Effect of CO₂ Phase on Pore Geometry of Saline Reservoir Rock

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Abstract
The phase of CO₂ present in a saline reservoir influences the change of the pore geometry properties of reservoir rocks and consequently the transport and storage integrity of the reservoir. In this study, digital rock physics was used to evaluate pore geometry properties of rocks saturated with the different phase CO₂-brine under reservoir conditions. The changes in the pore geometry properties due to the different phase CO₂-brine-rock interaction were quantified. In addition to compression, CO₂-brine-rock interaction caused a further reduction in porosity by precipitation. Compared to the dry sample, the porosity of the gaseous CO₂-br sample was reduced the most, and was lower by 15% after saturation and compression. There was reduction in the pre-compression porosity after compression for all the samples, however, the reduction was highest in the gaseous CO₂-br-saturated sample (13%). The flatness of pore surfaces was reduced, and pores became less rounded after compression, especially in supercritical CO₂-br-saturated rock. The results from this research provide a valuable input to guide a robust simulation of CO₂ storage in reservoir rocks where different phases of CO₂ could be present.

Highlights
- Effect of phase of CO₂ on changes in pore geometry of saline reservoir is evaluated.
- scCO₂-br caused the highest change in pore flatness and shape.
- gCO₂-br caused the greatest change in pore volume.
- Phase of CO₂ is important in CO₂-brine-rock relationship.

Keywords
Pore geometry · Digital rock physics · CO₂ phase · Reservoir rock · Brine

List of Symbols

\begin{align*}
Q & \quad \text{Flow rate (cm}^3/\text{s}) \\
A & \quad \text{Cross-sectional area of the sample (cm}^2) \\
\Delta P & \quad \text{Differential pressure in the direction of flow (atm/cm)} \\
L & \quad \text{Length of the sample in the flow direction (cm)} \\
K & \quad \text{Permeability (Darcy)} \\
\psi & \quad \text{Sphericity of pore} \\
V & \quad \text{Volume of a pore (mm}^3) \\
a & \quad \text{Surface area of a pore (mm}^2) \\
\pi & \quad \text{Pi} \\
\Phi & \quad \text{Porosity measured from the digital rock (\%)} \\
\theta & \quad \text{Initial connected porosity (\%)} \\
R & \quad \text{Equivalent radius of a pore (mm)} \\
D & \quad \text{Equivalent diameter of a pore (mm)} \\
\Theta & \quad \text{Initial total porosity (\%)} \\
\rho_b & \quad \text{Bulk density of samples (g/cm}^3) \\
\rho_p & \quad \text{Particle density taken as the average density of the minerals in the rock, i.e., 2.58 g/cm}^3. \\
C^{*} & \quad \text{End of shear enhanced compaction or the onset of dilation following the compaction}
\end{align*}
1 Introduction

Dissolution, residual and capillary trappings are methods of trapping CO₂ in a saline reservoir. The rate of dissolution of CO₂ is therefore a critical parameter for effective CO₂ storage. Yan et al. (2011), Ratnakar et al. (2020) and Mohammadian et al. (2015) provided solubility data of CO₂ in brine. It was seen that the solubility of CO₂ in brine depends on the temperature, pressure and molality of the brine. The solubility gets very low at low pressure, high temperature and high brine molality. Therefore, in a geosequestration site, there will be undissolved CO₂ for a few thousand years (Ennis-King and Paterson 2003; Blunt et al. 2013). The phase of CO₂ depends on the pressure and temperature (P–T) condition of the reservoir. Variability in P–T conditions result in a change of phase of CO₂. The P–T condition of reservoirs are variable (Barker 1972; KGS 2003; Wang et al. 2014). This means that the undissolved CO₂ will be present in different phases with the brine (Acevedo and Chopra 2017; Zhang et al. 2019a, b). The undissolved CO₂ is contained in the pores with the brine and will change the shape, sphericity and flatness of the pores, as well as the porosity. These changes are induced by the pressure of the pore fluid and the associated geochemical reactions. Al-Zaidi et al. (2018) showed that the pore pressure due to CO₂ depends on the phase of the CO₂ and Vanorio et al. (2011) opined that compositional changes control the change in the rock’s fabric as well as other changes in rock’s properties. So, different phaseCO₂-brine present in a reservoir will trigger unique kind of changes. Therefore, there is a need to understand the effect of the different phaseCO₂-brine on rock properties in a CO₂-brine-rock interaction. This study looks specifically at the effect of different phaseCO₂-brine on the pore geometry properties of saline reservoir rocks. Pore geometry properties is important because it controls the transport, petrophysical and geomechanical properties of the rock (Akbar et al. 2019).

Most researches on the effect of CO₂ on reservoir properties like Delle Piane and Sarout (2016), Pimienta et al. (2017), He et al. (2019), Isaka et al. (2020) and Huang et al. (2020) have assumed that CO₂ remains in a single-phase throughout the storage history. This assumption is not always true. Lu and Connell (2014) explained that the density and phase of the supercritical CO₂ injected into wells change over the time in response to the variation in P–T condition. The change in the phase of CO₂ in reservoir and its consequences have been reported by Paterson et al. (2008), Denney (2009) and Lu and Connell (2014). Researches have shown that CO₂ storage in saline rocks leads to change in transport, petrophysical and geomechanical properties in those rocks. For example, Saeedi et al. (2011) observed that the multiphase flow characteristics of a CO₂-brine reservoir changes due to chemical reactions and change in stress in reservoir conditions associated with flooding cycles. Similarly, Reynolds and Krevor (2015) reported changes in relative permeability of saline rocks due to CO₂ flow. Vanorio et al. (2011) has showed that injecting CO₂ into brine-rock system induced chemo-mechanical processes that permanently alters the rock’s framework, and Griffiths et al. (2017) showed that change in pore aspect ratio influences the strength and stiffness of porous rocks. Reduction in the strength of the rocks due to CO₂-brine-rock reaction has been reported in Delle Piane and Sarout (2016), Pimienta et al. (2017), Meredith et al. (2017), Espinoza et al. (2018), Keshavarz et al. (2019), Fuchs et al. (2019) and Zhang et al. (2020). The change in the strength of the rocks is seen to vary from slight to high depending on the mineralogy and the reservoir conditions. The types of rock used in these researches range from carbonate rocks, sandstones and shale rocks. It is clear that research on the effect of different phaseCO₂-brine on the pore geometry properties of reservoir rocks is lacking. Therefore, this research sets out to evaluate the changes to the pore geometry of a saline rock due to the different phaseCO₂-brine using digital rock physics. Digital rock physics involve evaluating properties of rocks from digital images from micro-CT, SEM or FIB-SEM using suitable software. Application of digital rock physics in studying rock properties is rapidly growing and can be found in Andrä et al. (2013), Lesueur et al. (2017), Berg et al. (2017) and Sun et al. (2019).

