Multi-scale experimental study of carbonated water injection: An effective process for mobilization and recovery of trapped oil

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HIGHLIGHTS

- Gas exsolution during carbonated water injection (CWI) is studied.
- Experiments are performed at macro and micro scales using X-ray CT imaging.
- Oil recovery factor increases significantly due to gas exsolution during CWI.
- CWI has applications in both environmental engineering and petroleum engineering.
- CWI offers a possibility for CO2 sequestration in petroleum oil reservoirs.

ABSTRACT

Steady flow of a disconnected gas phase (bubbles) is realized in porous media during carbonated water injection (CWI) under conditions that promote continuous exsolution of the dissolved gas. Using microfluidic pore networks etched on glass as well as a miniature core-flooding setup integrated with micro computed tomography (CT) imaging apparatus, we demonstrate capillary interactions of the flowing gas bubbles with a previously trapped oil phase (three-phase ganglion dynamics), which lead to mobilization of oil ganglia and remarkably high oil recovery. When three-phase ganglion dynamics are induced by carbonated water injection in low-permeability Berea sandstone core samples containing waterflood residual oil, more than 34% and 40% of the original oil in place additional recoveries are achieved in macro- and micro-scale flow tests, respectively, while a significant amount of CO2 is permanently sequestered in the pore space as capillary-trapped and dissolved gas. It is observed that when oil globules come into contact with CO2, they form thick spreading layers between brine and gas and are carried by moving gas clusters. The oil layers stay stable until the gas clusters leave the medium. Individual oil and gas blobs captured during micro-CT imaging are statistically analyzed to further examine underlying pore-level displacement physics of the process.

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1. Introduction

Sustained production of oil from a subsurface reservoir, initially driven by natural formation pressure and expansion of oil and reservoir rock, requires injection of water or gas in order to maintain reservoir pressure and displace oil toward production wells [1]. In the majority of cases, this so-called secondary recovery is achieved by waterflooding, a technique first considered in 1880 [2]. Waterflooding is a process of immiscible displacement leaving behind a significant amount of the original oil in place (OOIP), typically 30–50% in water-wet systems, in the form of disconnected oil ganglia strongly held within rock pores by capillary forces [3]. Of various enhanced oil recovery techniques targeting this trapped oil, injection of CO2 [4,5] has recently received renewed attention. CO2 is also a greenhouse gas that may be captured and sequestered [6]. Recent estimates indicate significant potential for CO2-based EOR for recovery of large volumes of incremental oil from watered-out reservoirs alongside storage of equally large volumes of CO2 [7].

The current paradigm of large-scale CO2 sequestration is injection of CO2 as a separate, usually supercritical, phase. The same is true of CO2 injection for enhanced oil recovery. Recently, however, attention has been drawn to the alternative of dissolving the CO2 in brine at the surface and injecting carbonated brine instead of CO2 [8,9] – an approach that would mitigate the risks associated with
buoyant migration of a free CO₂ phase since CO₂ is dissolved in brine and carbonated brine is denser than the native brine. Carbonated water injection (CWI) for oil recovery at the secondary and tertiary (EOR) stages was proposed more than fifty years ago [10]. Interest in this process has been recently renewed [11] given the potential for coupling oil recovery with CO₂ sequestration. Previous pore-scale visualization studies in glass micromodels [12] have shown that transfer of dissolved CO₂ from carbonated brine to oil causes swelling and coalescence of isolated oil ganglia, and that oil redistribution leads to local flow diversion. In combination with a reduction in the oil viscosity, these processes instigate displacement of additional oil. Sohrabi et al. [11] reported 10% additional recovery of the original oil in place by CWI in a previously waterflooded high-permeability core sample, while nearly half of the injected CO₂ was permanently stored. This was achieved by direct transfer of CO₂ from brine to oil without appearance of a free CO₂ phase. In other words, the impact of CO₂ exsolution during CWI and the effects of free gas on the mobilization of oil ganglia were overlooked. It was concluded that the success of CWI as an enhanced oil recovery method depends on deliverability of the dissolved CO₂ to the trapped oil [12,11]. Alizadeh et al. [13] presented the results of an experimental study where carbonated water injection and subsequent degassing in situ development of a gas phase, as a result of pressure depletion, were used to mobilize and recover residual oil in Berea sandstone. It was reported that the gradual increase in the pressure drop led to liberation of gas from the aqueous phase, internal gas drive, mobilization of oil ganglia, and reduction of residual oil saturation. Parallel pore-scale visualization studies using transparent glass micromodels indicated that the effectiveness of the approach was linked to the interaction between a flowing, disconnected gas phase and oil ganglia (three-phase ganglion dynamics). In addition to oil recovery, the process offered the possibility for simultaneous CO₂ sequestration in the pore space as capillary-trapped and dissolved gas. The experiments were conducted at ambient temperature and relatively low pressure. Zuo and co-workers [14–16], in a series of experimental studies, also investigated gas exsolution from carbonated water in micromodels and sandstone rocks but at elevated pressure and temperature conditions. It was observed that gas exsolution in water-filled pores resulted in water flow blockage, local water flow diversion into oil-filled elements, and mobilization of trapped oil. Additional oil recovery of 10% was obtained when this process was applied to a Berea core sample. The researchers mention that, under reservoir conditions, the exsolved gas phase has little or no mobility and its relative permeability remains very low, in the order of 10⁻³ to 10⁻², even at moderate gas saturations. Therefore, they conclude that, rather than flowing, the gas phase acts as a flow barrier (similar to a mobility control agent) and diverts water flow to oil-filled pores, thereby displacing oil. Expansion of gas bubbles was also considered to improve oil recovery by displacing trapped oil globules. Due to the applicability of CWI in environmental engineering and to understand the displacement physics under simplified conditions, we intend to investigate this process first at low pressure and temperature conditions prevailing at the groundwater table. However, we will address potential applications of the CWI process in petroleum oil reservoirs as well, even though the conditions relevant to petroleum engineering applications are the subject of our future studies. In this study, we focus on the impact of in situ degassing during carbonated water injection on oil recovery at conditions more relevant to the remediation of petroleum-contaminated zones. For this purpose, we deploy an array of experimental techniques to study this process and shed light on some of the subtle displacement physics. We use experiments in a two-dimensional micromodel as well as flow tests in naturally-occurring rocks with various sample sizes. For the latter, we utilize X-ray computed tomography (CT) techniques with significantly varying resolutions for in situ saturation measurements. We perform a set of proof-of-concept experiments on a large core sample (macro scale) to examine the effectiveness of CWI in recovery of trapped oil from a waterflooded core sample. We then conduct micro-scale flow tests (both in a small rock core sample and in a glass micromodel) to investigate the pore-level displacement physics responsible for the trends observed at the macro scale. We report pore-scale observations of three-phase flow involving two immiscible disconnected non-wetting phases (i.e., oil ganglia and gas bubbles) and one connected wetting phase (i.e., water). Steady flow of the discontinuous gas phase is established by carbonated water injection under conditions that promote in situ degassing [22,23]. This is a non-equilibrium process driven by supersaturation of the aqueous phase with CO₂, namely by the condition H – P_CO₂ > 0, where C and P are the local CO₂ concentration and pressure of the aqueous phase, respectively, and H is Henry’s constant. We demonstrate that oil ganglia are effectively and immiscibly mobilized by flowing gas bubbles. Additional recoveries of more than 34% and 40% of OOIP at the macro and micro scales, respectively, are achieved when this process is applied to low-permeability sandstone cores containing trapped oil due to a waterflood.

