High-Resolution X-Ray Computed Tomography of Liquid CO₂ Permeation in Water-Saturated Sandstone*

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Abstract

Geological sequestration of carbon dioxide (CO₂) is an effective method to achieve a substantial reduction in CO₂ emissions to the atmosphere at a relatively low cost; however, the migration process of CO₂ in deep saline aquifers has not been clarified. In order to evaluate the storage site and assess the CO₂ leakage risks and storage costs, fundamental visualization and study of immiscible two-phase flow in sandstone are required. This study observed the behavior of liquid CO₂ injected into water-saturated Berea sandstone by using microfocus X-ray computed tomography with high spatial resolution. The three-dimensional CO₂ distribution in the sample was clearly reconstructed. The bedding structure of the rock strongly determined the CO₂ permeation process, and a strong correlation was seen between the local porosity of the sample and the CO₂ saturation. The real time CO₂ permeation process was also observed in the transparent images that showed the CO₂ gradually permeating the rock in the axial direction with increased saturation in the higher porosity beddings. The effects of the sandstone micro-heterogeneity on the behavior of the injected liquid CO₂ are discussed.

Key words: CO₂ Geological Sequestration, Liquid CO₂, Porous Media, Two-Phase Flow, X-Ray Computed Tomography

1. Introduction

Since the industrial revolution, human activities have led to increasing atmospheric concentrations of carbon dioxide (CO₂), and these, along with other greenhouse gas emissions, have caused climate change via the Greenhouse Effect⁽¹⁻³⁾. Carbon capture and sequestration (CCS) can be used to mitigate the rise in atmospheric CO₂ levels by capturing CO₂ from large stationary sources (*e.g.* fossil fuel based power plants, cement manufacturing plants, etc.) and storing it underground (Fig. 1). Geological sequestration can quantitatively contribute to a reduction in CO₂ emissions and is considered to be a technically feasible method for CO₂ mitigation⁽⁴⁻⁷⁾.

Deep saline aquifers can be major candidate sites for CO₂ sequestration because of their enormous storage potential. Field tests of geological sequestration of CO₂ have been performed in many parts of the world^(3, 8, 9). Generally, injected CO₂ in aquifers is in the supercritical state; however, the geological temperature gradient around the Kanto region in Japan is mild compared with the general temperature profile found globally⁽¹⁰⁾; thus, sequestered CO₂ in this region exists in a liquid state. There are several advantages to geological sequestration with liquid CO₂. Injected CO₂ forms a plume that migrates upward

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Fig. 1 Schematic view of geological storage.

because the density of CO₂ is lower than that of water. Liquid CO₂ density is approximately 800 kg/m³, which is higher than that estimated for the Sleipner gas field (supercritical CO₂, approx. 650 kg/m³)⁽¹¹⁾. The higher CO₂ densities lead to a reduction of the buoyancy effect on the plume, and higher storage efficiency is also achieved.

The estimation of the CO₂ permeation process, storage potential, storage cost, and the risk of CO₂ leakage from the storage reservoirs are still important issues for implementation of CO₂ geological sequestration. To address these issues, visualization and analysis of CO₂ behavior in the aquifer is required. One of the visualization techniques is X-ray computed tomography (CT). Several previous studies have used X-ray CT to observe CO₂ injection in rock samples⁽¹²⁻¹⁵⁾. Fluoroscopic X-ray images were acquired by rotating the X-ray source and detector. Three-dimensional CT reconstructed images were obtained nondestructively and noninvasively. As a result, porosity, CO₂ saturation, and relative permeability were measured from these CT data.

The X-ray CT that was employed in previous studies was originally developed for medical use, and the spatial resolution, especially the slice thickness (z-direction), is low compared with that of the CT slice image (x-y cross section). This means that even if the CT image were to be reconstructed with 100 to 200 μ m resolution, the image would be integrated to the 1–3 mm thickness of the sample. This axial resolution is not sufficient to characterize the core structure. In order to achieve pore-scale resolution, microfocus X-ray CT was employed⁽¹⁶⁾ and the gas migration phenomena was observed with 20 μ m resolution; however, only small samples (8–15 mm diameter) were used because of the higher resolution obtained for smaller samples.