This research focuses on the changes of the pore geometry properties (pore volume, shape, flatness, equivalent radius, and sphericity) in the reservoir rocks when saturated with different phaseCO₂-brine. In this research, the phaseCO₂-brine states of the reservoir considered include br-saturated sample (reservoir saturated with brine), gCO₂-br-saturated sample (reservoir saturated with brine and gaseous CO₂), scCO₂-br-saturated sample (reservoir saturated with brine and supercritical CO₂) and gCO₂-saturated sample (reservoir saturated with gaseous CO₂). Samples were also tested in dry condition to represent the property of the dry natural reservoir rock. The gCO₂-br and the scCO₂-br reservoir conditions represent a scenario where gaseous and supercritical CO₂ exist with the brine, respectively. The gCO₂-saturated reservoir represents when gaseous CO₂ migrates into a dry reservoir while the br-saturated reservoir represents the normal saline reservoir before saturation with CO₂. Liquid CO₂ is not considered because the P–T condition of the reservoir at the suitable storage depth does not favour existence of liquid CO₂. CO₂ rarely exist in liquid phase in saline reservoirs except in deep sea or ocean sediments (House et al. 2006).

The result of this research will provide a better understanding of reservoir response to CO₂ storage and also to provide information with which to better constrain CO₂
storage modeling studies as well as open up new ways of simulating CO$_2$-brine-rock interaction.

### 1.1 Geology of the Study Site and Reservoir Rock

The Lower Cretaceous Captain Sandstone Member of the Inner Moray Firth in the UK has significant potential for the storage of anthropogenic CO$_2$ in the saline reservoir (Akhurst et al. 2015). Storage capacity also exists in the depleted hydrocarbon fields (Marshall et al. 2016). From simulation studies reported by Jin et al. (2012), the Captain sandstone in this region can store 358–2495 Mt of CO$_2$. Studies such as Jin et al. (2012) and Noy et al. (2012) have shown that displacement of existing pore fluids (brine) will be the mechanism of CO$_2$ storage in this reservoir. Details of the structural geology of the study area and other adjoining areas of influence can be found in Williams et al. (2016).

### 2 Materials, Experimental Setup and Procedure

#### 2.1 Materials

Five different Captain Sandstone samples obtained from the depth of 1638.5–1640.3 m in well 13/24a-6 (Red triangle in Fig. 1) were used for this experiment. The samples were collected from a potential CO$_2$ storage unit in the UK (Ekofisk CO$_2$ storage unit). Mineralogical analysis of the dry natural sample by XRD method showed that the sample contained the following by weight %: Quartz (79.5%), K-feldspar (orthoclase (0.66%), Microcline (4.57%), Plagioclase feldspar (Anorthite (0.72%) and Clay minerals (Illite (1.7%), Kaolinite (4.34%), Chlorite (1.73%) and Dickite 6.83%). The permeability of the dry natural samples before the experiment was measured using the method by Lenormand et al.

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*Fig. 1 Location map showing well 13/24a-6 (triangulated) where core samples were collected. Adapted from Williams et al. (2016)*
The permeability was calculated using Eq. (1)

\[ K = \frac{Q\mu L}{A(\Delta P)} \]  

(1)

The permeability was found to be 1.13e−12 m² (1.14 Darcy). The porosity of each of the sub-sample was measured before the experiment. It is seen that the core sample is largely homogenous. Therefore, all the core samples used for the experiment are homogenous, i.e., same starting material. The initial porosity for each sample is shown in Table 1. The porosity was determined as the ratio of the pore volume to the bulk volume Eq. (2).

\[ \text{Porosity} = \frac{\text{Pore volume}}{\text{Bulk volume}} \times 100. \]  

(2)

The weight of the samples in dry, water-saturated and submerged states was measured. The grain volume and pore volume was determined from the dry, saturated and submerged weights. The bulk volume was determined from measuring the dimensions of the samples. The measurement was repeated four times for each sample and the porosity was taken as the average of the four measurements. The samples used in this study can be described as weak, structureless, stained whitish, sub-rounded, fairly sorted quartz arenites (Fig. 2), with an average grain size measured from image analysis as 223 μm. To allow the comparison with the different phaseCO₂-brine-saturated samples, the mechanical (stress–strain) behaviour of the dry natural sample is presented and the pore and matrix distribution in the dry natural sample before and after compression is investigated. The measured properties of the dry natural sample is the properties of the natural rock before commencement of the experiments. These will be compared to the changes that occur in the other phaseCO₂-brine-saturated samples as a result of compression. Analysis of different Captain Sandstone samples by different researchers has shown that the Captain sandstone is similar in terms of physical, lithological, and mineralogical properties. For instance, the mineralogical characterisation of Captain sandstone conducted by researchers such as Hangx et al. (2013) and Rice-Birchall (2018) showed a similar composition. The major minerals are quartz and feldspar, with some quantity of clay. The permeability values of different samples of Captain Sandstone, as measured by Shell (2011), Jin et al. (2012), and Hangx et al. (2013), are within the same range.

