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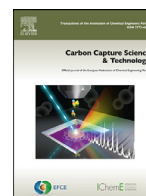
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## Full Length Article

Long tube flooding tests for investigating salt precipitation induced by CO<sub>2</sub> injection

Prinu Narayanan<sup>a</sup>, Maryam Khosravi<sup>a</sup>, Rikke Weibel<sup>b</sup>, Leonardo Teixeira Pinto Meirles<sup>c</sup>,  
Niels Hemmingsen Schovsbo<sup>b</sup>, Erling H. Stenby<sup>a</sup>, Wei Yan<sup>a,\*</sup>

<sup>a</sup> Center for Energy Resources Engineering (CERE), Department of Chemistry, Technical University of Denmark (DTU), Denmark

<sup>b</sup> Geological Survey of Denmark and Greenland (GEUS), Denmark

<sup>c</sup> CERE, Department of Environmental and Resource Technology, DTU, Denmark

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## ABSTRACT

Capturing a large amount of CO<sub>2</sub> from industry, energy production, or incineration plants and storing it in the subsurface such as depleted hydrocarbon reservoirs or saline aquifers can fundamentally reduce the footprint of atmospheric greenhouse gasses. Injection of CO<sub>2</sub> in a saline aquifer zone or depleted hydrocarbon reservoir will vaporize formation water and lead to potential salt precipitation in porous rock, thus increasing the risk of formation damage near the wellbore area and reducing the injectivity of CO<sub>2</sub> in the reservoir. Previous experimental studies on salt precipitation have focused on core flooding, including micro-core flooding, ordinary single-plug flooding, and composite core flooding. The length of the core plugs limits these flooding tests. They usually suffer from serious end effects, and it is generally challenging to investigate the variation along the flooding direction, especially after large pore volumes of injection. In this study (Project Greensand), we have researched and developed new equipment termed “long tube apparatus” to improve the understanding of salt precipitation and gauge the effects of formation damage in detail for a North Sea depleted oil field reservoir (Nini West) under investigation, while injecting CO<sub>2</sub>. The main aim of the experiment was to determine two properties such as salt and water content, which are used to evaluate the formation damage due to salt precipitation. Finally, a numerical simulation was carried out using CMG GEM in both 1D homogeneous model and another heterogeneous model honoring the channeling at the inlet. Results with qualitative agreement were achieved, especially with the heterogeneous model.

## Introduction

To achieve the ambitious goal of carbon neutrality by 2050 set by the Danish government, CO<sub>2</sub> capture and storage (CCS) is considered a significant contributor to the portfolio of technologies for CO<sub>2</sub> emission reduction. Currently, geological storage of CO<sub>2</sub> in depleted oil/gas reservoirs or aquifers is being seriously investigated in Denmark. The phenomenon associated with salt precipitation can reduce the productivity in gas-producing fields (Kleinitz et al., 2003) or the injectivity in natural gas storage aquifers. The injection of CO<sub>2</sub> will result in water vaporization near the wellbore region and subsequently trigger salt precipitation inside the porous reservoir rock (Tang et al., 2015). The phenomena associated with salt precipitation further reduce porosity and permeability, thereby inducing formation damage and potentially decreasing the injectivity of CO<sub>2</sub> (André et al., 2011; Baumann et al., 2014; Peysson et al., 2014). The formation damage associated with CO<sub>2</sub> injection will depend on reservoir condition, fluid composition, injection

rate, rock pore geometry, and mineralogy (Izgec et al., 2008). Reduction in CO<sub>2</sub> injectivity was observed in Snøhvit and Ketzin fields as part of carbon storage projects (Grude et al., 2014; Nooraiepour et al., 2018).

Several salt precipitation studies were carried out on a pore scale by using micromodel imaging and x-ray tomography in fully saturated brine (Kim et al., 2013; Norouzi Rad and Shokri, 2014; Miri et al., 2015; Berntsen et al., 2019; Falcon-Suarez et al., 2020; Akindipe et al., 2022) to determine the drying effects induced by CO<sub>2</sub> injection. Furthermore, core flooding experiments were carried out on ordinary single-plug to determine the drying process and the associated salt precipitation (Ott et al., 2011; Mahabadi et al., 2019). These flooding tests are limited by the length of the core plugs. They usually suffer from serious end effects (Andersen et al., 2017), and it is generally difficult to investigate the variation along the flooding direction, especially after large pore volumes of injection.

The slim tube test used in the oil industry influenced the long tube apparatus. The slim tube study experiment was carried out on an adequately pressure-rated steel tube pre-packed with glass beads or Ottawa sand with a length ranging from 40 to 80 ft. The test was carried out by saturating the slim tube with pressurized hydrocarbon oil at reser-

\* Corresponding author.

E-mail address: [weya@kemi.dtu.dk](mailto:weya@kemi.dtu.dk) (W. Yan).

### Nomenclature

|       |           |
|-------|-----------|
| Cont. | Content   |
| Dep.  | Deposited |
| Vap.  | Vaporized |
| Dis.  | Displaced |
| Red.  | Reduced   |

voir temperature, followed by gas injection above the saturation pressure of the displacing fluid. The recovery of different injection pressures was plotted in a graph. The intersection of immiscible and miscible recoveries obtained during the slim tube study constitutes the Minimum Miscibility Pressure (MMP) data and is extensively used by reservoir engineers in the oil and gas industry (Yan et al., 2012; Ekundayo and Ghedan, 2013).

