Research Article

Characterizing the Evolution of Trapped scCO₂ Curvature in Bentheimer and Nugget Sandstone Pore Geometry

Laura E. Dalton,1,2 Dustin Crandall,1 and Angela Goodman3

1U.S. Department of Energy’s National Energy Technology Laboratory, Morgantown, WV, USA
2Leidos Research Support Team, Morgantown, WV, USA
3U.S. Department of Energy’s National Energy Technology Laboratory, Pittsburgh, PA, USA

Correspondence should be addressed to Laura E. Dalton; laura.dalton@netl.doe.gov

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During a Geologic Carbon Storage process, supercritical CO₂ (scCO₂) is subjected to a series of dynamic and static conditions where the relationship between pore geometry and the trapped scCO₂ curvature remains to be established. To mimic the dynamic process, two sandstones, Bentheimer and Nugget, were subjected to two successive drainage and imbibition (D-I) cycles and X-ray computed tomography scanned at each residual state to capture the wettability evolution at static conditions in the same pore geometry. Both sandstones contain similar grain size distributions, pore size distributions, and pore interconnectivity but differ in that the Nugget formation contains approximately half the porosity of the Bentheimer sandstone, and the pore network contains dead-end pores. scCO₂ size distributions, strain calculations, and geometric contact angle measurements were used to characterize the curvature of scCO₂ in different pore types between cycles. An increase in geometric contact angle was the greatest when advancement along the pore network of the same ganglion occurred between cycles while strain increased the most with pore-filling trapping. Moreover, Nugget sandstone results in a greater aggregated residual saturation and shows a clear increase in scCO₂ sizes with an additional D-I cycle while scCO₂ in the Bentheimer core shows a more complex response with some ganglion increasing and some decreasing in size with an additional D-I cycle. From this work, we suspect the pore geometry is playing a role in scCO₂ size distributions and use this information to suggest using water pulses to enhance trapping capacity in lower porosity sandstones.

1. Introduction

Capillary trapping acts to store supercritical CO₂ (scCO₂) in saline aquifers via two mechanisms: snap-off and pore filling through coalescence of the nonwetting fluid [1]. While it is known that pore geometry controls the curvature of the trapped scCO₂ ganglia in any direction [2], the relationship between complex pore geometry and trapped curvature is not well established. Theoretically, scCO₂ becomes trapped in the center of large pores as the plume migrates through the pore network (Reynold, 2017; [3]) and remains stagnant until dissolution into the resident brine occurs. In field applications, mechanisms such as scCO₂ snap-off and coalescence result in temporal alterations to the fluid pathways that do not result in instantaneous trapping but instead are a continuous process evolving with time [1].

Experiments designed to represent drainage (injection of CO₂ nonwetting phase) and imbibition (injection of brine wetting phase) at the microscale have been used to characterize and quantify pore-scale multiphase fluid interactions in Geologic Carbon Storage (GCS) environments utilizing various methods [4–8]. Experimental and visualization techniques have advanced to enable the imaging of in situ geometric contact angle (θg) measurements of scCO₂ [2, 9–13] and temporal changes during the dynamic snap-off and pore-filling processes [14–18]. Moreover, Noiriel et al. [19–22] have studied pore-scale flow and permeability alterations to pore geometry by modeling the impacts of precipitated material, an added complexity inherent to pore networks in the presence of CO₂, as a function of time.

The primary mechanism controlling residual trapping has been studied at different scales (micro, meso, macro,
field, etc.) among geologic formations and remains disputed—particularly in sandstones which are highly heterogeneous in nature. One thing is certain—fluids do not remain stagnant indefinitely and continue to rearrange with time after a GCS injection. This study is aimed at analyzing the evolution of trapped scCO₂ curvature in the pores of two sandstones with differing pore structures. GCS is mimicked by subjecting sandstone cores to two successive drainage–imbibition (D-I) cycles to analyze how dynamic multiphase flow influences the curvature of trapped scCO₂ at static conditions. Microcomputed tomography (CT) scanning is used to capture each static residual saturation after each D-I cycle. The images of the static trapped scCO₂ are used to characterize scCO₂ curvature in relation to the pore geometry using strain calculations and measuring θ₂ in different pore types between cycles.