In the following sections, we first provide the specifics of the porous mediums and fluids used in the experiments at different scales. We then describe the details of experimental setups and procedures deployed to perform the flow tests. This is followed by the results of the experiments and discussion. We conclude the paper with a set of final remarks.

2. Flow experiments

Experiments presented in this paper were performed at two scales: macro (Category A) and micro (Category B). At the macro scale, we used a long consolidated naturally-occurring sandstone core sample to perform a set of core-flooding experiments in order to examine the effectiveness of the immiscible CWI (in situ degassing and consequent three-phase ganglion dynamics) in recovering trapped oil at two different carbonation pressures. We then used micro-scale studies to examine some of the complex pore-scale phenomena leading to the trends observed in the experiments under Category A. We performed miniature core-flooding experiments (Category B) in a similar, but smaller, core sample while using a micro-CT scanner to obtain high-resolution images of pore-scale fluid occupancies. We also investigated, still under Category B, more details of the pertinent displacement mechanisms using a two-dimensional glass micromodel.

Below, we present details of the porous mediums, fluids, and experimental conditions, setups, and procedures utilized in each of the above-mentioned categories of experiments.

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2.1. Porous mediums and fluids

**Macro-scale flow experiments (Category A)** – A core plug, 254 mm in length and 38.1 mm in diameter (Sample A), was cut from a block of Berea sandstone using tap water as a coolant, dried in an oven at 110 °C for three days and then cooled in a desiccator for two days. The three-dimensional X-ray images of the sample showed that it was nearly homogeneous, except for a thin heterogeneous layer at one end. Brine permeability of the core was 89.08 mD and its average porosity determined using X-ray imaging was 20.77%. The porosity value was consistent with an independent measurement made through helium porosimetry on another core sample cut from the same block of Berea sandstone. Details of porosity determination using X-ray imaging are provided later in this paper. Selection of a core sample with these length and permeability was intended to establish high pressure drop at relatively low flow rates during the degassing experiments.

The aqueous phase was formulated using distilled water, 2 wt% CaCl₂, 12 wt% NaI, and 0.01 wt% NaN₃. Sodium azide was added to brine as a biocide. Brine density was 1.116 g/cm³ at ambient pressure and temperature. Soltrol 170 (a mineral oil) was first purified by passing it through a dual-packed column of silica gel and alumina to remove polar contaminants, which might alter the water wetness of the rock, and then iodooctane (5 vol%) was added to the purified Soltrol to obtain the working oil phase of density equal to 0.804 g/cm³. All chemicals were reagent grade and used as received. CO₂ was used as the gas phase. It was withdrawn from a pressurized cylinder of 99% purity. In the rock-fluid system selected, the aqueous, oil, and gas phases were the wetting, the intermediate-wetting, and the non-wetting phases, respectively.

In the course of the macro-scale core-flooding experiments, two- and three-phase in situ saturations were measured using a macro-CT scanner. Accordingly, sodium iodide and iodooctane were added as X-ray dopants to the aqueous and oil phases, respectively, in order to better distinguish the fluid phases.

### Table 1
Geometrical and petrophysical properties of the core samples used in the core-flooding experiments.

<table>
<thead>
<tr>
<th>Experiment category</th>
<th>Core</th>
<th>Length (mm)</th>
<th>Diameter (mm)</th>
<th>φX-ray (fraction)</th>
<th>φhelium (fraction)</th>
<th>Kbrine abs (mD)</th>
<th>Khelium abs (mD)</th>
<th>Pore volume (cm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Berea</td>
<td>254</td>
<td>38.1</td>
<td>0.208</td>
<td>-</td>
<td>89.08</td>
<td>89.08</td>
<td>60.146</td>
</tr>
<tr>
<td>B</td>
<td>Berea</td>
<td>95</td>
<td>3.72</td>
<td>0.185</td>
<td>0.19</td>
<td>89.50</td>
<td>88.35</td>
<td>0.192</td>
</tr>
</tbody>
</table>

### Table 2
Chemical composition of the fluids used in the core-flooding experiments.

<table>
<thead>
<tr>
<th>Experiment category</th>
<th>Phase</th>
<th>Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Brine</td>
<td>Distilled water + 2 wt% CaCl₂ + 12 wt% NaI + 0.01 wt% NaN₃</td>
</tr>
<tr>
<td></td>
<td>Oil</td>
<td>Soltrol 170 + 5 vol% iodooctane</td>
</tr>
<tr>
<td></td>
<td>Gas</td>
<td>CO₂</td>
</tr>
<tr>
<td>B</td>
<td>Brine</td>
<td>Distilled water + 20 wt% NaI</td>
</tr>
<tr>
<td></td>
<td>Oil</td>
<td>Soltrol 170 + 7.5 vol% iodooctane</td>
</tr>
<tr>
<td></td>
<td>Gas</td>
<td>CO₂</td>
</tr>
</tbody>
</table>

**Fig. 1.** Experimental setup used to perform the macro-scale core-flooding experiments [24]. A similar flow system was utilized for the core-flooding experiments at the micro scale.
Micro-scale flow experiments (Category B) – A 95-mm long, 3.72-mm diameter core sample (Sample B) was cut from a 38.1-mm diameter core plug from the same block of Berea sandstone used to obtain Sample A. Prior to cutting the small sample, porosity and permeability of the 38.1-mm diameter core plug were measured with helium gas. The values were then compared with those obtained from dry, high-resolution imaging and flow experiments in Sample B. Table 1 lists petrophysical and geometrical properties of the core samples used in the core-flooding experiments of this study.