Similar to the slim tube design, we have designed a novel flooding test to investigate the salt precipitation effects in a long tube pre-filled with sand particles. The long tube apparatus uses unconsolidated sand rather than core plugs and thus allows investigating the drying-out experiment on a larger scale which are historically unavailable for conventional core flooding experiments. Furthermore, it will enable a direct inspection and measurement of the properties at different tube positions through destructive testing after the flooding followed by the determination of water and salt contents. This allows a more detailed investigation of salt precipitation and the effects of formation damage.

This study evaluates the risks associated with salt precipitation when CO<sub>2</sub> is injected into a depleted hydrocarbon reservoir in the Nini Field in the Danish part of the North Sea. The long-tube experiment presented here provides a novel complementary method to the conventional core flooding method. In the following, we first present the sample and material used in the experiment, then the detailed design of the long-tube apparatus. After that, we present the experimental results for long-tube tests. Finally, we present the simulation analysis of the long-tube experiment using CMG GEM.

## Rock, sand, and fluid properties

### Rock samples properties

The Danish reservoirs are mostly composed of Cretaceous-Danian Chalk. However, the Siri Canyon formed in the Paleocene was eroded into the underlying Cretaceous-Danian Chalk Group and filled with Paleocene-Miocene hemipelagic and turbidities marls and mudstones interbedded with Paleocene-Eocene well-sorted, fine-grained glauconitic sandstones deposited from sandy mass flows and sandy turbidities (Petersen et al., 2022).

Core samples collected from well Nini 4 were first cleaned using the Soxhlet method and subjected to routine rock analysis. The obtained routine rock properties are displayed in Table 1. The poorly cemented sandstone was easily disintegrated into individual grains. The cleaned grains were then used for Experiments 2, 3, 4, and 5.

### Sand properties

Two different types of sand has been used in this study: the Nini-4 sand and the BCS-315 quartz sand. The mineralogical composition of

**Table 2**

Synthetic brine composition that mimics formation water composition as recorded in produced water from the Nini field.

| Element/Ion                   | Concentration (mg/l) |
|-------------------------------|----------------------|
| Na <sup>+</sup>               | 29,300               |
| K <sup>+</sup>                | 211                  |
| Mg <sup>2+</sup>              | 1027                 |
| Ca <sup>2+</sup>              | 4800                 |
| Sr <sup>2+</sup>              | 602                  |
| Ba <sup>2+</sup>              | 123                  |
| Fe <sup>2+</sup>              | 0.9                  |
| Cl <sup>-</sup>               | 57,900               |
| HCO <sub>3</sub> <sup>-</sup> | 100                  |
| SO <sub>4</sub> <sup>2-</sup> | 6.3                  |

sandstones in the Nini-4 wells are shown in Fig. 1. They mainly consist of quartz and glauconitic clasts. On the other hand, BCS-315 quartz sand from Strobel Quarzsand GmbH (used as received) was procured from a third-party supplier with 99.1% of SiO<sub>2</sub>, <0.1% of Fe<sub>2</sub>O<sub>3</sub>, and <0.2% of Al<sub>2</sub>O<sub>3</sub> & TiO<sub>2</sub>.

The grain size distribution of both Nini sand and BCS-315 was found to be similar. Hence, BCS-315 sand was used for the trial experiment (Experiment 1).

Details of grain size distribution between Nini-4 and BCS-315 sand can be found in Fig. 2.

### Fluid properties

A synthetic brine was prepared by mimicking the produced water composition from the Nini field (Mohammadkhani et al., 2023; Schovsbo et al., 2023) and the exact composition details can be found in Table 2. The same synthetic brine was then used to perform Experiments 1, 3, and 4.

## Long-Tube experiment

### Experimental setup

As shown in Figs. 3 & 4, the long-tube experimental setup consists of a high-temperature oven, long-tube, high-pressure pumps, injection cylinders, accumulators, Back Pressure Regulator (BPR), temperature, and pressure sensors. Filters were used in the inlet and outlet of the long tube to ensure that the fluid flow was uniformly distributed. The key parameters used for the design of the Long-tube Flooding unit used in this study are provided in Table 3.

### Experimental procedure

A long tube with 80 cm for vertical tubing and 600 cm for spiral tubing was pre-filled with unconsolidated sand. A sufficient volume of CO<sub>2</sub> was transferred from the 50 L cylinder to a portable laboratory cylinder by using the equilibrium method. A brief overview of the long tube experimental procedure can be viewed in Fig. 5.

The long tube was initially saturated with brine or deionized water and its pore volume (PV) was determined. CO<sub>2</sub> was used to displace brine or de-ionized water over a prolonged period, covering ~ 80 PV at a flow rate of 0.38, 0.75 and 1.5 cm<sup>3</sup>/min. The experimental

**Table 1**  
Routine rock properties of Nini-4 samples.

| Sample Number | Well   | Direction  | Depth (m) | Gas Permeability (mD) | Grain Density (g/cm <sup>3</sup> ) | He Porosity (%) |
|---------------|--------|------------|-----------|-----------------------|------------------------------------|-----------------|
| 94            | Nini-4 | Horizontal | 1776.04   | 1190                  | 2.70                               | 35.4            |
| 96            | Nini-4 | Horizontal | 1776.66   | 1420                  | 2.69                               | 34.6            |
| 97            | Nini-4 | Horizontal | 1777.04   | 1230                  | 2.70                               | 35.5            |