The experimental setup consists of:

- a Vacuubrang GMBH, Model MZ 2D NT vacuum pump, to remove trapped gases from the samples and ensure complete saturation of brine in the samples,
- two (Parr Instruments, IL, USA) pressure vessels with a pressure range of 0–70 bar and 0–200 bars to contain the sample, brine, and CO₂ under the desired pressure during saturation,
- an oven to control the temperature as required while being saturated, and
- a 250 kN triaxial test machine and a 3–70 MPa confining pressure pump for applying triaxial compressive stress to the samples after saturation.

![Photograph of the dry core samples](image_url)
Accessory components for the triaxial compression test include:

- a temperature regulator (consisting of an N2006 PID temperature controller and a K-type thermocouple with a precision of 0.2% and a resolution of ±1 °C) to maintain the temperature of the sample and sample holder at 35 °C during triaxial compression,
- a pressure transducer (gems sensors and controls, with a range of 0–100 MPa and precision of 0.25%) to measure and log the real-time pressure impacted on the sample, and
- a micro link 751 multifunction data acquisition and control (set at 15 bits per channel, with each channel limited to ±10 Vdc and a resolution of 0.8 mV, over a ±10 V range with a precision of ±0.05%). This records the displacement data from the LVDT (Linear Variable Differential Transducer), which is also concurrently displayed on a computer using the windmill software.

A Zeiss Xradia micro-CT scanner was used to scan the samples before and after compression. A schematic illustration of the workflow of the experiment is shown in Fig. 3. Micro-CT scanning was conducted using a Zeiss Xradia 410 μ-CT with a beam energy of 80 keV and power of 7 W. 3D digital rock was reconstructed from 996 slices of 2D scanned images. The images were taken at a resolution of ~3 μm and a size of 988 × 1015 × 997 voxels. Image processing and analysis was done with Avizo software. A non-local means filter was applied to the images to remove the noise. The non-local means filter was chosen because of its robustness and the need to preserve edges. No smoothening was applied to preserve the structures. The filtered images were segmented to isolate the pore domain in the material using the thresholding and watershed function by setting grey-scale thresholds that segment the digital rock into pore and grain materials. The pore volume, shape, flatness, equivalent radius, and sphericity of pores that constitute the pore domain were computed using the volume fraction and label analysis module of Avizo. Details of how the pore shape, pore volume, and flatness were computed will be explained in Sect. 3.

### 2.2 Experimental Set up and Procedure

Different core samples containing various phaseCO2-brine were saturated and then subjected to reservoir pressure condition using multiple triaxial tests, as shown in Fig. 3. Dissolution of CO2 in brine produces an acidic solution. Results from Peter et al. (2020), Pimienta et al. (2017) and Zou et al. (2018) show that the impact of CO2 on the properties of rock starts vigorously almost immediately. Hence, for this experiment, any duration greater than 5 days is considered sufficient for the phaseCO2-brine to have an effect on the rock properties. The samples were saturated in different phaseCO2-brine for 7 days.
The experiment was carried out in two stages. In the first stage, the core samples were initially vacuum-saturated. They were soaked in brine (60,600 ppm NaCl solution) for 7 days in a vacuum (except the samples to be used as dry and gCO₂-saturated samples). The samples to be used as scCO₂-br and gCO₂-br-saturated samples were then flooded with the respective phaseCO₂ for a further 7 days while still soaked in limited brine. To maintain the CO₂ in their desired phases during the CO₂ flooding stage, pressure and temperature condition were held at 50 bars and 27 °C for the gCO₂ samples, while pressure and temperature condition was held at 100 bars and 36 °C for the scCO₂ bearing sample. The brine-saturated sample was kept at room temperature and 1 atm. By doing these, each rock sample was prepared to represent a given phaseCO₂-brine state of the reservoir, as described in Sect. 1.

In the second stage, all the samples were subjected to the reservoir’s pressure and temperature conditions using multiple triaxial compressive tests. The corresponding stress–strain curves are shown in Sect. 4.1. Confining pressures of 16, 26, 36, and 46 MPa were applied successively to each sample at a constant strain rate of 10⁻³ S⁻¹. The values of the confining pressure ranging from 16 to 46 MPa were chosen to represent the confining pressure at the site where the samples were collected, starting from the shallowest depth suitable for storage, i.e., 800 m, until a greater depth where the confining pressure is 46 MPa. The confining pressure in reservoir increases with depth. The confining pressure at the shallowest depth suitable for CO₂ storage in the Ekofisk storage unit in the central North Sea is 15–16 MPa, and the temperature at this depth is 34–44 °C (NERC 2019). The shallowest depth for suitable for CO₂ storage is 800 m (Holloway et al. 2006), and the minimum confining pressure is 15 MPa (Hangx et al. 2013). Therefore, the triaxial test was designed to simulate reservoir conditions ≥ 800 m. For each confining pressure, axial stress was increased until shear-enhanced compaction ends.

To get a scanned image of the samples in their respective states before compression, two samples were prepared in stage one for each phaseCO₂-brine state. The main sub-sample with a length of about 36 mm and diameter of 38 mm was used for the triaxial experiments and the smaller cylindrical sub-sample with a diameter of 4.5 mm and length of 7.5 mm was scanned for evaluation of the pore geometry. Before scanning, the saturated samples were allowed to dry. The image of a dry natural sample was also scanned before compression. After compression, another set of 4.5-mm by 7.5-mm cylindrical sub-samples were collected from the center of all the compressed samples and scanned after being dry, for evaluation of pore geometry properties after compression.