For the miniature core-flooding experiments, the aqueous and oil phases were prepared by adding 20 wt% NaI and 7.5 vol% iodoctane to distilled water and purified Soltrol 170, respectively. The levels of dopant concentrations were determined to make segmentation of the three fluid phases (i.e., brine, oil, and CO₂) possible. The physical properties of the fluids are listed in Table 2. The fluid system was believed to be spreading even though interfacial tensions between the fluids were not measured. This was confirmed by formation of spreading oil layers sandwiched between brine and CO₂ during the micro-scale core-flooding experiments.

A series of flow experiments was also carried out on a two-dimensional etched glass micromodel with a water/oil/CO₂ fluid system. The medium included an irregular network of pores connected by throats. It was equipped with a transparent glass cover that allowed monitoring the displacement physics during various steps of the experiments using a high-speed camera.

<table>
<thead>
<tr>
<th>Recipe</th>
<th>Scanned length (mm)</th>
<th>Number of projections</th>
<th>Resolution (μm)</th>
<th>Exposure time (s)</th>
<th>Camera binning</th>
</tr>
</thead>
<tbody>
<tr>
<td>Test scans</td>
<td>2.975</td>
<td>3000</td>
<td>3.4</td>
<td>0.75</td>
<td>2</td>
</tr>
<tr>
<td>Main scans</td>
<td>2.625</td>
<td>4500</td>
<td>1.5</td>
<td>6</td>
<td>1</td>
</tr>
</tbody>
</table>

Table 3
X-ray microtomography parameters used in different recipes to image Sample B during the micro-scale core-flooding experiments.

Fig. 2. Slice-averaged saturation distributions along the length of Sample A: (a) at the end of primary oil drainage and unadulterated brine injection (○: initial water saturation, ◦: waterflood residual oil saturation) and (b) at the end of unadulterated brine injection (○) and the first (●) and second (▲) CWI processes.
undesirable vibrations/torque on the core holder, which could detrimentally reduce the quality of images, by using ultra-flexible tubing. The imaging system was a VERSA-XRM500™ micro-CT scanner manufactured by Xradia with six different objectives (0.4×, 1×, 4×, 10×, 20×, and 40×). The X-ray source could reach a voltage of 30–160 kV and a power of 1–10 W. A resolution of 500 nm to 64 μm was attainable depending on the optical and geometrical magnifications used. An ANDOR™ camera with different camera binning (1, 2, 4, and 8) was used to obtain tomography projections.

2.3. Experimental procedures

The key objective of the study was to assess the extent of recovery of waterflood residual oil by in situ gas exsolution from carbonated brine during pressure depletion. To do so, the established residual oil was surrounded by CO2-saturated brine, and then pore pressure was gradually reduced using a tightly-controlled back pressure pump allowing gradual exsolution of the dissolved CO2. One could consider doing this in both ‘dynamic’ and ‘static’ modes meaning with and without injection of additional CO2-saturated brine. We selected the former mode seeking to achieve the maximum possible recovery of trapped oil. All the displacements were carried out on samples oriented vertically at ambient temperature. A net overburden pressure of 250 psi was applied during all core-flooding experiments.

For macro-scale experiments, the core was placed in the core holder under overburden pressure and flooded with CO2 gas to remove air. Then, the core was vacuum saturated with unadulterated brine. The core was oilflooded from the top to establish initial brine saturation and then waterflooded from the bottom with unadulterated brine to establish residual oil saturation. Both of these steps were performed at a highly stable back pressure of 90 psig. During primary oil drainage, the oil flow rate was gradually increased to a maximum value of 0.8 cc/min. During the subsequent waterflood, the brine flow rate was increased very slowly to minimize undesirable dynamic effects on maximum residual oil saturation that could be established. The maximum brine flow rate was 0.2 cc/min corresponding to a capillary number of 10⁻⁶. The core was considered to be at residual oil saturation when oil saturation did not change by doubling the brine flow rate. In the oilflood, as in the unadulterated brine injection, the pump was retracting oil from a bucket as the oil did not need to be at

Fig. 3. Macro-CT images showing oil saturation distributions at the end of primary drainage (first row from top), unadulterated brine injection (second row), first carbonated water injection (third row), and second carbonated water injection (fourth row). So, L, and BP represent slice-averaged oil saturation, the distance of the slice from the bottom of the core, and back pressure, respectively.
equilibrium with CO₂ for the CWI step. Prior to the experiment, the separator was filled with unadulterated brine and CO₂ at 90 psig, and both phases were recirculated through a line bypassing the core sample to reach equilibrium at this pressure.

The CWI process was started using constant flow rate mode during which carbonated brine was injected from the bottom of the core at 0.05 cc/min. This low flow rate ensured no oil mobilisation prior to depressurization. After injecting around one pore volume of carbonated brine into the core, the back pressure, initially set at 90 psig, was reduced in small increments, while injection of carbonated brine was continued. This approach was maintained, and the fluid saturations were measured regularly to monitor exsolution of CO₂ and possible reductions in oil saturation. During the process, for a given brine flow rate and pressure drop, fairly stable oil saturation was achieved after a relatively long time (e.g., 6–18 h). The slow reduction in back pressure was continued until the pressure drop across the core reached about 88 psi, when complete exsolution of gas took place. Since liberation of gas enhanced the differential pressure and caused the inlet pressure to increase for a given flow rate, it gave the opportunity to decrease the back pressure further to reduce the inlet pressure to 90 psig. It should be noted that, during this process, the injection flow rate was never allowed to increase above values corresponding to capillary numbers greater than 10⁻⁶.