In this study we observed the CO₂ migration phenomena in sandstone in laboratory experiments by using a microfocus X-ray CT with high spatial resolution on large diameter samples (50 mm in diameter and 142 μ m spatial resolution) and acquired images of the dynamic behavior of liquid CO₂ permeation and the CO₂ distribution in sandstone.

2. Materials and methods

2.1 Microfocus X-ray CT

This work takes advantage of the improved resolution achievable with microfocus X-ray CT. The fundamental geometry of microfocus X-ray CT is shown in Fig. 2. In this

Fig. 2 Sketch of magnification from microfocus X-ray CT.

Fig. 3 Berea sandstone sample 50 mm in diameter.

work, inspeXio SMX-225CT (Shimadzu Co., Ltd., Tokyo, Japan) with a very small focal spot size (4 μm) and a cone-shaped beam was used. The shadow image (which determines contrast) depends on the X-ray absorption of the sample and was projected on an image intensifier. Spatial resolution depends on the ratio between the source-to-object distance (SOD), source-to-image distance (SID), and the number of detector pixels. The spatial resolution in the x, y, and z direction is varied by changing the ratio of SOD to SID, and can be of the same order.

For their observations, most previous studies used a CT image resolution of 250–350 μm on a 35–50 mm diameter sample. In this study, we achieved a voxel resolution of 142 $\mu\text{m} \times 142 \mu\text{m} \times 142 \mu\text{m}$ by SOD = 450 mm, SID= 1000 mm, and detector pixels = 512 $\times$ 512. It allowed us to observe the micro-scale CO_2 distribution in a large sample (50 mm diameter).

2.2 Sample preparation

A core of Berea sandstone was used as the porous sample (Fig. 3). Thin beddings perpendicular to the axial direction are apparent. The core diameter and length were 50 mm and 140 mm, respectively. The permeability of the Berea sandstone was 300 mD and the average porosity was 16.4%. The pore volume (PV) of the sample core was approximately 45 mL. The sandstone was baked at 350°C for 6 hours to remove any organic components before being wrapped in silicone rubber and a heat-shrinkable PET tube. The core was subjected to a pressure of 10 MPa. The bottom of the core was drilled to allow an injection nozzle to be centrally set.

The Berea sandstone consists mainly of SiO_2 . On the SiO_2 , the CO_2 and water show a non-wetting and wetting phase, respectively. Hence, CO_2 permeation in the water-saturated Berea sandstone is a drainage process.

Fig.4 Experimental apparatus.

Fig. 5 Microfocus X-ray CT (left), high-pressure vessel on turntable (middle), and internal structure of vessel (right).

2.3 Experimental apparatus

The experimental apparatus consisted of two main components: the pressure control (for CO₂ injection) and the X-ray fluoroscope for observation. Figures 4 and 5 show the experimental set-up.

The sandstone was set in the sample holder in an appropriate position to provide the required spatial resolution. The axis of the cylindrical sample holder was set vertically in the microfocus X-ray CT. Previous studies that employed medical X-ray CT needed to set the sample horizontally; however, in this experimental set-up, gravity can be imposed in the sandstone as the original direction.

The CO₂ was injected under high pressure into the sandstone via the nozzle. The injection process and the pressure in the vessel were controlled throughout the experiment by a pressure control cylinder and a backpressure regulator valve. The pressure in the apparatus was maintained at 7.5 MPa, reproducing the conditions in a saline aquifer at a depth of 700 to 750 m. A syringe pump was used to inject the CO₂ into the water-saturated sandstone. The temperature was maintained at 20°C throughout the experiment; thus, the CO₂ was in a liquid state during the experiment.

2.4 Measurement of porosity and CO₂ saturation

The porosity and CO₂ saturation of the sandstone were measured by X-ray CT and mapped from several images or by varying the X-ray energy⁽¹²⁻¹⁴⁾. In this study, six images at constant X-ray energy were obtained for each of: the dry and water-saturated sample holder without the sandstone; the water and CO₂ saturated sandstone (the core was filled

with CO₂ under reservoir conditions); the dry sample; and an experimental image under steady state conditions. The local porosity and local CO₂ saturation were then calculated using the following equations:

$$\phi(i, j) = \frac{CT_{\text{water\_saturated\_sample}}(i, j) - CT_{\text{dry\_sample}}(i, j)}{CT_{\text{water}}(i, j) - CT_{\text{air}}(i, j)} \quad (1)$$

$$S_{\text{CO}_2}(i, j) = \frac{CT_{\text{CO}_2\text{-injected}}(i, j) - CT_{\text{water\_saturated}}(i, j)}{CT_{\text{CO}_2\text{-saturated}}(i, j) - CT_{\text{water\_saturated}}(i, j)} \quad (2)$$

where CT is the brightness value of the pixel in the CT image and i and j are the location coordinates of the pixel in the CT image.