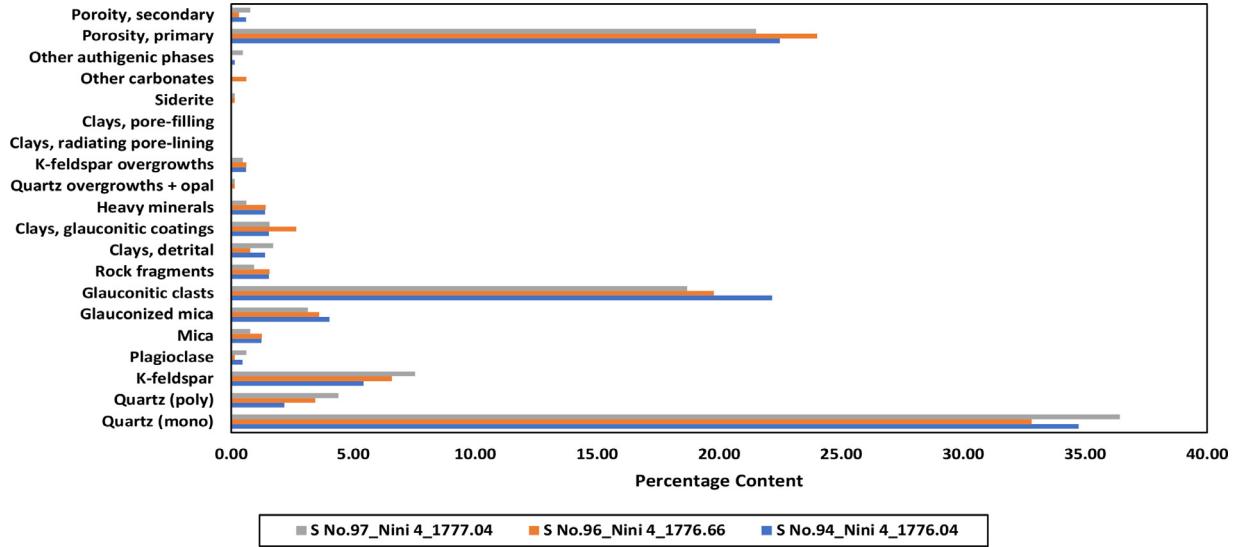


Fig. 1. Bulk mineralogical composition of the Nini samples.

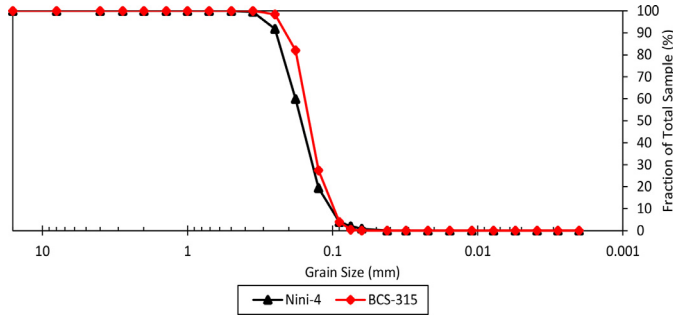


Fig. 2. Grain size distribution of Nini-4 and BCS-315 sand.

Table 3

Key parameters of long-tube apparatus.

| Description                                    | Values                 | Unit            |
|--|------------------------|-----------------|
| Long Tube Material                             | Stainless steel        |                 |
| Internal Diameter of Vertical Long-Tube        | 0.94                   | cm              |
| Length of Vertical Long-Tube                   | 80                     | cm              |
| Vertical Long Tube Area                        | 0.69                   | cm <sup>2</sup> |
| Internal Diameter of Spiral Long-Tube          | 0.46                   | cm              |
| Length of Spiral Long Tube                     | 600                    | cm              |
| Spiral Long Tube Area                          | 0.16                   | cm <sup>2</sup> |
| Injection Fluid                                | CO <sub>2</sub>        | n/a             |
| Displacing Fluid                               | Brine/De-Ionized Water | n/a             |
| CO <sub>2</sub> Viscosity at 60 °C and 200 bar | 0.06                   | cP              |
| Brine Viscosity at 60 °C and 200 bar           | 0.57                   | cP              |

geometry was vertical, and fluids were injected into the long tube by positive displacement pumps (ISCO pumps) at a constant rate. A back-pressure of 200 bar was set to control the outflow and stabilize the pressure during the experimental timeframe. The Joule-Thomson cooling effect was observed during the trial experiments by using CO<sub>2</sub> as the

injecting fluid near to the BPR owing to the high permeability medium (Oldenburg, 2007). Henceforth, a series of accumulators were installed before the BPR to ensure that the Joule-Thomson cooling effect does not affect the flow regime during the experiment.