In order to investigate if higher carbonation pressure, i.e., a higher level of CO₂ dissolution in brine, could further decrease the new oil saturation, the experiment was repeated (still under Category A) with a new brine saturated with CO₂ at 180 psig. The only difference with the previous step was that constant pressure mode was selected to operate the brine pump as more insight into possible variations of the injection flow rate had been developed. The back pressure was decreased gradually from 180 to 17 psig, i.e., a pressure drop of 163 psi across the length of the core.

The micro-scale core-flooding experiments were carried out with an experimental procedure similar to that of the macro-scale experiments, but with smaller flow rates and a slightly different initial carbonation pressure (i.e., 92 psig). After obtaining a dry
reference image, the core sample was vacuum saturated with unadulterated brine and then oilflooded to establish initial brine saturation. During primary oil drainage, the oil flow rate was gradually increased and the core sample was regularly scanned at a resolution of 3.4 \( \mu \text{m} \) (test scans) to obtain approximate values of phase saturations. At the maximum oil flow rate of 0.03 cc/min, the system was allowed to reach steady state, which was identified from stable pressure drop and similar fluid configurations from consecutive test scans. After establishing steady state, the sample was scanned at a higher resolution of 1.5 \( \mu \text{m} \) to accurately determine initial brine saturation and fluid occupancies. The subsequent waterflood was performed under capillary-dominated flow regime, and the brine flow rate was very slowly increased to a maximum value of 0.017 cc/min. At this point, the waterflood residual oil saturation had been established and therefore a high-resolution image of the sample was attained. Thereafter, unadulterated brine was displaced with carbonated brine saturated with CO\(_2\) at 92 psig, while back pressure was tightly maintained at 92 psig. The back

![Fluid occupancy maps during the micro-scale core-flooding experiments](image)

*Fig. 8. Fluid occupancy maps during the micro-scale core-flooding experiments (slice distance from bottom of the core = 47.31 mm; resolution = 1.5 \( \mu \text{m} \); brine:blue, oil:red, gas:yellow, and grains:gray). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)*
pressure was then reduced incrementally to 0 psig to allow gas exsolution. During pressure depletion, carbonated brine was continuously injected into the sample, while the sample was scanned at various saturation intervals.

For micromodel experiments, we applied constant pressure as boundary conditions at both inlet and outlet of the medium. Water was first equilibrated with CO₂ in a membrane contactor at ambient temperature and 70 psig. The equilibrated water was then injected into the pore network initially saturated with unadulterated brine and trapped residual oil. Residual oil had already been established by waterflooding the medium containing water and large quantities of well-connected oil. A flow rate controller was used to adjust the water flow rate according to the changes in pressure drop across the medium due to in situ exsolution of CO₂. Pore-level displacement physics of mobilization and recovery of waterflood residual oil due to injection of carbonated water was investigated with a spreading oil.

3. Data acquisition and analysis

During the macro- and micro-scale core-flooding experiments, we determined porosity of the core samples and in situ fluid saturations using X-ray imaging techniques. Details of data acquisition and processing at each scale are presented in this section.

3.1. Porosity and in situ saturation measurements at macro scale

In addition to the common application of in situ saturation measurements, an X-ray macro-CT scanner can be used to determine porosity distribution within a porous medium. To this end, the sample and the core holder need to be scanned only at one energy level when they are fully saturated with fluid 1 and when they are fully saturated with fluid 2. For simplicity, fluids 1 and 2 were selected here to be water (i.e., brine) and air. To measure porosity, the core holder was scanned when it was filled with air and when it was filled with water. Similarly, the core sample placed in the core holder was scanned when it was dry and when it was fully saturated with water. Porosity was then calculated from:

\[
\phi = \frac{C_{T_{cw}} - C_{T_{cg}}}{C_{T_{w}} - C_{T_{g}}}
\]

where \(C_{T_{cw}}\) and \(C_{T_{cg}}\) are the CT numbers of the core sample fully saturated with water and air, and \(C_{T_{w}}\) and \(C_{T_{g}}\) are the CT numbers of water and air (in the core holder without the core), respectively.

Fig. 9. Visualization of multiphase pore-scale fluid occupancies at different CWI steps during the micro-scale core-flooding experiments (slice distance from bottom of the core = 47.31 mm; resolution = 1.5 μm; brine:blue, oil:red, gas:yellow, and grains:gray). [For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.]
To calculate three-phase saturations, the scanner was first calibrated at two different energy levels, i.e., (1) 80 kV and 125 mA and (2) 130 kV and 100 mA. The core was scanned at both energy levels during the experiments, and saturations were determined by obtaining a simultaneous solution for the following system of equations [26]:

\[
A = \frac{(CT_{t1} - CT_{cg2})(CT_{o1} - CT_{cq1}) - (CT_{t2} - CT_{cg2})(CT_{o2} - CT_{cq1})}{(CT_{t1} - CT_{cg1})(CT_{o1} - CT_{cq1}) - (CT_{t2} - CT_{cg1})(CT_{o2} - CT_{cq1})} \quad (2)
\]

\[
B = \frac{(CT_{t1} - CT_{cq1})(CT_{cw2} - CT_{cg2}) - (CT_{t2} - CT_{cq1})(CT_{cw1} - CT_{cq1})}{(CT_{t1} - CT_{cq1})(CT_{cw2} - CT_{cg2}) - (CT_{t2} - CT_{cq1})(CT_{cw1} - CT_{cq1})} \quad (3)
\]

\[
C = \frac{(CT_{o1} - CT_{cq1})(CT_{cw2} - CT_{cg2}) - (CT_{o2} - CT_{cq1})(CT_{cw1} - CT_{cq1})}{(CT_{o1} - CT_{cq2})(CT_{cw2} - CT_{cg2}) - (CT_{o2} - CT_{cq2})(CT_{cw1} - CT_{cq1})} \quad (4)
\]

\[
S_w = \frac{A}{C} \quad (5)
\]

\[
S_o = \frac{B}{C} \quad (6)
\]

\[
S_g = 1 - S_w - S_o \quad (7)
\]

where \( S_i \) is the saturation of phase \( i \), \( CT \) is the CT number of the core sample containing all phases, and subscripts 1 and 2 refer to energy levels 1 and 2 (low and high energy levels). The terms \( CT_{cw}, CT_{co}, \) and \( CT_{cg} \) denote reference scans and are the CT numbers of the core fully saturated with water, oil, and gas, respectively.