The experiments followed these key steps: first, X-ray CT images were taken of the empty vessel and the water-filled vessel. Aqueous sodium iodide (NaI) was used as a contrast agent to provide contrast between the water and CO₂ in the CT images.

Previous studies focused on the permeation process of supercritical CO₂, where the difference between the X-ray absorption coefficient of supercritical CO₂ and that of water is easily detected. In order to investigate liquid CO₂ in water-saturated sandstone, we set the NaI concentration as 25% by mass. After draining the water from the vessel, X-ray CT images of the dry sample in the vessel at atmospheric pressure and of the CO₂-saturated sample in the vessel at 7.5 MPa were acquired. The experimental apparatus was then filled with water, and CO₂-saturated water injection was performed at a high back pressure of 7.5 MPa. The CO₂ was injected into the sample at a constant flow rate using a syringe pump, and CT images were acquired after specific injection amounts. Because the axial scan area of the X-ray CT was smaller than the sample length, scanning images were obtained at different sample axial positions (bottom, middle, and top).

The reconstructed images were visualized and analyzed using VGStudio MAX (Volume Graphics) and MATLAB (The MathWorks, Inc.).

2.5 Fluoroscopic observation of CO₂

Medical X-ray CT generally uses a line detector, while microfocus X-ray CT employs a plane detector. This is an additional advantage of microfocus X-ray CT. It allows real time two-dimensional observation because the conical beam irradiates a square shaped detector. The CO₂ permeation process was observed through transparent images that were taken over a short time period. By determining the difference of X-ray absorption between the CO₂ and NaI solutions, the CO₂ permeated region can be observed by transparent images.

3. Results and discussion

3.1 Three-dimensional CO₂ distribution in Berea sandstone

The CO₂ injection rate was 0.02 PV/min (1.0 mL/min), which is similar to that in an actual injection well. The distributions of local CO₂ saturation were reconstructed three-dimensionally after 0.55 PV and 2.12 PV of CO₂ were injected. Figure 6 shows the distribution of local CO₂ saturation as an anaglyph image, and the highly saturated voxels are clearly reconstructed.

The CO₂ permeation can be observed by the increment of gray colored volume (through the red/blue glasses) from 0.55 PV to 2.12 PV. When the CO₂ permeates the water-saturated sandstone, water in the micro-scale pore structure is displaced by the CO₂, and the water flows toward the outlet end of the sandstone. In this experiment, CO₂ dissolution into the

Fig. 6 Three-dimensional anaglyph image of CO₂ distribution. Red-colored (left) and blue-colored (right) glasses are recommended. The gray volume through the red/blue glasses indicates the $S_{CO_2} > 60\%$ region.

water was prevented by using CO₂-saturated water in the sandstone.

The CO₂ distribution indicates a thin layer structure. The bedding structure of the sandstone as seen by external observation indicates different local porosity and permeability which results in the inhomogeneous CO₂ distribution.

3.2 Distribution of local porosity and local CO₂ saturation

The local porosity and local liquid CO₂ saturation distributions were observed at each of the three stages of injection: 0.55 PV, 1.00 PV, and 2.12 PV (Fig. 7). The local porosity distribution and CO₂ permeation process were obtained quantitatively with high spatial resolution.

Compared with the slight heterogeneity in the distribution of the local porosity, the CO₂ distribution clearly shows a significant heterogeneity in the vertical section. A striped pattern was observed in the CO₂-saturated region (mainly green and yellow area). The CO₂ permeated a specific area of the porous media during the early stages of the CO₂ injection. Capillary fingering would occur under the present experimental conditions on a pore-scale⁽¹⁷⁾; however, the CO₂ distribution shows a piston-like permeation front. Large-scale fingering structure was not observed during the injection process.