2. Materials and Methods

A simplified methodology was implemented for this study. Two sandstone cores, Bentheimer and Nugget, were selected for analysis because both formations have been utilized at the reservoir-scale [3] and both provide pore spaces well-resolved at the highest fidelity scanning capabilities (1.66 μm/voxel) available with the micro-CT scanner at the National Energy Technology Laboratory. Each core was subjected to two D-I cycles at identical conditions (confining pressure = 14 MPa, temperature = 45°C) to reach in situ conditions. The system was equilibrated for a total of 24 hours before the series of D-I injections and micro-CT scans. Ten pore volumes of each fluid were injected for each drainage (Figures 1(b) and 1(e)) and imbibition (Figures 1(c) and 1(f)) flow. CT scans were collected in Figures 1(d) and 1(g) steps using identical parameters (2700 projections and 1.66 μm/voxel resolution). Identical subvolumes were cropped from each sandstone scan to track the evolution within identical pores. This data is available on the Digital Rocks Portal [23] for additional analyses. Grayscale cross-sections perpendicular to injection and corresponding 3D volumes of the trapped scCO₂ from D-I cycle 1 are shown in Figures 2(a)–2(d) and D-I cycle 2 in Figures 2(e)–2(h). The various colors in the 3D volumes represent individual trapped ganglion. For additional information on the Xradia MicroXCT-400 scanner and fluid injection system, refer to Tudek et al. [13] and Dalton et al. [10].

2.2. Analysis Procedure. Innova-X® Handheld X-Ray Fluorescence Spectrometer was used to measure relative elemental abundance of the samples [24]. Residual saturation, pore size distribution (PSD), grain size distributions (GSDs), and scCO₂ size distribution (SSD) of the entire cropped subvolumes were analyzed using CT image processing software equivalent spherical diameter (ESD; FEI 2017) measurements. ESD is defined as \(\sqrt[3]{(6 \times \text{Volume})/\pi}\) (FEI 2017; [25, 26]) where Volume is the volume of an individual scCO₂ ganglion. Each was plotted and compared between sandstones. Thirty ganglions were randomly selected from each subvolume and individually cropped for improved local segmentation prior to using Blob3D [27] to obtain the three axes (minimum, intermediate, and maximum) required to calculate strain using EllipseFit 3.5.2 [28] and Hsü-Nadai plots [10, 25]. Hsü-Nadai plots use the three principal axes of the 3D scCO₂ volume to determine total strain (\(\varepsilon\), radial coordinate axis) and normal strain (\(\nu\), polar coordinate axis). These plots allow the comparison of strain magnitude
and the measure of symmetry [28]. Symmetry expresses variations using relative ratios of the three principal axes which distinguish prolate (cigar-shaped) or oblate (pancake-shaped) ganglion.

Lastly, geometric contact angle (θ<sub>g</sub>) measurements were completed on three ganglions following the methodology described by Andrew et al. (2014). A detailed analysis on θ<sub>g</sub> measurements between D-I cycles for a single ganglion located in the center of a regular pore from each sandstone was completed. These are referred to as Bentheimer pore and Nugget pore throughout the remainder of the paper. Additionally, the influence of dead-end pores in the Nugget core was evaluated; dead-end pores were not present in the Bentheimer subvolume evaluated (possibly the entire core). It should be noted that identifying dead-end pores was completed through visual observation. It is possible the observed dead-end Nugget pore used for analysis is connected by a subresolution (1.66 micron/voxel) pore throat; however, this is a limitation of the analysis and is termed Nugget dead-end pore throughout this paper.