Fluid saturations were determined at 22 locations equidistant along the length of the core, that is, the distance between two consecutive locations was 11 mm. Each slice had a thickness of 2 mm.

### 3.2. Micro-CT data acquisition and processing

For micro-CT imaging, the slice thickness was 1.5 \( \mu \)m and fluid saturations were obtained for 2.625-mm long portion in the middle of the core, i.e., 46–48.625 mm from the bottom of the core sample. In this subcategory of experiments, we used two types of scans: (1) test scans, which were two hours long to quickly monitor fluid saturations and (2) main scans, which had a higher resolution and were used for accurate saturation measurements and statistical analysis. Imaging parameters for each scan type were saved as a recipe including optimum source voltage and power, objective type, camera binning, tomography locations, exposure time, number of projections, and source and detector positions. Table 3 lists the parameters of the test and main scans.

The sample stage rotated during the scans to permit collection of a set of projections at different angles. A special sample drift correction and a secondary reference were applied to the projection sets to correct effects of sample movement (in the order of 0–5 pixels) and secondary ring artifacts, respectively. The collected

![Visualization of multiphase pore-scale fluid occupancies at different CWI steps during the micro-scale core-flooding experiments](image-url)

(a) CWI (back pressure = 92 psig) (b) CWI (back pressure = 80 psig)

(c) CWI (back pressure = 60 psig) (d) CWI (back pressure = 24 psig)

(e) CWI (back pressure = 8 psig) (f) CWI (back pressure = 0 psig)

Fig. 10. Visualization of multiphase pore-scale fluid occupancies at different CWI steps during the micro-scale core-flooding experiments (slice distance from bottom of the core = 47.47 mm; resolution = 1.5 \( \mu \)m; brine:blue, oil:red, gas:yellow, and grains:gray). [For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.]
tomography files were reconstructed using XM-Reconstructor to provide raw TIFF images for analysis. The main parameters and operations to reconstruct a tomography file included center shift, beam hardening, defect correction, ring removal, secondary referencing, global intensities, and Kernel smoothing filter size. Global intensities ranging from $-3271$ to $6560$ were used to assign close attenuations for each phase in images of various experiments. Each data set exported for image analysis included 1750 stacked slices.

Phase saturations, fluid occupancies, and statistical analysis of oil and gas globules were extracted from the raw data using AvizoFire™ 8 with a workflow based on histogram thresholding (histogram-based binarization). Histogram thresholding is a well-known method for micro-CT image analysis [27] and used by many researchers with a simple workflow, that is, they segment grains and different fluid phases by identifying peaks in the grey scale histogram of filtered flooded images [28]. This approach complicates the image analysis and results in poor segmentation due to close intensities of doped fluids (mainly brine) and grain. To mitigate this issue, we modified the conventional histogram thresholding workflow with the following steps: (i) preprocessing: we applied different filters to smoothen the raw data and reduce salt-and-pepper noises to avoid difficulties such as phase mixing during the segmentation step. In doing this, the non-local means filter produced the best results. A volume edit module assigned a zero value for the exterior and created a mask for arithmetic operations prior to the segmentation; (ii) registration: this step aligned the flooded image sets with the dry reference image in a way that the grains had the same pixel number in all of them. All the flooded image sets were registered, transformed, and resampled using the dry reference image in order to match them pixel-by-pixel prior to arithmetic operations; (iii) arithmetic operations: we combined the resampled flooded image sets with the pore map created from the dry reference image to extract distribution maps of fluids in the pore space. To do this, the pore map was multiplied by each flooded image set, pixel-by-pixel, to establish an image set with non-zero values for the fluids present in the pore space; (iv) segmentation: we used the histogram thresholding approach to construct a final five-phase labeled file that included exterior, grains, gas, oil, and brine; and (v) quantification and statistical analysis: slice-averaged and total saturations were calculated for brine, oil, and gas at the end of drainage, imbibition, and CWI processes. AvizoFire was used to extract and statistically analyze the individual oil and gas blobs for each data set. The three-dimensional visualizations, fluid volumes, surface areas, and vertical center of mass for oil and gas blobs were also determined.

4. Results and discussion

In this section, we present the results of macro and micro scale experiments discussed earlier. We assess the effectiveness of carbonated water injection and consequent in situ degassing in mobilization and recovery of trapped oil ganglia. We use observations at the micro scale to explain the trends observed at the macro scale. The relevant pore-scale displacement physics are also discussed.

4.1. Macro-scale flow experiments

Fig. 2(a) shows the distributions of initial brine and waterflood residual oil saturations across Sample A. At the end of primary oil drainage, the average brine saturation was about 31.6%. This was mainly the brine left in the corners, crevices, and small pores as

![Fig. 11. Volume of pores occupied by (a) oil and (b) gas versus pore radius during the micro-scale core-flooding experiments.](image1)

![Fig. 12. Cumulative contribution of various oil blob sizes during the micro-scale core-flooding experiments.](image2)
Table 5
Properties of the largest individual oil globules at the end of imbibition and CWI steps for the micro-scale core-flooding experiments (BP stands for back pressure).

<table>
<thead>
<tr>
<th>Process</th>
<th>Volume (mm$^3$)</th>
<th>Area (mm$^2$)</th>
<th>Vertical center of mass (µm from the inlet)</th>
<th>Ratio to total oil volume</th>
</tr>
</thead>
<tbody>
<tr>
<td>Imbibition</td>
<td>0.102</td>
<td>12.13</td>
<td>46540</td>
<td>0.071</td>
</tr>
<tr>
<td>CWI (BP = 92 psig)</td>
<td>0.102</td>
<td>12.13</td>
<td>46540</td>
<td>0.071</td>
</tr>
<tr>
<td>CWI (BP = 80 psig)</td>
<td>0.815</td>
<td>295.59</td>
<td>47536</td>
<td>0.771</td>
</tr>
<tr>
<td>CWI (BP = 60 psig)</td>
<td>0.751</td>
<td>302.92</td>
<td>47440</td>
<td>0.722</td>
</tr>
<tr>
<td>CWI (BP = 40 psig)</td>
<td>0.593</td>
<td>238.54</td>
<td>47515</td>
<td>0.617</td>
</tr>
<tr>
<td>CWI (BP = 32 psig)</td>
<td>0.180</td>
<td>73.85</td>
<td>47040</td>
<td>0.203</td>
</tr>
<tr>
<td>CWI (BP = 24 psig)</td>
<td>0.145</td>
<td>43.75</td>
<td>46542</td>
<td>0.182</td>
</tr>
<tr>
<td>CWI (BP = 16 psig)</td>
<td>0.199</td>
<td>80.64</td>
<td>47188</td>
<td>0.280</td>
</tr>
<tr>
<td>CWI (BP = 8 psig)</td>
<td>0.126</td>
<td>22.40</td>
<td>46364</td>
<td>0.233</td>
</tr>
<tr>
<td>CWI (BP = 0 psig)</td>
<td>0.046</td>
<td>18.24</td>
<td>46529</td>
<td>0.085</td>
</tr>
</tbody>
</table>