Digital rock physics was used to obtain pore geometrical properties such as pore volume, shape, flatness, equivalent radius, and sphericity, which are then analysed to obtain the changes in pore geometric properties in the different samples. Due to heterogeneity in the distribution of pores (as shown in Fig. 4), computational limitation, and the need to have a workable volume of the digital rock, various schemes have been used in the literature to achieve numerical efficiency in the computation of multi-scale and heterogeneous properties in digital rock physics (Lesueur et al. 2017). One of such is the representative element volume (REV) of the digital rock. REV is the domain size where material properties are representative of a heterogeneous material. Hurley et al. (2012) described REV as the volume size that can be modeled to yield consistent results, within acceptable limits of the variance of the modelled property. Fernandes et al. (2012) described REV as the size of the digital rock that is

Fig. 4 2D slices of micro-CT image showing variation in the size, shape, and distribution of pores in different zones.
big enough to represent the property of the sample and small enough to allow for successful computation. This implies that REV is a trade-off between laboratory and computationally efficient rock volume size and accurate measurement.

In this research, the REV is considered as the sub-volume of the micro-CT images that shows the least or no variation in the measured porosity when taken from different locations of the whole image. A schematic workflow to establish the REV is shown in Fig. 5. The REV was evaluated from the scanned images of the sub-sample of the original sample.

The total volume of the digital rock refers to the whole volume of the image obtained from the scanned sub-sample and is equal to the volume bounded by $3.36 \times 3.43 \times 3.37$ mm. The sub-volume of the digital rock is a portion of the total volume of the digital rock. The total volume of each digital rock was cropped to remove the edges and then divided into sub-volumes from which REV was determined. This was achieved by taking incremental sub-volume sizes from different parts of the image and measuring the porosity. Smaller sub-volumes were taken from different corners of the total

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**Fig. 5** Schematic workflow for transforming core samples into digital rock REV

**Fig. 6** Porosity measured from different sub-volumes sizes (A–G) of the original sub-sampled digital rocks
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The sub-volume size was successively increased until the largest possible sub-volume size, which allows more than one sub-volume within the total volume. The measured porosity is presented in Fig. 6. The sub-volume size (E) bounded by 1.04 × 2.15 × 3.37 mm was chosen as the REV because the measured porosity is seen to show the least variation. This volume meets the criteria for REV and is a representative volume of the sample in this research. The sub-volume sizes used are shown in Table 2.

The average grain size of the samples was determined from measurement of the size of grains in SEM images. Five SEM images taken at different locations of each sample were used and the grain sizes were averaged by Image J software. In this study, except the nature of the saturating phase CO2-brine, other factors that affect pore geometry were the same for all the samples. Therefore, only the effect of the different phase CO2-brine on pore geometry was evaluated.

### Table 2 Bounding physical size dimensions of the sub-volumes

| Sub-volume | Bounding physical size of digital rock (mm) | Volume of sub-volume (mm³) |
|------------|---------------------------------------------|---------------------------|
| A          | 0.74 × 0.58 × 3.37                           | 1.45                      |
| B          | 0.74 × 1.16 × 3.37                           | 2.89                      |
| C          | 0.81 × 1.65 × 3.37                           | 4.50                      |
| D          | 1.48 × 1.16 × 3.37                           | 5.79                      |
| E          | 1.04 × 2.15 × 3.37                           | 7.54                      |
| F          | 1.48 × 1.65 × 3.37                           | 8.23                      |
| G          | 1.48 × 1.88 × 3.37                           | 9.38                      |

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### 3 Theoretical Background

The shape of the pore is taken as the aspect ratio. The shape of the pore was taken as the ratio of the maximum Feret diameter to the minimum Feret diameter (Sympatec 2017; Thermofisher 2018). Typical shapes corresponding to various aspect ratios can be seen in Vision (2012) and Chen et al. (2018). In this research, pores with an aspect ratio greater than 1.9 are rod-like (Delle Piane and Sarout 2016) while those with aspect ratio 1 < S < 1.9 will be considered as sub-rounded. Pores with 0.8 < S < 1 are considered to be rounded but there were no rounded pores in the samples.

Flatness is commonly used to express the amount of waveness or variation in a plane surface (GD&T 2014), and it is measured as the width of the tolerance zone. The tolerance zone is defined by two parallel planes that bound the surface of the pore. Numerical methods for evaluating the flatness of surfaces include the best fit and the minimum zone method. The best fit method is prone to the error of false negatives (FARO 2019). The minimum zone method, which is the preferred method and is used in this research, requires two envelop planes enclosing the data points measured and their associated uncertainty. The data points are sampled from the whole surface, therefore the path of measurement and the number of points are important (Calvo et al. 2014).

The pore volume is the volume occupied by the pore according to the shape. The change in pore volume (contraction/dilation) can evaluated based on the evolution of pore structure during CO2 underground storage. The sphericity of a pore (Ψ) is the ratio of the surface area of a sphere that has the same volume as the pore to the surface area of the pore (Wadell 1935). According to Ji et al. (2012), the sphericity is expressed as:

\[
Ψ = \frac{\pi}{3} (\frac{6V}{a})^{\frac{1}{3}}
\]

where \( a = 1 \) is a sphere and a \( Ψ \) value increasingly less than one is highly non-spherical.

The equivalent radius is the radius of a sphere whose volume is identical to a pore. According to Ji et al. (2012), the equivalent radius and the equivalent diameter are expressed as:

\[
D = \frac{(6V)^{\frac{1}{3}}}{\pi}
\]

\[
R = \frac{D}{2}
\]

### 4 Results and Discussion

Natural core samples suitable for core flooding triaxial experiments are difficult to get, hence, one sample was used to represent each phase CO2-brine state of the reservoir. Initial characterisation of the samples was done and the samples are seen to be homogenous. The samples are of the same lithology, i.e., weak, sub-rounded, medium grained quartz-arenite. Properties such grain volume, pore volume, dry density, porosity and lithology are related to permeability, pore geometry properties and mineralogy. The sample is majorly composed of Quartz, Feldspars and different types of clayey minerals. The minerals identified in the photomicrograph (Fig. 7) are same with the mineral phases identified from the XRD result.