### 3. Results

#### 3.1. X-Ray Fluorescence

Mining-Plus Suite utilizes a two-beam X-ray analysis that captures major elements (Mg, Al, Si, P, S, Cl, Fe, K, Ca, and Ti), minor elements (V, Cu, Ni, Cr, Mn, and Pb), trace elements (Co, Zn, As, Zr, Mo, Ag, Cd, Sn, Sb, Hf, W, and Bi), and an aggregated “light element” (H to Na). The results were taken at 6 cm resolution for 60-second exposure time per beam which was run on each sandstone sample and reported relative to the total elemental composition in Figure 3 for comparison. Most of the elements detected are comparable in concentration between the two sandstones. The most notable deviations are the calcium, zinc, and aluminum concentrations. Nugget sandstone contains a high concentration of calcium while Bentheimer contains no traces of calcium. Zinc and aluminum contents are slightly elevated in the Nugget sample. Clay is primarily comprised of alumina silicate suggesting a slight increase in clay content which may be present in the Nugget compared to the Bentheimer sandstone pore network.

#### 3.2. Residual Saturation

With successive D-I cycles, an increase in residual saturation occurred in each sandstone core (see Figure 2 and Table 1). In general, after each cycle, higher percent residual saturation was observed in the Bentheimer core (6.4% for cycle 1 and 16.5% for cycle 2), as compared to the Nugget sandstone (1.8% for cycle 1 and 12.4% for cycle 2) (Table 1). This is also evident from the visual representations of the 3D volumes of trapped scCO<sub>2</sub> in Figures 2(b) and 2(f) for Bentheimer and Figures 2(d) and 2(h) for Nugget. In addition, the aggregated increase in scCO<sub>2</sub> between cycles in the Nugget core was more substantial when compared to the Bentheimer core (Table 1). An increase in residual saturation between D-I cycles is consistent with past literature [6]; however, residual saturations for D-I cycles measured in this work are approximately half the residual saturations reported by Herring et al. [6] for a Bentheimer core. Herring et al. [6] performed experiments at 8.3 MPa pore pressure, 12.4 MPa overburden pressure, and 38°C and waited for the system to reach a specific pressure before injecting 30 pore volumes for imbibition to reach residual conditions. In this work, experiments were conducted at 12.4 MPa pore pressure, 14.5 MPa overburden pressure, and 45°C, and 10 pore volumes were used for

![Figure 2: D-I cycle 1: (a) Bentheimer 2D cross-section, (b) Bentheimer scCO<sub>2</sub> 3D volume, (c) Nugget 2D cross-section, and (d) Nugget scCO<sub>2</sub> 3D volume; D-I cycle 2: (e) Bentheimer 2D cross-section, (f) Bentheimer scCO<sub>2</sub> 3D volume, (g) Nugget 2D cross-section, and (h) Nugget scCO<sub>2</sub> 3D volume. The different colors represent separate scCO<sub>2</sub> ganglion.](image-url)
drainage and imbibition injections. Moreover, looking at the residual condition images in Herring et al. [6] the images suggest pore filling was the primary trapping mechanism whereas snap-off was the primary mechanism in this study. The slightly lower residual saturation in this study is likely a result of the lower number of pore volumes used.

### 3.3. Pore, Grain, and scCO$_2$ Size Distributions.

PSDs, GSDs, and each SSD per cycle are plotted for each sandstone sample in Figure 4 where the length-scale volume identifier is ESD on the $y$-axis. To simplify, volume will be used for the following explanation. PSDs and GSDs are similar in scale and count between sandstones. The main difference between the two sandstones is in the SSDs. Some ganglion volumes exceed the largest pore volumes in the Bentheimer core while ganglion volumes in the Nugget pore network (Figure 4(a)) are consistently smaller than the pore space available. The largest trapped ganglion present in the Bentheimer core reduced in size between cycles. More notably, the SSD curves and the changes between D-I cycle 1 and D-I cycle 2 are different between sandstone formations. Each sandstone follows the same general shape between D-I cycles, but in the Bentheimer sandstone, trapped scCO$_2$ has a significant increase in ganglion sizes $1E + 4$ to $1E + 6 \mu$m. In contrast, the scCO$_2$ ganglion trapped in the Nugget sandstone increases across all sizes.