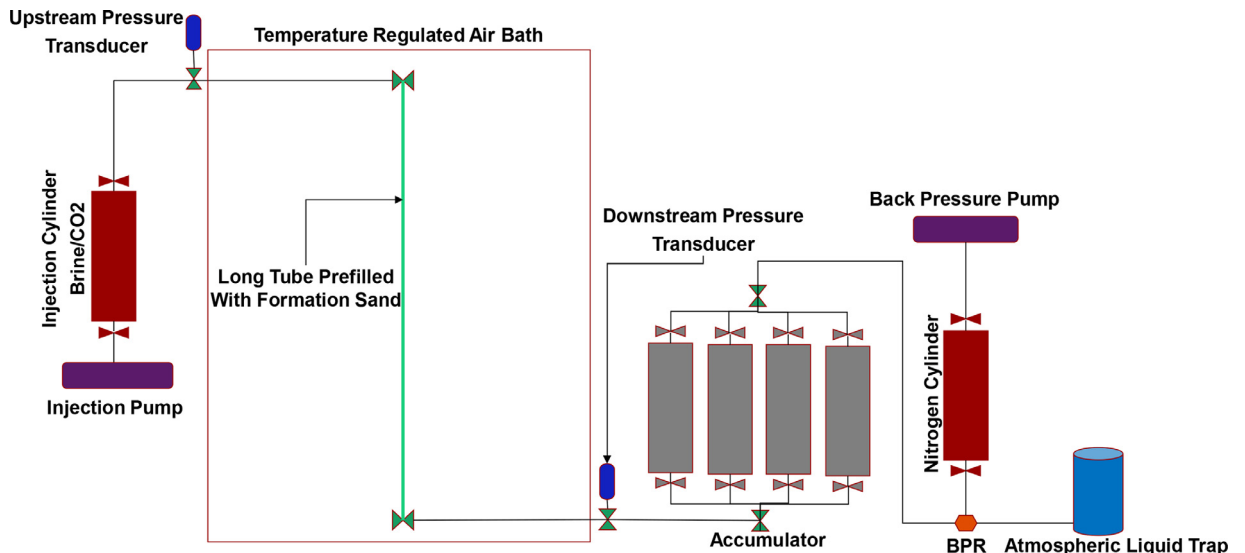


Fig. 3. Schematic of long tube apparatus with vertical tubing.

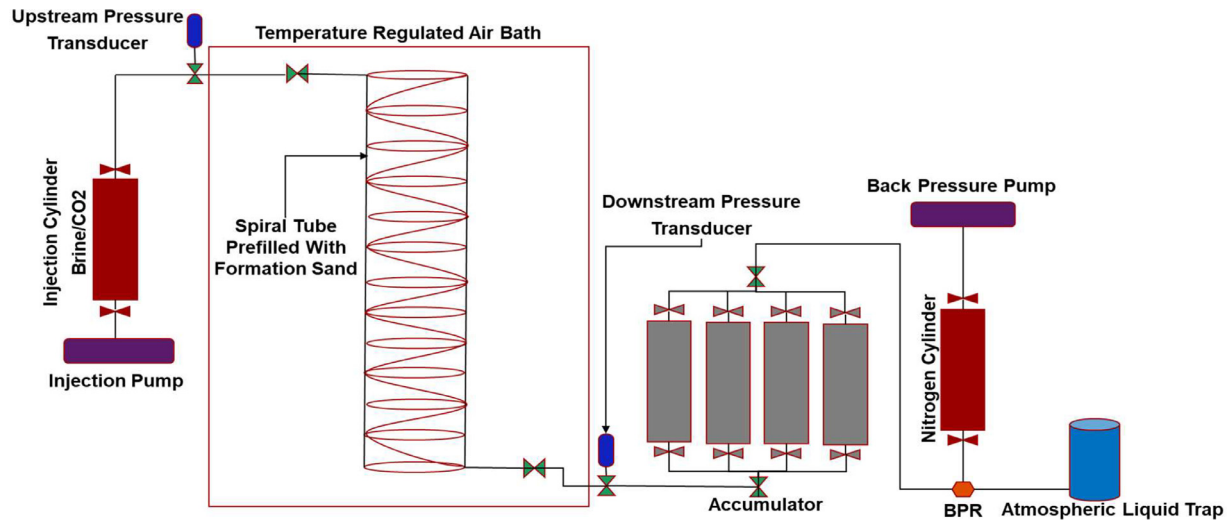


Fig. 4. Schematic of long tube apparatus with spiral tubing.

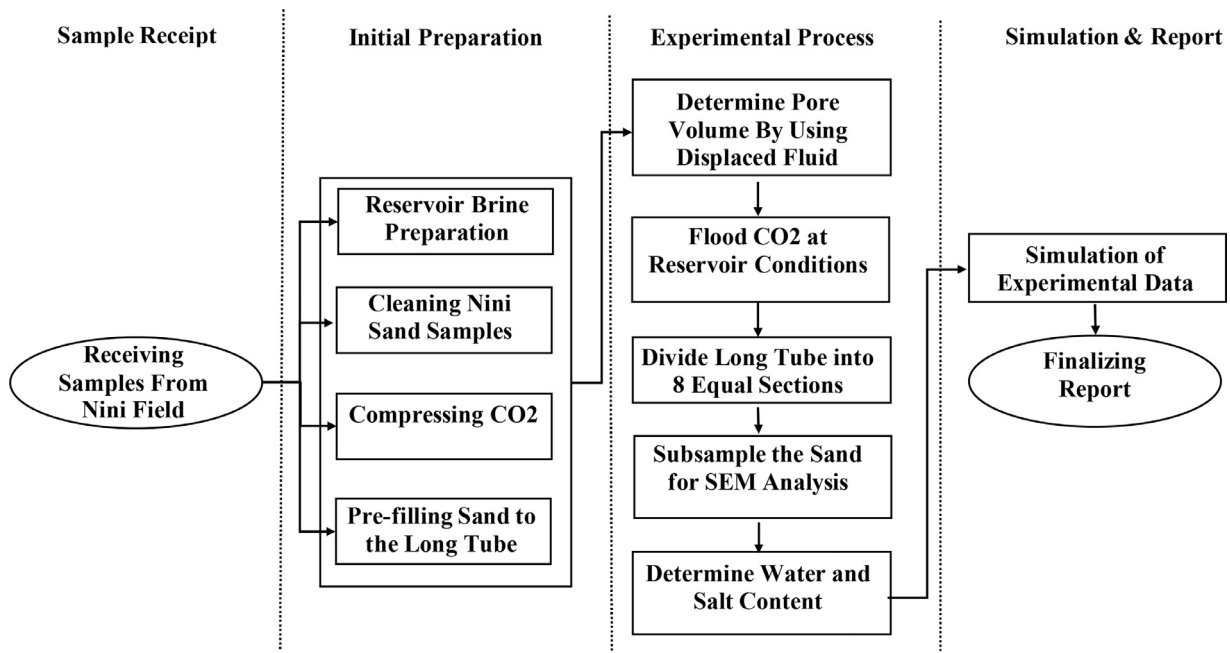


Fig. 5. Experimental flowchart.