Table 3 provides evidence of change in mineral composition in the samples saturated with different phase CO2-brine. It shows that injecting CO2 into brine-rock system induces geochemical reaction that alters the composition of the minerals, and the alteration in the composition of the minerals is affected by the phase of CO2.
4.1 Triaxial Compression and Micromechanical Properties

During the multiple triaxial compression test, axial stress was increased until shear-enhanced compaction ends (Fig. 8a–d). It is difficult to exactly pinpoint the end of shear enhanced compaction or the onset of dilation following the compaction (commonly termed $C^*$) without acoustic emission (AE) data. In this experiment, the end of shear enhanced compaction is taken as the point where the stress–strain curve just begins to deflect. It marks the point where the increased or sustained hardening due to compaction stops and the sample begins to dilate. A sample from the same formation was also tested to obtain the stress–strain characteristics before the experiment and the stress–strain
Fig. 8 Stress–strain curve of:
(a) dry sample, (b) br saturated sample, (c) gCO₂-br saturated sample, (d) scCO₂-br saturated sample, (e) gCO₂ saturated sample.
curve is shown as Fig. 8a. The test was stopped at the onset of dilation following compaction to avoid macro-failure/shearing as the aim of the experiment was to ensure that all the samples should be brought to the same stage of deformation.

Shear enhanced compaction is the hardening of rocks due to a reduction in porosity in response to increase in deviatoric stress. It is a micromechanical process that occurs alongside grain-scale micro-cracking which eventually coalesce to cause marked shearing and failure (Baud et al. 2006; Wong et al. 1997, 1992). Shear enhanced compaction was originally thought to be a phenomenon observed at the transition from brittle faulting in porous rocks (Wong and Baud 2012). However, research has shown that change in stress state causes a change in porosity and this relationship affects the mode of failure (Wong 1990). Shear enhanced compaction is the precursor to the onset of macro-failure/shearing in the rocks as has been explained in Baud et al. (2006) and Schock et al. (1973). The multiple triaxial compression test is a great method for estimating the strength and failure mode of rocks, however, it may introduce difficulty in continuing the test beyond a certain stress state after the sample has approached failure, due to post-peak strain softening (Akai et al. 1981a, b). There is also the tendency for the mechanical data from the later part of this kind of test to be affected by the deformation due to repeated cycles of axial loading, leading to an underestimation of strength. To mitigate this issue, deformation of the rock in this research was not allowed to advance into macro-shearing that would adversely affect the subsequent data. That is the reason why the test was stopped at $C^{w'}$ for each loading step.

It was observed that shear-enhanced compaction caused pore collapse and grain compaction across the entire sample leading to rock hardening with the increase in modulus. This phenomenon has been explained by Wong et al. (1997). This mode of deformation of rock has been ascribed to the mineralogy, and grain size by Klein et al. (2001) but the mode of deformation seen in this experiment is more likely due to mineralogy, grain size and the influence of the phase $\text{CO}_2$-brine that saturates the rock sample.

Figure 8a–e present the stress–strain behaviour of each sample during the multiple triaxial compression test. The total strain recorded by each sample (Fig. 8a–e) was sample during the multiple triaxial compression test. The confining stress level at a constant strain rate of $10^{-3}$ $\text{s}^{-1}$ was not allowed to advance into macro-shearing that would adversely affect the subsequent data. That is the reason why the test was stopped at $C^{w'}$ for each loading step.

Table 4 Axial and volumetric strain in each samples

| Sample | Dry (µS) | Brine (µS) | g$\text{CO}_2$-br (µS) | sc$\text{CO}_2$-br (µS) | g$\text{CO}_2$ (µS) |
|--------|---------|-----------|------------------------|-------------------------|-------------------|
| Axial strain at 16 MPa confining stress | 14,728 | 11,255 | 33,433 | 16,942 | 17,818 |
| Axial strain at 46 MPa confining stress | 6008 | 20,351 | 56,156 | 3354 | 5724 |
| Total volumetric strain | 51,154 | 56,053 | 110,707 | 46,686 | 41,612 |
rate even at low stress that less straining occurs at the higher stress level due to the resultant stress hardening and shear-enhanced compaction hardening.

In all the samples, stress hardening and shear enhanced-compaction hardening is responsible for the increase in stiffness. It is seen that at 16 and 26 MPa confining pressure, the sample’s stiffness increased significantly but the increase in stiffness was reduced at the 36 and 46 MPa confining pressure cycles except for the gCO$_2$-br sample where the stiffness at 46 MPa confining pressure increased. The decrease in stiffness as against stress hardening at those confining pressures for those samples is thought to be due to the effect of stress corrosion depending on phaseCO$_2$-brine that saturated the sample. Stress corrosion is a progressive chemo-mechanical action of pore fluid that weakens exposed surface bonds and facilitates progressive sub-critical crack growth, reduces the rock’s strength, and influences other properties of the rock (Brantut et al. 2013; Heap et al. 2015). The mechanism for this has been explained by Lawn (1993) and Heap (2009). The exposed bond in contact with the fluid breaks and accepts a new atom into the bridging bond. This is followed by bond lengthening and weakening, then newer surfaces are exposed and the crack grows deeper.

The result as seen from Fig. 8a–d shows that the samples exhibit significant ductility for all the confining pressures. A similar ductile behaviour of highly porous sandstone has been reported by Klein et al. (2001) in Bentheim sandstone as “quasi-ductile failure”. Similar ductile behaviour was reported by Sun et al. (2017) and they explained that it is a result of reduction in the cement size due to CO$_2$-brine-rock interaction. Furthermore, Scott and Nielsen (1991) explained that brittle-ductile transition depends on the initial porosity of the sandstone but that increasing the confining pressure has the effect of shifting the brittle-ductile boundary to lower porosities and vice-versa. Rohmer et al. (2017) assessed the ductile failure in cap rock due to CO$_2$ and found that ductile failure is likely under fixed vertical stress conditions and that the ductile failure would reduce the zone of influence of the CO$_2$ induced changes. All these indicates that CO$_2$-brine makes porous rock become ductile.