Table 6
Properties of oil globules at the end of imbibition and CWI steps for the micro-scale core-flooding experiments (BP stands for back pressure). The coefficient of variation is the ratio of standard deviation to the mean value.

<table>
<thead>
<tr>
<th>Process</th>
<th>Number of blobs</th>
<th>Min. volume (µm$^3$)</th>
<th>Max. volume (mm$^3$)</th>
<th>Mean volume (µm$^3$)</th>
<th>Coefficient of variation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Imbibition</td>
<td>7579</td>
<td>1002</td>
<td>0.102</td>
<td>189112</td>
<td>9.1</td>
</tr>
<tr>
<td>CWI (BP = 92 psig)</td>
<td>7579</td>
<td>1002</td>
<td>0.102</td>
<td>189112</td>
<td>9.1</td>
</tr>
<tr>
<td>CWI (BP = 80 psig)</td>
<td>10660</td>
<td>533</td>
<td>0.815</td>
<td>99172</td>
<td>79.6</td>
</tr>
<tr>
<td>CWI (BP = 60 psig)</td>
<td>14583</td>
<td>530</td>
<td>0.751</td>
<td>71281</td>
<td>87.2</td>
</tr>
<tr>
<td>CWI (BP = 40 psig)</td>
<td>17437</td>
<td>503</td>
<td>0.593</td>
<td>55069</td>
<td>81.5</td>
</tr>
<tr>
<td>CWI (BP = 32 psig)</td>
<td>19004</td>
<td>320</td>
<td>0.180</td>
<td>46534</td>
<td>31.1</td>
</tr>
<tr>
<td>CWI (BP = 24 psig)</td>
<td>20086</td>
<td>303</td>
<td>0.145</td>
<td>39773</td>
<td>28.6</td>
</tr>
<tr>
<td>CWI (BP = 16 psig)</td>
<td>19466</td>
<td>317</td>
<td>0.199</td>
<td>36560</td>
<td>42.4</td>
</tr>
<tr>
<td>CWI (BP = 8 psig)</td>
<td>16862</td>
<td>209</td>
<td>0.126</td>
<td>32151</td>
<td>6.3</td>
</tr>
<tr>
<td>CWI (BP = 0 psig)</td>
<td>8335</td>
<td>408</td>
<td>0.046</td>
<td>63966</td>
<td>9.5</td>
</tr>
</tbody>
</table>
the sample was believed to be strongly water-wet. Subsequent unadulterated brine injection led to an average residual oil saturation of 41%, i.e., 40.1% of the original oil in place recovery. This residual oil saturation is typical of waterflood residual oil saturation in water-wet low-permeability Berea sandstone [29,30]. The trapped oil clusters resided in large pores, as oil was the non-wetting phase with respect to brine, and were surrounded by unadulterated brine. Fig. 3 presents selected CT slices along the length of the core and their respective average oil saturations at the end of primary oil drainage and unadulterated brine injection (the first two rows). The images exhibit a fairly uniform distribution of fluids that is an indication of both the homogeneity of the core and capillary-dominated displacement regime.

Initial injection of carbonated brine, i.e., without CO₂ exsolution, resulted in miscible displacement of unadulterated brine. The CO₂-saturated brine therefore came into contact with trapped oil clusters in the core. Subsequent exsolution of CO₂ led to direct contact between CO₂ gas and the majority of trapped oil clusters, a key advantage of this method compared to a gas injection process during which gas has to first displace brine before gaining access to the trapped oil. It was observed that the first CWI process was able to recover about 51% of the original waterflood residual oil reducing oil saturation to about 20% (30.7% additional recovery of the original oil in place). This is a significant reduction in trapped oil saturation. The process also established about 26% CO₂ saturation in the pore space at the end of in situ degassing – a possibility that inspires the application of the CWI process for simultaneous CO₂ sequestration in addition to oil recovery in petroleum oil reservoirs. This was in addition to the amount of CO₂ dissolved in the brine (with a saturation of 54%) left in the core sample. The sample had a pore volume of 60.146 cm³ and solubility of CO₂ in the brine was about 0.01 g/cm³ at 90 psig and 20 °C [31]. Variations of average oil and gas saturations with pressure drop are shown in Fig. 4. The second CWI process, however, did not lead to significant reduction in oil saturation. The average residual oil saturation was about 17.3% resulting in additional recovery of 6.6% of the original waterflood residual oil (about 4% of the original oil in place). That is, higher differential pressure drop was not able to further reduce oil saturation. This point may be important from economic and operational points of view. Namely, the majority of incremental oil recovery took place at the lower carbonation pressure. The oil saturation distributions along the length of the core at the end of unadulterated brine injection and the first and second CWI processes are exhibited in Fig. 2(b). The difference between the two distributions on the right again highlights the success of the first CWI process in recovering significant fractions of the residual oil.

To verify saturation values obtained during three-phase flow, the outlet pressure of the core at the end of the first CWI process was gradually increased to 180 psig in order to dissolve the free CO₂ back into the brine. During this step, a large number of pore volumes of brine (equilibrated with CO₂ at 90 psig) was injected.
at a low flow rate (<0.2 cc/min). The sample was scanned several times to make sure that there was no free gas present before reporting oil saturation. The value was closely consistent with the oil saturation measured at the end of three-phase flow. Similarly, at the end of the second CWI process, the pressure of the system was increased to 360 psig and saturation values were obtained.