After completion of the CO<sub>2</sub> flooding, the tube was retrieved from the flooding unit and subdivided into eight equal segments by using an appropriate cutting tool for stainless steel tubing. A sub-sample was collected from the top and bottom of each section to undergo SEM analysis. The remaining sand sample was collected in eight different beakers with appropriate weights recorded. The collected sand samples were used for a gravimetric methodology (Margolis et al., 2019) for water and salt content measurements. Using gravimetric methodology, de-ionized water was used as an effluent for determining salt content. The experimental results were then used for simulation. Details of simulation data can be found in Section 5.

### Experimental results

Five experiments were carried out using the long-tube flooding unit at reservoir conditions (60 °C and 200 bar). A summary of experimental

conditions can be found in Table 4, followed by a summary of water and salt content in Figs. 6 and 7 respectively.

#### Experiment 1 (BCS-315 & synthetic brine)

A trial experiment with the BCS-315 quartz sand was carried out before the actual experiment with reservoir sand, owing to its limited availability. The information and learnings gained during the trial experiments were incorporated for the real experiment with the reservoir sand and formation brine. After the first flooding experiment was completed, the tube was divided into eight segments with a length of 10 cm each.

Based on the close evaluation of each segment, it was observed that sand consolidation had occurred due to the presence of water, predominantly in the first section of the tubing resulting in the aggregation of

**Table 4**  
Summary of experimental conditions.

| Exp No. | Experimental Type             | Long Tube Length (cm) | Flowrate (cm <sup>3</sup> /minute) | Measured PV | PV Injected |
|---------|-------------------------------|-----------------------|------------------------------------|-------------|-------------|
| 1       | BCS-315 & Synthetic Brine     | 80                    | 1.50                               | 19.89       | 47.53       |
| 2       | Nini-4 Sand & Deionized Water | 80                    | 1.50                               | 29.15       | 74.72       |
| 3       | Nini-4 Sand & Synthetic Brine | 80                    | 1.50                               | 29.42       | 81.68       |
| 4       | Nini-4 Sand & Synthetic Brine | 80                    | 0.75                               | 29.37       | 79.54       |
| 5       | Nini-4 Sand & Synthetic Brine | 600                   | 0.38                               | 49.50       | 80.15       |

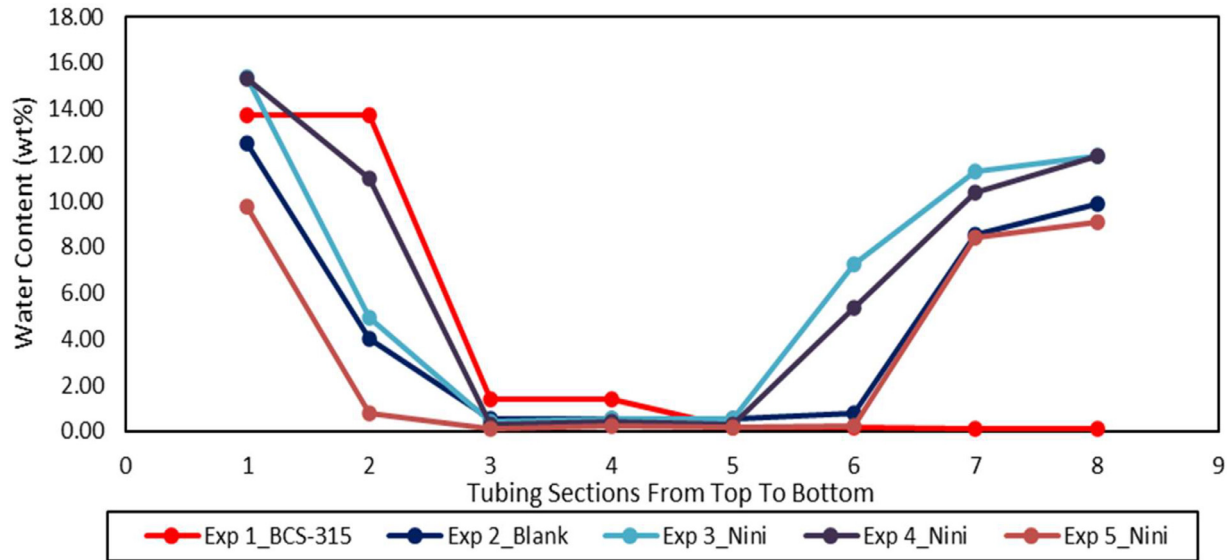


Fig. 6. Summary of water content.

