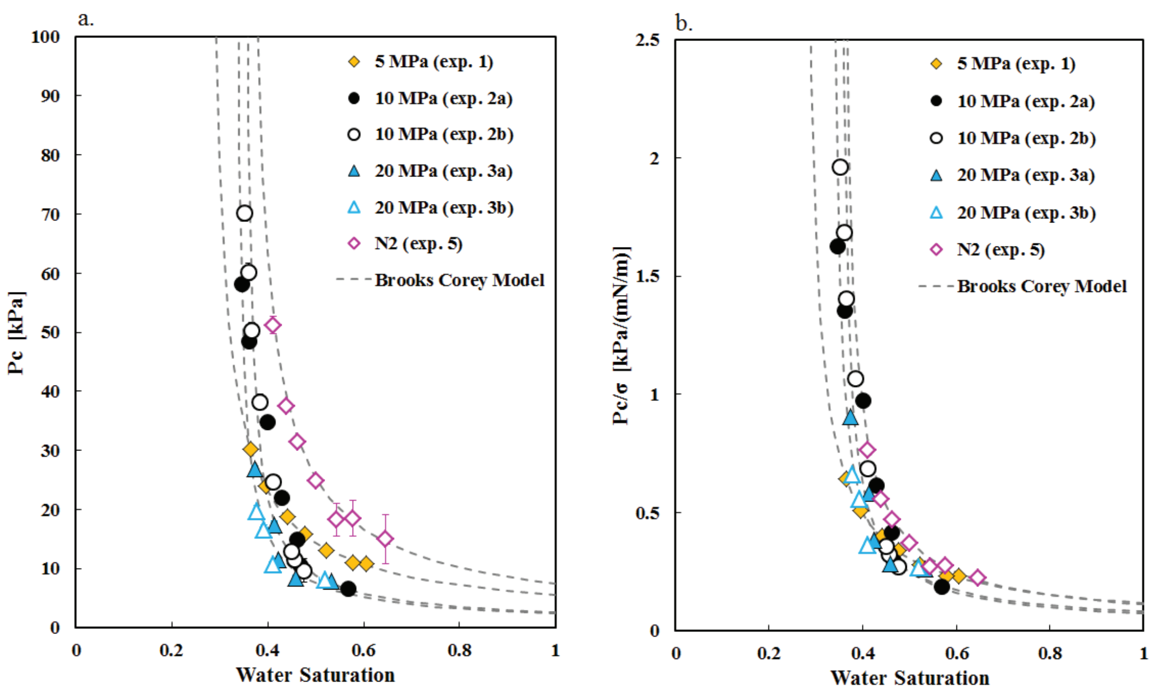

Figure 3. [10] a. Core-flood capillary pressure measurements of CO_2-water system at 50 ^{o}C, 0 mol/kg and pressures ranging from the gas phase (5 MPa) to low density $scCO^2$ (10 MPa) to high density $scCO_2$ (20 MPa). Nitrogen-water capillary pressure was included for comparison and to show the effect of IFT on Pc as measured by semi-dynamic technique. The repeats demonstrate the reproducibility of the semi-dynamic Pc measurements performed in this study. b. The Pc curves were scaled by IFT values representative of the experimental conditions, Table 1, and show no significant change in wettability with regard to pressure.

CT Images Accuracy as a Function of Fluids Density Ratio

Fluids saturations were measured in-situ by CT scanner using a combination of experimental and background scans as described in a previous section. This method depends on the contrast in density between the imaged phases (rock, wetting and non-wetting fluids). The density ratio between the wetting and non-wetting fluids varies depending on the experimental conditions as shown in Table 1. Thus, CT image quality is dependent on the density ratio of the experimental fluids and the noise associated with CT images can affect the corresponding reconstruction of 3-D saturation map. Depending on the significance of noise, a false fluids saturation distribution can be measured without proper coarsening. Figure 4 shows the saturation distribution of experiments 1 to 4 where CO_2 phase transfers from the gas phase to $scCO_2$ and finally to high density $scCO_2$, the ρ_w/ρ_{CO_2} range from 9.5 to 1.3. Experiment 4 was made at the same condition as 3 (50^{o}C, 20 MPa) with the exception of doping the water with 5 wt% Nal to enhance the contrast between the fluids for the CT image.