4.2 Pore Geometry Properties

In continuum mechanics, a major hypothesis is that the behaviour of many physical elements are continuous. Thus, physical quantities, such as mass, density, grain and pore geometry, and porosity, etc., contained in a large volume or mass of a material can be represented by a chosen representative element volume (REV). This assumption provides a way of effectively predicting the behaviour of macroscopic variable of the entire volume from the REV. Sometimes, multi-scale imaging is used especially for highly heterogeneous rocks as the smallest pores determine the resolution to be used for optimal imaging (Goral et al. 2020). Upscaling has also been used in different researches to circumvent this challenge (Long et al. 2016; Piller et al. 2014; Mehmani et al. 2020). Generally, rocks are heterogeneous in nature. Studying properties of rock encompassing the inherent
heterogeneity has remained a challenge due to the limitation of the size of rocks that can be studied, and how well it represent the core or field. In this research, REV was used to ensure that all the digital rock used for the analysis is representative of the inherent heterogeneity in the scanned core sample.

The effect of the phaseCO2 on the pore geometry properties is shown by comparing it with that of brine sample as curves. Because most storage reservoir contain brine, the result of the brine-saturated sample is used as the standard. X-ray micro-CT scans in Fig. 9 show the pores in the digital rocks of the different samples before and after compression. There was reduction in the porosity of all the samples after compression. SEM images (Fig. 10) of the micro-structure of all the samples also show closure of the pores in all the samples after compression.

4.2.1 Bulk Porosity

The porosity of the samples which is the total pore volume of all the pores in each sample is presented as percentages in Fig. 11 at pre-compression and post-compression stages. The pre-compression porosity is seen to vary with the samples, this shows the effect of the different phaseCO2-br on the porosity of the rock during saturation. All samples containing brine showed a significant reduction in porosity compared to the dry sample, this indicate that the brine solutions caused closure of pores by precipitation of fines. This is contrary to Foroutan et al. (2020) who observed a 3.6–2.87% increase in porosity (dissolution). This difference may be due to the mineralogy of the rocks. There was calcite and anorthoclase feldspar in the samples used by Foroutan et al. (2020) while those were absent in the sample used for this research. Aside from the fact that calcite readily dissolves in an acidic medium, anorthoclase is known to be stable only at very high temperatures, hence they are likely to have dissolved at the temperature under which the test was performed. In addition, the brine solution used by Foroutan et al. (2020) is a mixture of NaCl, KCl, CaCl2, Na2SO4, MgCl2, and MgSO4 and this will understandably trigger a different effect compared to the NaCl brine used in this research. It is also seen that there is a reduction in porosity after compression for all the samples. However, the amount of reduction varies according to the state of the sample. For instance, the gCO2-br-saturated rock undergoes the greatest reduction in bulk porosity (~13%), followed by both scCO2-br- and gCO2-saturated rocks, at 10% each (Figs. 9 and 11).

In this research, the need for all parameters especially the porosity of all the samples to be well characterized. The initial connected porosities of all the samples used are similar as seen in Table 5. The initial total porosity (\(\phi_t\)) was then estimated from the measured density and the mineral composition of the samples using:

\[
\phi_t = \left(1 - \frac{\rho_b}{\rho_p}\right) \times 100, \tag{6}
\]

where \(\rho_b\) is the bulk density and \(\rho_p\) is the particle density taken as the average density of the minerals that make up the rock i.e. 2.58 g/cm³.

The initial total porosity of all the samples before the commencement of the experiments showed that the samples had identical porosities (Table 5). The initial connected porosities measured from all the core samples using saturation and buoyancy technique are also similar.

The measured porosity from the digital rock physics (Fig. 11) is higher than that measured by saturation and buoyancy method because some of the pores are unconnected and so are not measured by the saturation and buoyancy method. However, such closed pores are measured by a well-resolved digital rock physics method. Since the initial connected porosity measured from the saturation technique (Table 5) and the initial total porosity estimated from the density of all the samples (Table 5) are similar in each case, it implies that the porosity of the dry sample measured using the digital rock physics represents the initial porosity of the other samples when measured digitally before commencement of the experiments. Therefore a comparison of the changes in porosity measured digitally before compression and after compression is shown in Fig. 11.

The pre-compression porosities shown in Fig. 11 are seen to be different from each other, with the dry sample having the highest porosity of 34%. The difference in porosity is due to precipitation of fines from the different phaseCO2-brine-rock reaction, which clogs the pores as seen in Fig. 12. The reduction in porosity after compression as shown in Fig. 11 is a resultant effect of both precipitation of fines that clogs the pores (Fig. 12) and the closure of pores due to compression. The precipitates are a thought to be a product of dissolution of feldspars by the CO2-brine as Akono et al. (2019) had reported the dissolution of K-feldspars in a CO2-brine-saturated rock. Pimienta et al (2017) reported dissolution after two hours exposure of rocks to supercritical CO2 and precipitation after four hours of exposure. This implies that the exposure time of CO2 and reactivity of the rock minerals affect the nature or stage of rock-CO2-brine reaction (dissolution or precipitation). Differences in exposure time of CO2 and reactivity of the minerals explains why some research report dissolution (Foroutan et al 2020) while others report precipitation (Pimienta et al 2017).