The distribution of CO₂ saturation in horizontal sections C and D shows higher penetration near the permeation front and a change from blue to green. On the other hand, the CO₂ saturation shows an almost steady distribution in horizontal sections A and B in which the CO₂ had already permeated.

Figures 8 and 9 depict the slice-averaged porosity ϕ_{ave} and slice-averaged saturation S_{ave} as a function of sample height. The slight fluctuation of ϕ_{ave} can be highly resolved, and it is closely related to the laminar structure of thin beddings throughout the entire sample as shown in Fig. 3. The sandstone is formed by compaction of periodic deposition of sand grains in the sea or lakebeds. The grain diameter is graded because the deposition speed is dominated by Stokes' law. Thus, each laminar structure in the sandstone consists of

Fig. 7 Distribution of porosity and CO₂ saturation in sandstone.

different diameter sand grains. The thickness of each layer is determined by its depositional history, and the porosity in each layer is considered to depend on the property of the grain diameter and its shape.

When 0.55 PV CO₂ was injected, an unsteady region of slice-averaged CO₂ saturation was observed. The thickness was approximately 10 mm and the CO₂ saturation rapidly increased in the invading front region. This caused a piston-like CO₂ permeation process as seen in Fig. 7. After passing the CO₂ front, the CO₂ saturation approached a steady value (when 1.00 and 2.12 PV CO₂ was injected). In this experiment, the overall averaged saturation by liquid CO₂ was 33%.

Fig. 8 Slice-averaged porosity ϕ_{ave} as a function of sample height.

Fig. 9 Slice-averaged CO₂ saturation S_{ave} as a function of sample height.

Fig. 10 Correlation between slice-averaged porosity and slice-averaged saturation (corresponding to $z = 40\text{--}70$ mm in Figs. 10 and 11).

Between 1.00 PV and 2.12 PV CO₂ injection, the CO₂ flux in each slice was conserved because the CO₂ injection rate was constant, and the conservation can be written by the

Fig. 11 Transparent images showing the CO₂ permeation process. Each image is subtracted from the original image that was obtained before CO₂ injection. Scale bar indicates 10 mm and arrow shows direction of CO₂ permeation.

Fig. 12 Signal intensity ratio (qualitative CO₂ saturation) as a function of time.

following equation in each slice:

$$Q = \phi_{ave} \times S_{ave} \times U_{CO_2_{ave}} = const. \quad (3)$$

where Q is the CO₂ flux (injection rate) and $U_{CO_2_{ave}}$ is the averaged axial velocity of the CO₂ phase in each slice. It is clear from Figs. 8 and 9 that $\phi_{ave} \times S_{ave}$ in each slice is not constant and fluctuates considerably. Thus, $U_{CO_2_{ave}}$ also changes its value in each slice and the fluctuation of $U_{CO_2_{ave}}$ is estimated to be 6.0 to 14.1 mm/min in this experiment.

As shown in Fig. 9, the CO₂ permeation was strongly affected by the local porosity distribution and consequently a strong correlation is seen between the local porosity and CO₂ saturation, see Fig. 10. The fluctuations in the slice-averaged porosity were caused by the thin bedding structure. The slice-averaged CO₂ saturation was very sensitive to changes in the slice-averaged porosity. A change in CO₂ saturation of approximately 10% was induced by a $\pm 1\%$ fluctuation in the slice-averaged porosity.

Fig. 13 CO₂ permeation process on lower porosity bedding and higher porosity bedding.

3.3 Permeation process of CO₂

The permeation process of CO₂ was observed by CT and transparent images. Figure 11 shows transparent images of the CO₂ permeation process. Each image was subtracted from the initial image ($t = 0$ sec). When CO₂ permeates the water-saturated sandstone, the X-ray absorption coefficient changes are relatively small in the pore where CO₂ displaces the water (indicated as white spots in the image). In Fig. 11, the striped pattern of the CO₂ saturation area is clearly present as white streaks. The image was integrated into the distribution of the absorption coefficient along the X-ray path; thus, the striped pattern indicates that the CO₂-saturated region emerges in layers in the sandstone. The permeation front appears as a flat shape and each horizontal white stripe appears in order from the upstream CO₂ permeation. The higher porosity beddings were preferentially saturated.