Figure 2 provides a visual of the evolution of saturation in both sandstone cores. The Bentheimer core initially residually trapped ganglia along limited areas of the pore-network subvolume and after the second D-I ganglion was trapped throughout the entire subvolume (Figures 2(b) and 2(f)). In the Nugget subvolume, large grains were present towards the middle (Figures 2(c) and 2(g)) reducing the available pore space for trapping. Additionally, trapped ganglia are visually smaller compared to those trapped in the Bentheimer pore network particularly after the first D-I cycle. Connectivity analysis [29] was completed on each pore network with Bentheimer resulting in 32,112 and Nugget resulting in 29,773 connected voxels revealing both sandstones (over similar cropped volumes) have similar interconnectivity. Volume measurements do not account for or consider the shape/geometry of an entity. Given similar PSDs and interconnectivity between the two sandstones, this suggests that a microscopic feature not accounted for in the geometric description of the porous structure, such as dead-end pores, may influence residual saturation.

### 3.4. Ganglion Strain.

Deformation of a material depends on the size and magnitude of the applied stress and is measured as a normalized value to avoid scale dependency. For a non-rigid body (i.e., a fluid or gas), deformation results in distortion (change in geometric shape) and/or dilation (change in volume). Here in Figure 5, the three primary axes (maximum, intermediate, and minimum) of thirty scCO$_2$ ganglion volumes from each cycle were measured to quantify the strain using Hsü-Nadai plots [10, 25]. $\varepsilon$ is the amount of strain in the radial coordinate axis, and $\nu$ is the symmetry strain in the polar coordinate axis. A scCO$_2$ droplet in brine under no stress would be a perfect sphere which means each ganglion in its undeformed shape would naturally fall at the zero centerline at the bottom of the Hsü-Nadai plot where zero strain is observed. Pore space will inevitably have a deforming (strained) effect on trapped scCO$_2$, particularly in the case of pore filling, as scCO$_2$ will deform to the shape of its immediate surroundings until snap-off or pore filling occurs.

After D-I cycle 1, all ganglion strains fall below $\varepsilon = 0.5$ or to the left of the centerline above $\varepsilon = 0.5$. No ganglion falls...
within the oblate quadrant above $\varepsilon = 0.5$ until after D-I cycle 2 in both sandstones. Ganglia spanned from prolate (constriction) states to moderate oblate (expansion) strained states. In both sandstones, ganglion strain does not span into the upper right quadrant of the Hsü-Nadai plot meaning the ganglia did not become trapped in an “expanded” state.

Snap-off occurs when a pressure equilibrium is reached where a single ganglion “snaps” apart creating two separate ganglia which retract into separate pores reaching a point of equilibrium [14]. Pore filling occurs through ganglion coalescence eventually “filling” the pore space inevitably deforming the scCO$_2$ to the shape of the surrounding pore.

Figure 4: Pore, grain, and scCO$_2$ size distributions (SSD): (a) Bentheimer and (b) Nugget.

Figure 5: Hsü-Nadai plots: (a) Bentheimer and (b) Nugget.
geometry. A moderate shift towards the centerline is observed between D-I cycles which may or may not continue with additional D-I cycles.

3.5. Geometric Contact Angles ($\theta_g$). The number of grain contact surfaces and the average $\theta_g$ along each surface were manually measured and averaged to characterize the evolution of $\theta_g$ with successive D-I cycles. Figures 6(a)–6(c) are provided as a reference for the contact surfaces. The average contact angle is the combined average of the angles on both sides of the contact surface (Figure 6(a)). From one angle, the trapped ganglion is in contact with six grain surfaces (Figure 6(a)), and from another angle, the ganglion is in contact with four surfaces (Figure 6(b)). In 3D, the trapped ganglion is in contact with a total of six surfaces represented by the different colors in Figure 6(c).