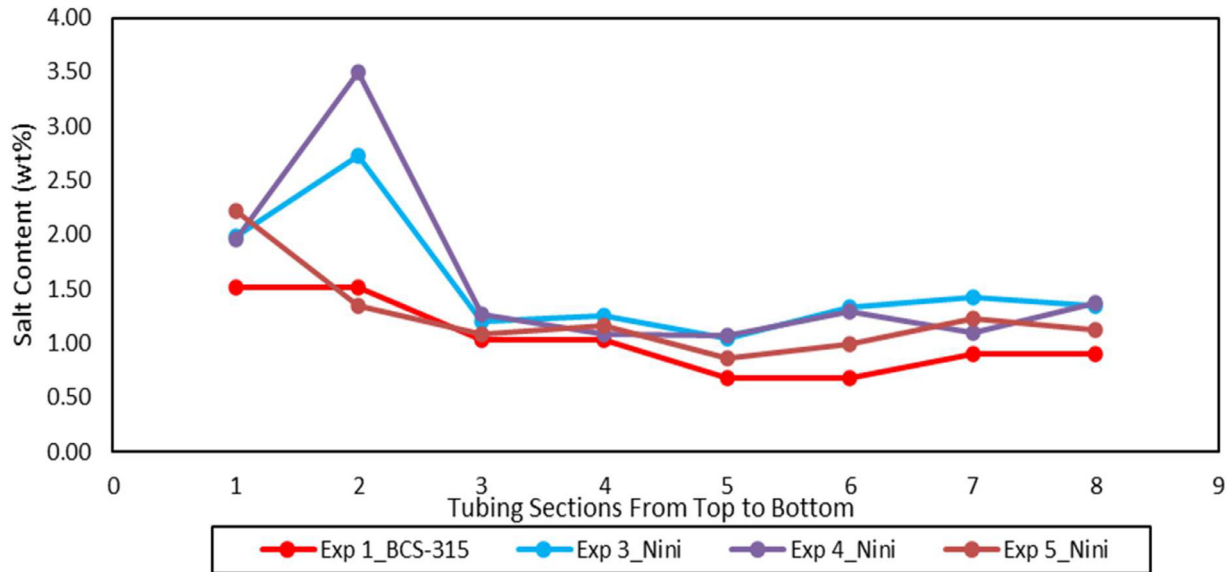


Fig. 7. Summary of salt content.

the former unconsolidated sandstones corroborating with quantitative analysis by gravimetric methodology (Fig. 8 and Table 5).

The consolidation phenomenon could be due to either one or a combination of several mechanisms as below;

- (3) Enhanced water evaporation from near polycrystalline aggregate due to enhanced surface area and self-enhancing effect.
- (4) Channeling of displacing fluid (CO<sub>2</sub>) over displaced fluid (brine or de-ionized water) (Bacci et al., 2013).

- (1) Increased salt precipitation due to the evaporation of water from the brine caused by CO<sub>2</sub> injection.

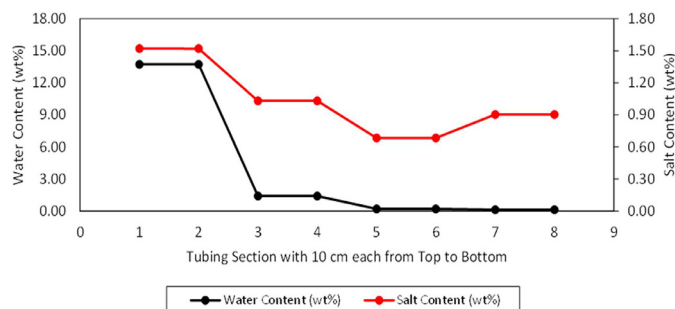
- (2) Brine returns to the displaced segment or dried-out region owing to the capillary force.

The complete evaporation of water in the middle zone and subsequent salt precipitation did not lead to any blockage or reduction in permeability.

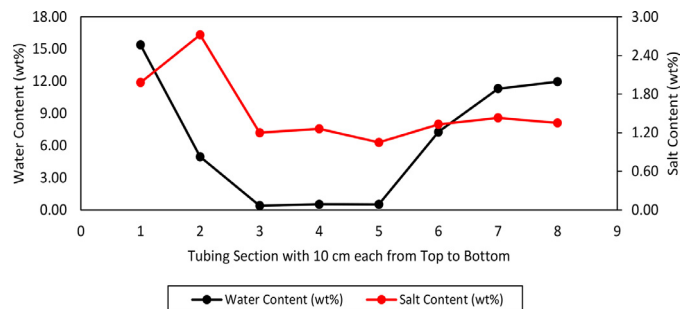


**Table 5**  
Sand consolidation on the tubing segments for experiment 1.

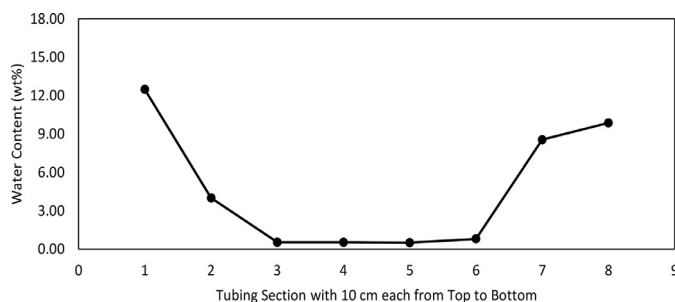
| Exp No. | Experimental Type         | Long Tube Sections of 10 cm each from Top to Bottom |     |    |    |    |    |    |    |
|---------|---------------------------|---|-----|----|----|----|----|----|----|
|         |                           | 1   | 2   | 3  | 4  | 5  | 6  | 7  | 8  |
| 1       | BCS-315 & Syntehtic Brine | Yes   | Yes | No | No | No | No | No | No |



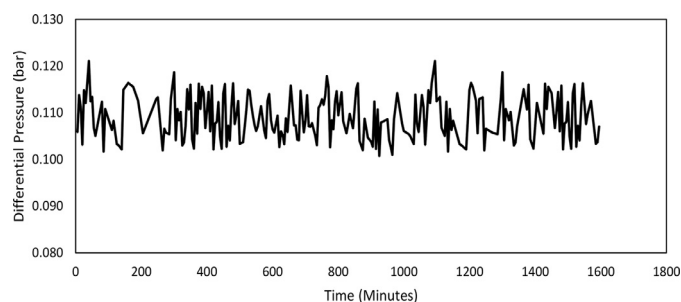
**Fig. 8.** Summary of water and salt content for experiment 1.