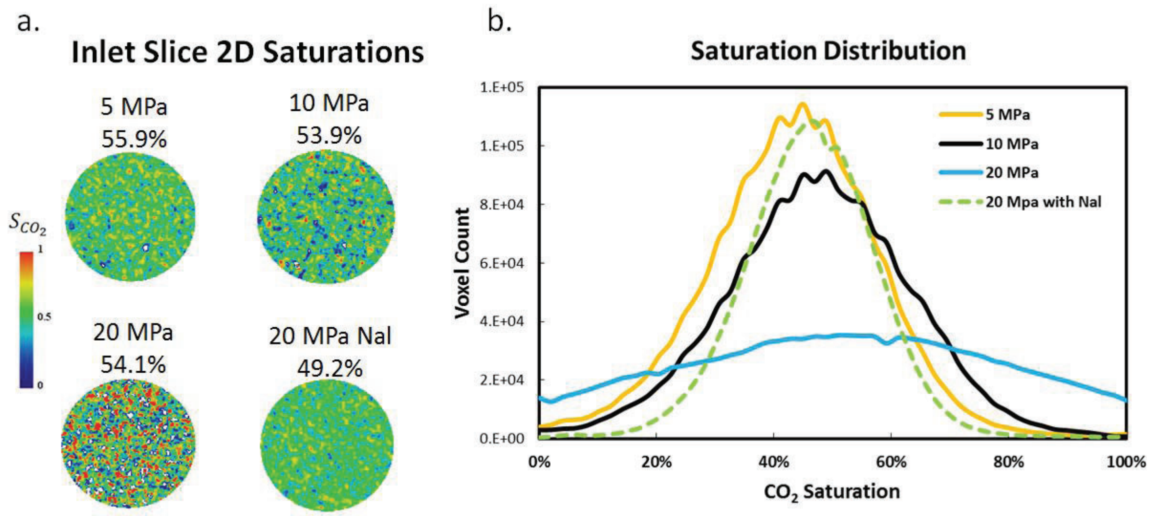

Figure 4. a. 2D saturation map of the inlet slice for 4 different experiements examining the impact of pressure on multiphase flow properties. The 2D slices have similar average saturations. b. Saturation distributions of the 2D inlet slices are directly proportional to experimental pressure and as CO_2 becomes denser. This is not representing insitue saturations but rather noise in CT measurements due to low water/CO_2 density ratio. This was confirmed by observing the saturation distribution at the high density CO_2 experiment after dopping water with Nal.

This test proves that the actual saturation distribution in the core sample remains unchanged and that the difference in the saturation distribution between experiments 1 to 3 is mainly due to the noise associated with lower density contrast. This noise has random nature and is irrelevant to the experimental conditions, as shown in Figure 5a. By subtracting two CT images at same conditions and location, the noise at voxel by voxel level was obtained. Usually, the noise has normal distribution and no relationship with fluid density. The uncertainty on calculated saturation induced by random noise for CO_2-brine system can be calculated by [21].

$$\sigma_S = \frac{\sigma_\Delta}{CT_{brine} - CT_{CO_2}} \sqrt{1 + \left(\frac{CT_{brine} - CT_{brine+CO_2}}{CT_{brine} - CT_{CO_2}}\right)^2} \quad (2)$$

where σ_S is the uncertainty on saturation, and σ_Δ is the standard deviation of random noise. The average uncertainty at various experiment ranges from 0.115 to 0.379, which is large enough to influence the comparison of saturation maps at different conditions. Therefore, different coarsening schemes have been applied to images from 1x1 to 15x15, as shown in Figure 5b. With increasing degree of coarsening, the level noise can be reduced. Therefore, the images at different experimental conditions have been processed with proper coarsening scheme before the reconstruction of saturation maps. By using the above approach, the range of the average uncertainty on saturation at various experiments has been reduced to 0.115~0.129, which is acceptable for the comparison between saturation maps.