Fig. 3 also compares CT slices and their respective average oil saturations at the end of unadulterated brine injection to those at the end of the first and the second CWI processes, after the free CO₂ in the core was dissolved back into brine. It was observed that, during the first CO₂-saturated brine injection, about 25% of the original waterflood residual oil was recovered with only 60 psi pressure drop, 68% of the maximum pressure drop achieved, whereas the next 25% was recovered with 28 psi more pressure drop, totaling 88 psi. In other words, recovery of oil did not follow a linear relationship with pressure drop across the core in the CWI process and was more efficient at the late stages of the process. Fig. 5 shows variations of pressure drop across the core with time. Pressure drop increases significantly as oil is recovered and as back pressure is reduced. Even though recovery of oil acts to reduce the pressure drop, exsolution of CO₂ significantly increases it. This is mainly due to the fact that CO₂ occupies the center of pores and throats as the most non-wetting phase causing major blockage for water flow and initial reduction of water relative permeability [14,16]. This completely dominates the trend observed in the pressure drop.

4.2. Micro-scale flow experiments

In the micro-scale core-flooding experiments, primary oil drainage and the subsequent waterflood established an average initial water saturation of 32.8% and an average residual oil saturation of 43.7%, respectively, in the pore space. These values were comparable with those obtained during the macro-scale experiments. Both flow processes were carried out at 92 psig back pressure. After displacing unadulterated brine with carbonated brine, the back pressure, pre-set at 92 psig, was slowly decreased allowing CO₂ to exsolve.

Fig. 6 exhibits oil saturation distributions along the scanned length of Sample B. As observed, oil saturation gradually decreased from initial waterflood residual oil saturation of 43.7% to 16.3% at the end of CWI, i.e., the total pressure drop of 92 psi recovered 62.7% of the original trapped oil in place. In other words, the recovery factor of 35% at the end of waterflooding increased to 75.7% after performing the CWI process. The results were again consistent with those of the macro-scale experiments. Variations of average oil saturation (within the scanned length) with respect to pressure drop are shown in Fig. 7. It was observed that the first 12 psi pressure drop resulted in 11.5% reduction in oil saturation. Oil saturation then stayed relatively constant to the pressure drop of about 52 psi after which it reduced significantly with additional increases in pressure drop.

Flow conditions for different steps of the experiments and examples of three-phase fluid occupancy maps (for a given
location across the scanned length of Sample B) corresponding to
each step are presented in Table 4 and Fig. 8, respectively.
Figs. 8(a) and 8(b) show uniform distributions of the aqueous
and oil phases in the pore space at the end of primary drainage
and imbibition, respectively. Close inspection of the micro-CT
images (not shown here) indicated that the displacement of
unadulterated brine with carbonated brine, prior to depressuriza-
tion, did not change the configuration of trapped oil ganglia as
the displacement was conducted under the capillary-dominated
displacement regime.

Figs. 9 and 10 provide closer snapshots of two- and three-phase
fluid occupancy maps before and during in situ degassing. Images
in Fig. 9 represent the black rectangles in Fig. 8. Figs. 9(a) and 10(a)
show that oil is trapped in large pores and surrounded by brine
present in the crevices and neighboring elements. The degassing
process starts with gas nucleation and bubble growth. When the
gas bubbles are sufficiently large, they may merge with other gas
blobs, resulting in formation of large gas clusters. Meanwhile, gas
bubbles also contact trapped oil upon which oil forms thick
spreading layers sandwiched between brine in the corners and
gas in the center of the pores (see Fig. 9(b)). The internal gas drive
process produces a gas-to-oil-to-brine double-displacement mech-
nism by which oil is mobilized. This allows oil globules to connect
to each other through coalescence and spreading oil layers. The oil
layers tend to stay stable, thereby allowing the oil to drain, until
gas clusters leave the medium or are fragmented by injected brine.
In addition to displacing oil, gas may directly displace brine. When
the gas clusters grow, they may invade into brine-filled elements
leaving small volumes of the defending fluid in the crevices.

While liberated gas drives the above-mentioned displacement
processes, brine too can impact the fluid occupancy through its
own direct displacements. Here, during CWI, brine is continuously
injected into the medium. This means that the trapped oil, which
has been reconnected by introduction of gas, can now be displaced
by brine as well through direct brine-to-oil imbibition, leading to
possibly re-trapping of the connected oil. While being displaced
by brine, oil may also displace gas (a brine-to-oil-to-gas double-
displacement mechanism). Furthermore, gas itself can be displaced
by brine resulting in fragmentation of large gas clusters. This may

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**Fig. 16.** Individual gas blobs with various sizes during the micro-scale core-flooding experiments at back pressure of 60 psig. The box represents the relative volume and position (in y and z directions) of the biggest globules for various blob sizes.

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**Fig. 17.** Interfacial areas between brine/oil (\(\bullet\)), oil/gas (\(\square\)), and brine/gas (\(\triangle\)) during micro-scale core-flooding experiments.
occur through direct brine-to-gas snap-off and pore-body filling displacement mechanisms.

Mobilization of oil by gas and brine creates a flow of oil through connected oil layers and oil-filled elements. This flow may locally create favorable capillary pressures for oil-to-brine and oil-to-gas displacements. The former is a drainage process and will allow oil to move into smaller pores whereas the latter is an imbibition process that could trap gas, particularly through swelling of oil layers.