**Fig. 10.** Summary of water and salt content for experiment 3.



**Fig. 9.** Summary of water content for experiment 2.



**Fig. 11.** Differential pressure data for experiment 3.

#### Experiment 2 (Nini-4 sand & deionized water)

To understand the consolidation process on the first flooding in detail and test the above hypothesis, a blank experiment was performed to determine the interaction between the injecting ( $\text{CO}_2$ ) and displaced fluid (de-ionized water). Like the first experiment, all the relevant properties were measured except salt content (Fig. 9). Consolidation was observed for the first, second, seventh, and eighth section of this test, suggesting that aggregation of sand is not merely due to salt precipitation (Table 6).

#### Experiment 3 (Nini-4 sand & synthetic brine)

Based on the understanding from the first two experiments, a representative filling material (reservoir sand) and saturating fluid (formation brine) were used for the third experiment at reservoir conditions.  $\text{CO}_2$

was then used as the displacing fluid, injected at  $1.5 \text{ cm}^3/\text{minute}$  over a period of 81.68 PV. Differential pressure data was recorded (Fig. 10) during the experiment and no significant decrease or increase in data was observed. Upon completion of the flooding experiment, the long tube was divided into 8 segments and the information on the segment consolidation can be viewed in Table 7. Unlike the 1st trial experiment associated with BCS-315, the consolidation was not limited to 1st & 2nd segments, instead, it was observed at the tail end of the tube covering the 6<sup>th</sup>, 7<sup>th</sup>, and 8<sup>th</sup> (Fig. 11).

After completing the  $\text{CO}_2$  flooding experiment, a sub-sample from each section (both top and bottom) of the tubing was collected and underwent SEM analysis, where a petrographical investigation was performed on platinum-coated rock chips using a Zeiss Sigma 300 VP field emission Scanning Electron Microscope (SEM) at 15 kV and 40–100 nA.

**Table 6**  
Sand consolidation on the tubing segments for experiment 2.

| Exp No. | Experimental Type             | Long Tube Sections of 10 cm each from Top to Bottom |     |    |    |    |    |     |     |
|---------|-------------------------------|---|-----|----|----|----|----|-----|-----|
|         |                               | 1   | 2   | 3  | 4  | 5  | 6  | 7   | 8   |
| 2       | Nini-4 Sand & Deionized Water | Yes   | Yes | No | No | No | No | Yes | Yes |

**Table 7**  
Sand consolidation on the tubing segments for experiment 3.

| Exp No. | Experimental Type             | Long Tube Sections of 10 cm each from Top to Bottom |     |    |    |    |     |     |     |
|---------|-------------------------------|---|-----|----|----|----|-----|-----|-----|
|         |                               | 1   | 2   | 3  | 4  | 5  | 6   | 7   | 8   |
| 3       | Nini-4 Sand & Synthetic Brine | Yes   | Yes | No | No | No | Yes | Yes | Yes |

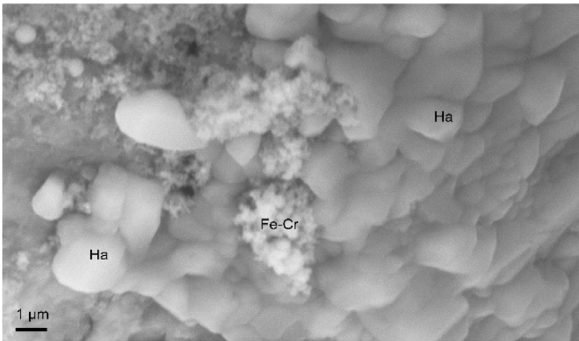


Fig. 12. SEM image of Section 1-top.

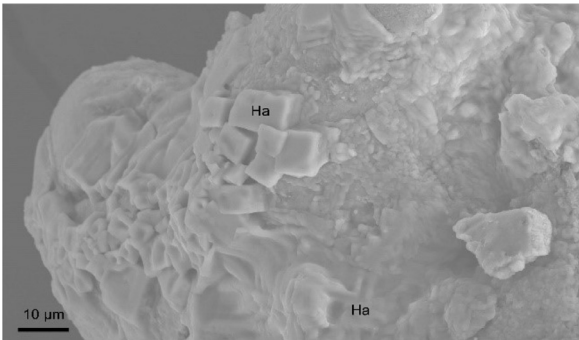


Fig. 13. SEM image of Section 2-top.

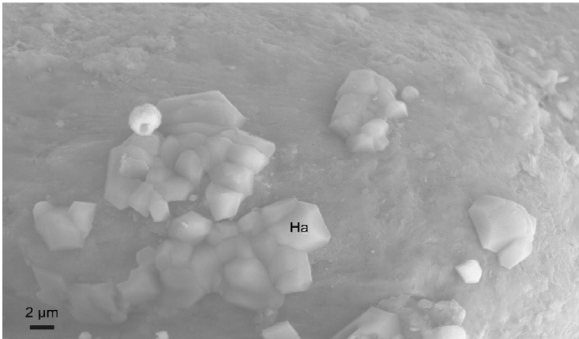


Fig. 14. SEM image of Section 4-top.