$\theta_g$ ranged between 40° and 50° for the two samples (Table 2). Among the three ganglia evaluated, the average $\theta_g$ increased from one D-I cycle to the next. The ganglion trapped in the dead-end pore remained in contact with only two contact surfaces, and the average $\theta_g$ increased by less than 1°. Both regular pores showed more pronounced differences between cycles. The Bentheimer ganglion evolved from being in contact with six to four surfaces after D-I cycle 2 with a 4° increase in the $\theta_g$ average. The ganglion trapped in the regular Nugget pore remained in contact with five surfaces between cycles but evolved from a snap-off ganglion to a coalesced ganglion. This resulted in an 8° increase in the average $\theta_g$ between cycles.

Identical series of intermittent cropped slices from each pore analyzed between cycles are presented in Figure 7. Figure 7(a) displays the evolution of the trapped ganglion.
Table 2: Detailed pore analyses.

| Sandstone pore             | Trapping type | D-I cycle | Volume (mm$^3$) | Contact surfaces | AVG (°) |
|---------------------------|---------------|-----------|-----------------|------------------|---------|
| Bentheimer pore            | Snap-off      | 1         | 0.00511         | 6                | 41.4    |
| Bentheimer pore            | Snap-off      | 2         | 0.00470         | 4                | 45.5    |
| Nugget pore                | Snap-off      | 1         | 0.00150         | 5                | 41.8    |
| Nugget pore                | Pore filling  | 2         | 0.00532         | 5                | 50.1    |
| Nugget dead-end pore       | Snap-off      | 1         | 0.00105         | 2                | 45.2    |
| Nugget dead-end pore       | Snap-off      | 2         | 0.00111         | 2                | 46.1    |

Figure 7: Bentheimer pore, Nugget pore, and Nugget dead-end pore cross-section views. Red circles highlight high density particles that migrated in the Bentheimer pore between D-I cycles.
between D-I cycles in the Bentheimer pore. The main observations are the original trapped ganglion advanced along the pore network between cycles, in both cycles the ganglion became trapped via snap-off, and high density particles (highlighted by red circles) dislodged and moved through the pore space. Noiriel et al. [19–22] have looked at the precipitation effect on multiphase flow, but the influence of detached particles has not been extensively studied. These particles could result in restricted flow paths leading to localized pressure build up. Additionally, both Nugget pore types are shown in Figures 7(b) and 7(c). The Nugget pore (Figure 7(b)) shows a different transition than the Bentheimer pore. The ganglion became trapped via snap-off after the first D-I cycle and evolved to pore filling after the second D-I cycle. A clear increase in trapped volume is observed which is consistent with Figure 4(b) which shows an increase in all trapped ganglion in the Nugget formation. Similarly, the ganglion trapped in the Nugget dead-end pore resulted in a minor increase in volume but remained trapped via snap-off between both D-I cycles.

4. Discussion

Andrew et al. (2014) reported well-connected pore space fosters larger scCO₂ clusters while poorly connected pore space contains smaller volumes and concluded differences in rock chemistry mattered less to trapping dynamics than differences in pore structure and connectivity. A greater aggregated storage increase was exhibited in the Nugget pore network with an additional D-I cycle while Bentheimer showed greater overall storage (higher porosity). The pore connectivity, PSDs, and ganglion strain developments of each sandstone were similar. The main areas where the two sandstones deviate are in the elemental compositions, trapping mechanisms between D-I cycles, the shape of the SSD curves, and pore types present throughout the pore networks. A primary difference between the elemental compositions of the Bentheimer and Nugget sandstones was the presence of calcium in the Nugget formation. In the presence of water, calcium can form calcium hydroxide (Ca(OH)₂) which in the presence of carbon dioxide and water can form calcium carbonate (CaCO₃). Carbonate reactivity occurs at a rapid rate and has potential to influence storage capacity with time. Moreover, Nugget exhibited an increase in pore-filling trapping after the second D-I cycle compared to Bentheimer which demonstrated primarily snap-off trapping.