Continuous formation and fragmentation of oil and gas clusters and associated displacements create a certain trend by which fluid occupancy is changed during the CWI process. Figs. 11(a) and 11(b) plot the volume of pores occupied by oil and gas, respectively, versus pore sizes. Before gas was liberated, trapped oil occupied largest pores (see Fig. 11(a)) while brine was located in smaller elements. When gas exsolution started, oil was forced into intermediate-sized pores while it was being recovered. This is evident by comparing oil occupancies of imbibition and CWI at 60 psig in

---

**Fig. 18.** (a) Schematic diagram of three-phase ganglion dynamics. Blue, orange, and white represent water, oil, and gas, respectively. (b) A snapshot of a displacement step taken during the transparent glass micromodel experiments. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

**Fig. 19.** Snapshots of displacement steps observed during the transparent glass micromodel experiments.
From this stage onward, the volume of oil continues to shrink (through recovery) and oil gradually gives up more large pores to gas. Gas, on the other hand, occupies the largest pores (see Fig. 11(b)) early during the process and, as the CWI process progresses, loses some of its smaller pores to oil and brine. Individual oil blobs were analyzed statistically to determine the blob frequency distribution. Fig. 12 depicts the cumulative contribution of various oil blob sizes. Here, the cumulative contribution is defined as the ratio of the cumulative volume of oil blobs equal to or smaller than a given volume to the total volume of all oil blobs in the pore space. As observed, at the start of CWI (prior to pressure depletion), the oil blob distribution is relatively uniform, meaning that the oil blobs span a broad range of sizes, and a substantial portion of the total oil volume is supplied by isolated medium-sized blobs. Fig. 12 indicates that, at the back pressure of 80 psig, the medium-sized oil blobs (with a volume ranging from $10^{-3}$ to $10^{-2}$ mm$^3$) reconnected and created a large blob, which in turn increased the cumulative contribution of large-sized blobs. Close to the final steps of the process, more CO$_2$ exsolution again gradually intensified the role of the medium-sized blobs in the total oil volume due to the fragmentation of oil clusters. The oil blobs were also divided into several blob volume groups, and then the blob volume frequency of each group was determined (Fig. 13). For primary drainage, apart from some blobs scattered at small volumes, the oil phase was primarily connected forming a large cluster throughout the pore space. As opposed to primary drainage, oil blobs at the end of the imbibition process showed a wider distribution and were present over a wide range of sizes. Comparing to imbibition, CO$_2$ exsolution during carbonated water injection initially increased the volume of the oil globules by reconnecting the trapped oil ganglia established at the end of imbibition, decreased the number of the individual medium-sized blobs and created several large clusters. At the late stages of the CWI process, however, the frequency of small oil blobs increased again due to fragmentation. The statistical analysis of the largest and all oil blobs in each step of the micro-scale core-flooding experiments are provided in Tables 5 and 6, respectively. The analyses support the above-mentioned findings. The individual oil and gas blobs with various sizes for different steps of the micro-scale core-flooding experiments are shown in Figs. 14–16.

Brine/oil, oil/gas, and brine/gas interfacial areas were also computed from micro-CT images for different steps of the experiments (Fig. 17). At the beginning of in situ degassing, exsolved CO$_2$ contacted trapped oil ganglia and caused oil to spread between brine and gas, which led to initial increases in the gas/oil and oil/brine interfacial areas. The increases were then followed by continuous reductions as CWI progressed. These are attributed to reduction in the volume of oil as it was recovered. The gas/brine interfacial area, however, is negligible because gas is separated from brine by spreading oil layers. In the micromodel experiments, it was observed that exsolution of CO$_2$ from a locally supersaturated aqueous phase in the medium started with heterogeneous gas bubble nucleation and led to steady-state gas cluster dynamics (growth, mobilization, fragmentation, collision, and coalescence) consistent with observations previously reported [22,23]. Steady-state gas cluster dynamics means each pore is visited by gas a great number of times. When the intermediate-wetting phase ganglia (e.g., oil in a water-wet medium) are also present, the flowing, albeit disconnected, gas phase sets up a cascade of double drainage and double imbibition displacements during which parts of large oil clusters or small oil ganglia are gradually mobilized with the gas phase (see Figs. 18 and 19). The efficiency of oil recovery at the pore scale is related to the local intensity of gas phase dynamics. In all the visualization studies, oil was the intermediate wetting phase and this appeared to be important for the success of the process.

5. Summary and conclusions

We presented the results of a multi-scale three-phase experimental study of carbonated water injection (CWI) and subsequent CO$_2$ exsolution, as a consequence of pressure depletion, that led to recovery of significant fractions of trapped oil. We investigated the process at low pressure and temperature conditions due to the applicability of CWI in environmental engineering as well as in petroleum engineering and to understand the underlying flow physics under simplified conditions before applying the process to more complicated petroleum reservoir conditions. Experiments were performed at both macro and micro scales using spreading fluid systems. At the macro scale, a long consolidated Berea core sample along with macro-CT imaging was utilized to perform a set of core-flooding experiments in order to examine the effectiveness of the immiscible CWI (in situ degassing and consequent three-phase ganglion dynamics) in recovering waterflood residual oil. We then used micro-scale studies to investigate the pore-level displacement physics responsible for the trends observed at the macro scale. Miniature core-flooding experiments were performed in a similar, but smaller, Berea sample using a micro-CT scanner to obtain high-resolution images of pore-scale fluid occupancies. We reported pore-scale observations of three-phase flow involving two immiscible disconnected non-wetting phases (i.e., oil ganglia and gas bubbles) and one connected wetting phase (i.e., water). More details of the pertinent displacement mechanisms were also investigated using a two-dimensional glass micromodel.

In the macro-scale core-flooding experiments, the initial unadulterated brine injection resulted in 40.1% of the original oil in place recovery. The following CWI process, however, increased the recovery factor to 74.7%, i.e., 34.6% additional recovery of the original oil in place. Similar results were also obtained from the micro-scale core-flooding experiments. The recovery factor of 35% at the end of unadulterated waterflooding increased to 75.7% (i.e., 40.7% additional recovery) after performing the CWI process. Micro-CT visualization of pore occupancy showed that the gradual increase in the pressure drop led to exsolution of CO$_2$ internal gas drive, mobilization of oil ganglia, and reduction in waterflood residual oil saturation. When contacted by CO$_2$, oil globules formed thick spreading layers between brine and gas and were displaced toward the outlet along with moving gas clusters. We observed significant re-connection of trapped oil globules due to oil layer formation during early stages of CWI. The oil layers stayed stable until very late stages of gas exsolution. Analysis of isolated oil blobs showed higher cumulative volumes for small- and medium-sized blobs during the waterflood compared to the initial stages of CWI. Similar behavior was also observed in the final steps of CWI because re-connected oil clusters were fragmented by the exsolved CO$_2$.

In addition to trapped oil recovery, entrainment of CO$_2$ at the end of the CWI process as free gas as well as dissolved gas in brine offers a potentially effective scheme for geologic sequestration of CO$_2$ in petroleum oil reservoirs.

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References