Figure 12 shows the variation of the qualitative CO₂ saturation with time, and was obtained from averaged X-ray intensities at the same height in the sandstone. The CO₂ front advanced at a rate of approximately 5 mm/min in the sandstone sample at an injection rate of 1.0 mL/min. The CO₂ saturation rapidly increased at the permeation front region, which was quite thin (10 to 15 mm). The thickness of the permeation front is in good agreement with the result of the 0.55 PV injected CO₂ shown in Fig. 9. The CO₂ distribution exhibited an almost steady state condition after passing the CO₂ permeation front.

3.4 Effect of thin laminar heterogeneity for CO₂ permeation process

Our results indicate that at the CO₂ permeation front the CO₂ saturation suddenly increased and after the front passed it maintained an almost steady state. The CO₂ saturation distribution reached a steady state only after 2.12 PV CO₂ was injected. On the other hand, previous studies in which supercritical CO₂ was used indicate a thicker permeation front region with 3 to 13.4 PV injected before CO₂ saturation reached a steady distribution^(14, 15).

The influence of the sub-core scale heterogeneity was discussed by Perrin et al.⁽¹⁴⁾ and Shi et al.⁽¹⁵⁾, and recent studies noted that permeability discontinuities cause saturation discontinuities^(18, 19). In this study, a thin laminar structure in a large-diameter sandstone sample can be observed by employing microfocus X-ray CT. The thin laminar structure is considered to have a strong effect on the CO₂ permeation process.

CT images in a typical slice plane parallel to the laminar structure are shown in Fig. 13. The difference between the CO₂ permeation of the low and high porosity slices is clearly seen in the change of CO₂ saturation. It is difficult for the CO₂ to permeate the low-porosity slice because of capillary pressure. Voxels of increased CO₂ saturation are scattered evenly on the low-porosity slice and the slice-averaged CO₂ saturation is relatively low. On the other hand, at the high-porosity slice the CO₂ permeates easily. The CO₂ saturation increases locally and unevenly, and the CO₂ spreads horizontally. In the final stages, the CO₂ saturation reaches higher values.

It is considered that the discontinuity of the bedding structure causes a capillary end effect and the low porosity layers act as a barrier to CO₂ permeation. During the CO₂ permeation from a higher to a lower porosity region, the boundary forms a temporary barrier to the flow of CO₂ and prevents CO₂ flow until the threshold pressure at the boundary is overcome^(18, 19).

For these reasons, thin laminar structure is considered to act as a barrier. It reduces the permeation front thickness and stabilizes the CO₂ saturation instantly. From the viewpoint of CO₂ migration, the lower porosity bedding acts as a local caprock structure. In order to evaluate the storage potential, flow pattern, and upward migration of liquid CO₂ in sandstone, micro-scale heterogeneities should be considered even in macro-scale CO₂ permeation phenomena.

4. Conclusion

The permeation process of liquid CO₂ in water-saturated Berea sandstone was studied using microfocus X-ray CT. High resolution observations of a large sample (sample diameter, 50 mm; image resolution, approximately 142 μ m) were performed. Microfocus X-ray CT can provide detailed images of the CO₂ permeation process even if the large sandstone sample contains quite thin laminar heterogeneities. A three-dimensional reconstructed image of the liquid CO₂ distribution in the Berea sandstone was obtained. The average CO₂ saturation was approximately 33% in sandstone with an average porosity of 16% at an injection rate of 0.02 PV/min. The value of the slice-averaged CO₂ saturation was strongly affected by the slice-averaged porosity, and highly saturated regions showed laminar structure. The bedding structure strongly determined the CO₂ permeation path. The X-ray transparent images showed the real-time behavior of CO₂ permeation in sandstone. The injected CO₂ gradually permeated in the axial direction with increasing saturation in the higher porosity beddings because the lower porosity bedding acted as a barrier due to capillary pressure. After the CO₂ permeating front passed, the local CO₂ saturation exhibited an almost steady state condition with a non-homogeneous distribution. In order to evaluate the storage potential of the sandstone and the flow pattern and upward migration of the CO₂, micro-scale heterogeneities should be considered even in macro-scale CO₂ permeation phenomena.

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