Figures 8 and 9 reveal that there is a relationship between volumetric strain and change in porosity. Amongst the samples that contain CO2 and/or brine, it is seen that the total
Fig. 10 SEM images showing the microstructures at pre-compression and post-compression for each of the samples. Note that the pre-compression images were taken after saturation of the sample with the different phase CO$_2$-brine.
volumetric strain is in the order: gCO₂-br sample > brine sample > scCO₂-br sample > gCO₂ sample. It is also seen that the gCO₂-br sample had the least porosity after compression. Similar relationship between volumetric strain and the change in porosity has been reported by Wong (1990), however, it was observed in this study that the reduction in porosity as a function of volumetric strain is higher in all the CO₂-bearing samples compared to the brine sample.

CO₂-brine has been shown to trigger chemical precipitation or dissolution (Olabode and Radonjic 2013, 2017) or swelling (Fuchs et al. 2019). This either decreases or increases the porosity of the rock. The porosity after saturation (pre-compression) was seen to be reduced but the reduction varied according to the phaseCO₂-br. For instance, compared to the dry sample, the porosity of br, gCO₂-br, scCO₂-br, and gCO₂ samples in Fig. 11 was lower by 14%, 15%, 11% and 1% respectively after saturation. After compression, the porosity of the br, gCO₂-br, scCO₂-br, and gCO₂ samples was lower by 13%, 15%, 6% and higher by 4%, respectively. These results imply that the presence of brine cause a reduction in porosity of reservoir rocks under stress but this reduction is further accelerated by the presence of CO₂ (gCO₂-br > scCO₂-br), that gCO₂-br-saturated sample showed the highest reduction in porosity after saturation and after compression indicates that the process responsible for change in porosity in this research (precipitation) is most active if the CO₂ in the brine saline reservoir is gaseous compared to the other phases. This result shows that the saline reservoir in its natural brine-containing state is undergoing change in porosity which is further triggered by CO₂, most especially the gaseous CO₂.

4.2.2 Cumulative Percentage of Pore Volume

Figures 13 are the pore volume distribution (PVD) of the different classes of pore volumes for pores whose volume are between 2e−5 mm³ and 3.4e−04 mm³. A change in the PVD of the pores after compression is an indication of complete closure, contraction, or expansion of the pores.

For the gCO₂-br-saturated rock as seen in Fig. 13, there was a significant downward movement of the PVD curve after compression for all the classes of pore volume. The pre-compression and post-compression PVD for the gCO₂-br showed the highest deviation from each other compared to the other curves. This implies that there was a significant reduction in the population of pores in all the classes of pore volumes after compression due to pore filling or pore closure. Before compression, there was no significant difference between the PVD of the gCO₂-br and brine sample. However, after compression, there was a significant difference in the PVD between the two samples for all the classes of pore volumes.

For the scCO₂-br-saturated rock as seen in Fig. 13, there was upward movement of the PVD curve after compression for smaller classes of pore volume. This suggests that there was an increase in the population of smaller pores after compression due to pore filling or pore closure. Before compression, there was no significant difference between the PVD of the scCO₂-br and br samples. However, after compression, there was a significant difference in the PVD between the two samples for all the classes of pore volumes.

For the gCO₂-saturated rock as seen in Fig. 13, there was downward movement of the PVD curve after compression for smaller classes of pore volume. This suggests that there was an increase in the population of smaller pores while the population of the larger pores remained the same. Increase in the population of smaller pore is suggestive of contraction of some larger pores to smaller ones. There was a significant difference in the PVD of scCO₂-br and br samples for the smaller classes of pore volumes before and after compression. Similarly, Huang et al. (2020) have reported increase in micro-pores after scCO₂ injection into brine-saturated sandstone sample.

For the gCO₂-saturated rock as seen in Fig. 13, there was downward movement of the PVD curve after compression for smaller classes of pore volume. This suggests that there

| Sample     | Dry  | br   | gCO₂-br | scCO₂-br | gCO₂  |
|------------|------|------|---------|----------|-------|
| Initial total porosity (Ø) (%) | 31   | 32   | 31      | 31       | 31    |
| Initial connected porosity (Ø) (%) | 26   | 27   | 26      | 27       | 26    |
| Bulk modulus (GPa) | 1.8  | 1.8  | 1.6     | 1.4      | 1.6   |
was a decrease in the population of smaller pores while the population of the larger pores did not change significantly. Decrease in the population of smaller pore is suggestive of expansion or coalesce of some smaller pores to form larger ones. There was a significant difference in the PVD of gCO$_2$ and br samples for the smaller classes of pore volume before and after compression. There was higher population of smaller pores in the gCO$_2$ sample compared to the brine.

4.2.3 Pore Surface Flatness

The saturation of rocks with brine-CO$_2$ and compression affect pore surface flatness. Flatness index approaching zero indicates flat pore surface while flatness index approaching one indicates wavy pore surface. Figure 14 shows the cumulative percentage count of pores for all classes of flatness index after saturation and after compression for all the samples. After saturation, the percentage count of pores in all classes of flatness index was similar for all the samples, irrespective of the nature of saturation. After compression, the percentage count of pores in each class of flatness index is similar for all samples of rock except the scCO$_2$-br, where the percentage count of pores with flatness index greater than 0.37 differ considerably (Fig. 14). This indicates that there are more pores with index above 0.37 in the scCO$_2$-br rock compared to the others after compression. This implies
that reservoir with scCO$_2$-brine have the tendency to make pore surface become wavy under stress, which could be because scCO$_2$ has a higher density compared to gCO$_2$, and scCO$_2$ dissolve edges of organic materials more easily (Chitanvis et al. 1998). Similarly, He et al. (2019) observed from their hydraulic fracturing experiment that scCO$_2$ creates more complex fracture and micro-cracks.
4.2.4 Shape

The percentage count of pores belonging to each shape (aspect ratio) after saturation and after compression is shown as curves. After saturation, percentage count of pores belonging to each shape in the CO₂-bearing samples compared to the brine sample is largely similar (Fig. 15). This implies that the shape of pores may be insignificantly affected by the different phaseCO₂-brine during saturation. However, after compression (Fig. 15), there was a significant difference in the percentage count of pores in both sub-rounded and rod-like shaped pores when the brine sample is compared to other CO₂-bearing samples. This difference was also seen to vary according to the phaseCO₂-brine. For instance, the difference is highest in the gCO₂ sample followed by the scCO₂-br sample (Fig. 15). Generally, there was an increase in the percentage of sub-rounded and rod-like shaped pores in the gCO₂ and scCO₂-br samples after compression as seen in Fig. 16.