Bentheimer SSDs show a distinct increase in the number of ganglion between sized 1E + 4 and 1E + 6 μm between D-I cycles and a reduction in the largest ganglion volumes from the first D-I cycle which exceeded the size of the largest pores. Nugget SSDs show a consistent growth across all ganglion between D-I cycles. Given similar PSDs and interconnectivity between the two sandstones, this suggests something else not accounted for in a volume measurement is contributing to the differences observed. While the PSDs do not show differences between the two sandstones, no dead-end pores were observed in the Bentheimer volume of interest while dead-end pores were present in the Nugget PSD. According to Ruprecht et al. [18] and Krevor et al. [16], snap-off is controlled by pore topography, throat radius, and local fluid arrangement as well as ratio of pore body to pore throat diameter which can either promote or restrict snap-off. The geometry of the pores and throat to pore transitions not accounted for in the analysis may explain the differences observed in the SSDs between sandstone formations.

Figure 8 displays the evolution of each scCO₂ ganglion from the three detailed pore evaluations and the average θₐ along the different contact surfaces. These plots show the scCO₂ trapped in the Bentheimer core (Figure 8(a)) advanced along the pore network, decreased in volume, and initially was in contact with six surfaces which reduced to four surfaces after the second D-I cycle. The average θₐ increased along two grain surfaces of contact and slightly decreased along the other two contact surfaces appearing to reach a new state of equilibrium where the strain slightly decreased after the second D-I cycle. With the scCO₂ migration, the immediate surrounding pore geometry changed and thus a change in θₐ along the advancing and receding ends occurred. The ganglion trapped in the Nugget pore (Figure 8(b)) increased in volume after the second D-I cycle. Five surfaces of contact were present between cycles with θₐ remaining constant along two surfaces and increasing along the remaining three surfaces. The pore-filling occurrence increased the local strain while the Nugget dead-end pore (Figure 8(c)) remained consistent in volume, number of contact surfaces, and the average θₐ along each surface remained unchanged. These observations show how changes in the trapped scCO₂ curvature can influence continued migration or promote more permanent residual trapping.

5. Conclusions

Two sandstone cores, Bentheimer and Nugget, were subjected to two D-I cycles at identical in situ conditions (45°C, 12.4 MPa pore pressure, and 14.5 MPa confining pressure) and micro-CT scanned at each residual saturation to capture the evolution of wettability in the pore geometry. Four primary deviations between the pore formations were observed: porosity, primary trapping mechanism between D-I cycles, SSDs, and pore types present in the pore networks. The main conclusions from this work are as follows:

(1) While the Nugget sandstone contains lower porosity than the Bentheimer sandstone, a greater aggregated residual saturation was observed in the Nugget sandstone with one additional D-I cycle. This suggests additional water pulses may increase the trapping efficient in lower porosity aquifers

(2) Ganglia trapped in the Nugget sandstone uniformly increased in size between D-I cycles while the largest ganglia trapped in the Bentheimer core decreased and some ganglia increased between D-I cycles. Both sandstones contain similar PSDs and pore interconnectivity with one primary difference being the presence of dead-end pores in the Nugget sandstone and
absence in Bentheimer sandstone. This suggests local pore geometry is playing a role in the SSDs between sandstones.

(3) θ_g increased between cycles with greater changes observed in regular pores compared to dead-end pores while strain rates decreased in snap-off and increased in pore-filling trapping. An increase in θ_g was the greatest when advancement of the same ganglion occurred between cycles while strain increased the most when pore-filling occurred. These findings suggest an increase in strain does not result in an increase in θ_g.
It is possible sending periodic pulses of water with a CO₂ injection may result in increased residual saturations, particularly in lower porosity sandstones, and might be a way to mitigate the spread of CO₂ from an injection well. Two sandstone formations were analyzed in this work, and additional processes may have at the macroscopic scale.

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