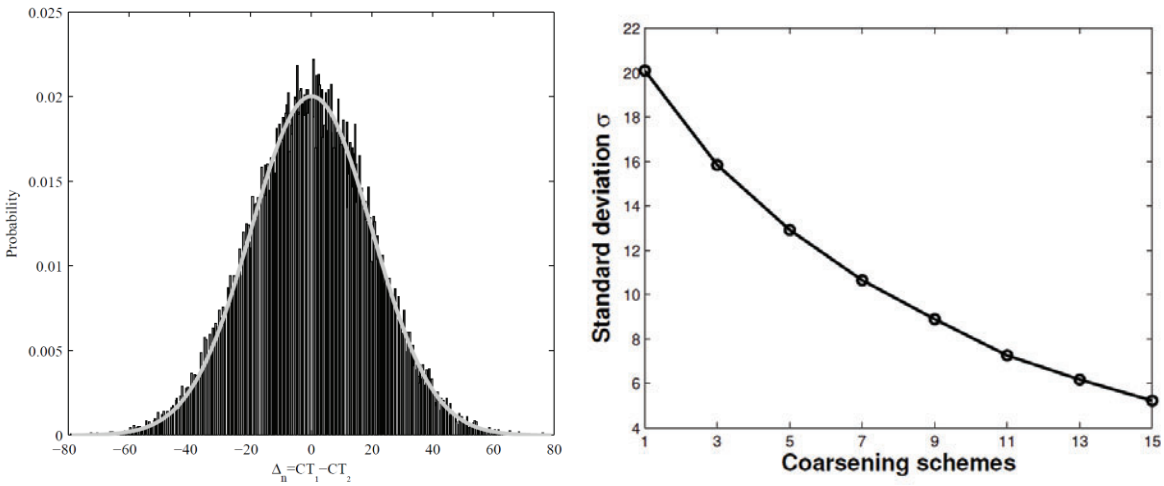

Figure 5. a. A histogram of CT numbers obtained by taking the difference of two scans of the same location. Various shades of grey represent difference values obtained at various conditions of pressure and temperature but they are indistinguishable indicating that the nature of the noise was intrinsic to the x-ray scanner. From light to dark the histogram bars are taken from: CT images of CO_2 saturated core at 5MPa-50°C, 10MPa-25°C, and 20MPa-50°C respectively, Grey line: fitted normal distribution curve with mean value μ = 0.04 and standard deviation σ_Δ = 19.9. b. Random noise vs. coarsening schemes.

Quantify the impact of density ratio on error from image noise is crucial for studies targeting voxel resolution saturations from CT x-ray scanning. In this study, coarsening the images is not necessary for obtaining accurate saturation values as the slice average saturation remains unaffected. This analysis was made to inspect dissolution or gravity segregation and assure that the experiments were performed as designed by visualizing comparable images with less noise. This was also useful to confirm that the saturation distribution remains unaffected with different experimental conditions.

CONCLUSION

Wettability determines the efficiency of enhanced oil recovery operations as well as our ability to inject and store CO_2 in geological formations. This study investigated the effective core-scale wetting properties at a wide range of reservoir conditions to observe the impact of pressure, temperature and brine salinity on the wetting properties of the CO_2-brine system in siliciclastic rocks during primary drainage. Core-flood semi-dynamic capillary pressure were measured to observe the impact of different reservoir conditions on effective wettability from observations of multiphase flow properties. This allowed for measuring the core-scale effective and dynamic wetting properties representative of CO_2 injection processes such as EOR and carbon storage. No significant change in capillarity observed with regard to pressure even with different CO_2 phases (gaseous and supercritical). The saturation distribution of the fluids remained similar within the core sample with varying water/CO_2 density ratios. This was confirmed by a repeat of high density supercritical CO_2 and doped brine measurement. The error associated with noise from density ratio has to be considered in studies looking into voxel size saturations from CT imaging or spatial saturations distribution and up scaling resolution is advised. Analogue fluids can be used for characterising multiphase flow properties when reservoir conditions cannot be replicated. In general, reservoir conditions are not having a major impact on the wettability of CO_2-brine-sandstone system during CO_2 injection process and the system remains strongly water-wet during primary drainage for the experimental conditions investigated in this study.

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