There was no rounded pore in the sample and this may be a reflection of diagenetic processes. Delle Piane and Sarout (2016) reported no major difference in terms of the statistical distribution of shape descriptors in the identified pores and grains of minerals except kaolinite. However, our findings indicate that some pores in the scCO₂-br- and gCO₂-saturated samples showed significant alteration in shape after compression.

4.2.5 Equivalent Pore Radius and Sphericity

The distribution of the equivalent radius of the pores as well as the distribution of the sphericity within each class of pore radius is shown in Fig. 17 and Appendix. In all the samples, spherical pores dominate and the sphericity of smaller sized pores remained largely unchanged even after compression. However, the sphericity of the larger-sized pores changed significantly. In the dry sample, compression led to a decrease in radius and sphericity of most pores, larger pores became less spherical. In the brine sample, compression led to closure of some larger pores. In the CO₂-bearing samples, compression led to the closure and reduction of the sphericity of more pores compared to the brine sample. The change in pore size and sphericity is seen to depend on the phaseCO₂-brine state of the sample and this has implications for porosity, pore connectivity, transport properties, and storage capacity of the reservoir.

4.2.6 Hydro-Chemo-Mechanical Framework for the Change in the Rock Properties

Earlier, it was discussed that the pore fluid and its pressure triggers geochemical reaction between the minerals and the CO₂-brine. In this research, the change in pore geometry is due to the pore fluid and its pressure, the associated geochemical reaction, and the change in the mineral composition. The different phaseCO₂-brine triggers different geochemical reaction that causes different change in the mineral composition of the rocks as seen in Table 5. This change in mineral composition is then reflected as changes in mechanical properties such as the different stress–strain behaviour (Fig. 8), bulk modulus (Table 5) as well as porosity and pore geometry. For instance, Quartz is the most stable mineral in the rock. It is the cementing agent and accounts for most of the strength of the rock. The fact that the wt. % of Quartz is lowest in the scCO₂-br sample means that the Quartz is most degraded by the scCO₂-br compared to the others. Consequently, it also had the lowest bulk modulus. This agrees with Vanorio et al. (2011) who opined that compositional
changes control the change in the fabric as well as other changes in rock’s properties, and that injecting CO₂ into brine-rock system-induced chemo-mechanical processes that permanently alters the rock’s framework.

5 Conclusions

Changes in pore geometry of sandstone in CO₂ storage conditions were evaluated with different phaseCO₂-brine. The results showed that presence of CO₂ in saline rocks affect the geometry of pores, but the scCO₂-br saturation caused the highest change in the flatness of pore surface and shape of pores. This is because scCO₂ has a higher density compared to gCO₂ and it dissolves edges of organic materials more easily. The lowest bulk modulus is seen in the scCO₂-br sample (i.e., 12.5% less than the bulk modulus of brine-saturated sample). The gCO₂-br saturation caused the greatest reduction in porosity (~15%), with the highest volumetric strain. This is because at both low and high stress levels, the gCO₂-br affect the intergranular/effective strength of the rocks more than the other phaseCO₂-br. The change in pore volume is dependent on the phase of CO₂. Compression reduces the pore volume of phaseCO₂-brine-saturated rock but the reduction in pore volume is also influenced by the CO₂ phase. Precipitation as a result of CO₂-brine-rock interaction also contributed to the reduction in porosity. Presence of CO₂ increased the strain recorded in the samples. This implies that CO₂ affect the geomechanical behaviour of rocks. More specifically, a reservoir containing gCO₂-br will undergo significant strain at both shallow and greater depth with about 70% more strain at the greater depth, while a reservoir containing scCO₂-br will show comparatively lower strain at both shallow and greater depth with about
80% less strain at the greater depth. The result of this work can be useful for predicting the change in the geometry of pores and the strain of saline reservoir rocks in planned and already existing giant CO₂ storage sites such as Sleipner, In Salah, etc. Understanding of the change in the geometry properties of pore is critical to understanding the change in transport, physical and geomechanical properties of the reservoir rocks. Further research is recommended to model pore geometry control of the geomechanical and transport properties of reservoir rocks in a geosequestration site using the different possible phase CO₂-brine.

Fig. 17 Distribution of equivalent pore radius and sphericity for (a) brine sample at precompression, (b) brine sample at post-compression, (c) gCO₂-br sample at pre-compression, (d) gCO₂-br sample at post-compression. (Notice that the pre-compression samples in both cases had more spherical pores compared to the post-compression samples)
Effect of CO₂ Phase on Pore Geometry of Saline Reservoir Rock

Appendix

See Fig. 18.

Fig. 18 Distribution of equivalent pore radius and sphericity for (e) Dry sample at pre-compression, (f) Dry sample at post-compression, (g) scCO₂-br sample at pre-compression, (h) scCO₂-br sample at post-compression, (i) gCO₂ sample at pre-compression, (j) gCO₂ sample at post-compression
Author contributions AP: Conceptualization, methodology, formal analysis, investigation, writing—original draft, writing—review and editing. XJ: Formal analysis, writing—review and editing, visualization. XF: Writing—review and editing, visualization. KI-IE: Writing—review and editing, formal analysis, visualization. YS: Writing—review and editing, visualization. DY: Conceptualization, methodology, writing—review and editing, visualization, supervision, software, project administration.

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Data availability Data are available. The remnant material has been disposed.

Code availability Not applicable.

Declarations

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Consent to participate All authors are consented to participate in this research.

Consent for publication All authors have given consent to publication of this research.

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