The chemical composition of the investigated minerals was obtained by Energy Dispersive Spectroscopy (EDS).

Figs. 12–15 provide the SEM images of the samples from Experiment 3. Halite (Ha) is precipitated from brine and Fe-Cr precipitates (Fe-Cr) are probably the result of dissolution of steel during the experiment.

From the SEM analysis, it was observed that halite was the most common mineral formed during the flooding experiment. Halite was precipitated as scattered tiny crystals and occasionally as patchy coatings with squared terminations. Halite was found to be predominant in the top and bottom of the long tube.

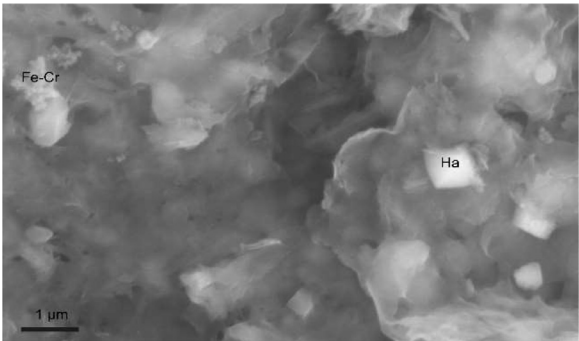


Fig. 15. SEM image of Section 8-bot.

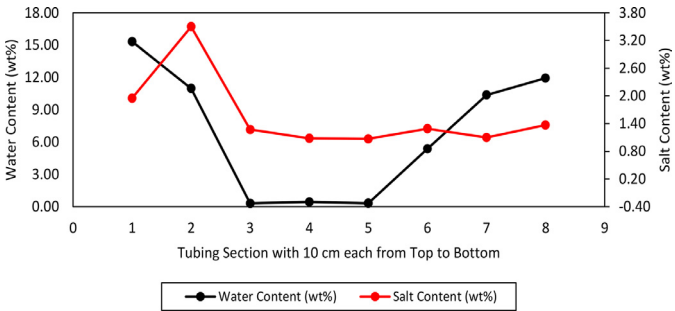


Fig. 16. Summary of water and salt content for experiment 4.

Experiment 4 (Nini formation sand & synthetic brine)

The flow rate was reduced from 1.5 to 0.75 cm<sup>3</sup>/min for the fourth experiment to determine the impact on salt precipitation. The result (Table 8 and Fig. 16) after flooding with CO<sub>2</sub> as part of Experiment 4 was comparable with Experiment 3.

Experiment 5 (Nini formation sand & synthetic brine)

The fifth experiment was carried out by using spiral tubing with 600 cm in length. The fifth experiment was performed in order to compare the salt and water content from the previous four experiments performed with vertical tubing length of 80 cm.

The experimental results from Experiment 5 (Table 9 and Fig. 17) on the spiral tube were found to be comparable with previous vertical tube experiments (i.e. 1, 2, 3 & 4).

Observations and findings from the long-tube experiment

This study has resulted in the following important observations and findings:

- The long tube flooding apparatus developed here is an effective and complementary method to conventional core flooding to study salt precipitation during CO<sub>2</sub> injection. The method removes the limitation on the flooding length and provides more insightful results on water and salt distribution after flooding.
- The SEM analysis indicated the presence of halite in each section of the tubing.

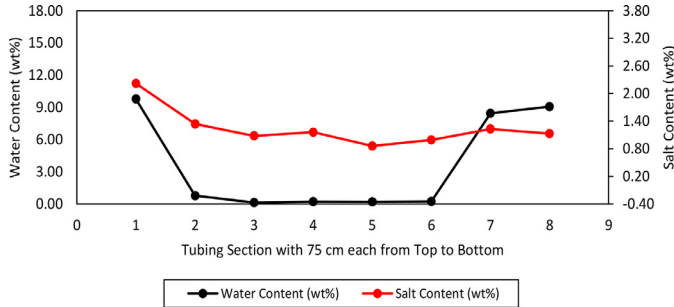
Table 8  
Sand consolidation on the tubing segments for experiment 4.

| Exp No. | Experimental Type             | Long Tube Sections of 10 cm each from Top to Bottom |     |    |    |    |     |     |     |
|---------|-------------------------------|---|-----|----|----|----|-----|-----|-----|
|         |                               | 1   | 2   | 3  | 4  | 5  | 6   | 7   | 8   |
| 4       | Nini-4 Sand & Synthetic Brine | Yes   | Yes | No | No | No | Yes | Yes | Yes |



**Table 9**  
Sand consolidation on the tubing segments for experiment 5.

| Exp No. | Experimental Type             | Long Tube Sections of 75 cm each from Top to Bottom |     |    |    |    |    |     |     |
|---------|-------------------------------|---|-----|----|----|----|----|-----|-----|
|         |                               | 1   | 2   | 3  | 4  | 5  | 6  | 7   | 8   |
| 5       | Nini-4 Sand & Synthetic Brine | Yes   | Yes | No | No | No | No | Yes | Yes |



**Fig. 17.** Summary of water and salt content for experiment 5.

- Based on the gravimetric analysis, only the inlet and outlet showed relatively high-water saturation, and the water in the middle part was largely evaporated.
- A salt content larger than 1 wt% was observed throughout the tubing, but only the middle segments, where water is largely vaporized, have resulted salt in the deposited form.
- Consolidation of the packed sand was found in some segments close to the inlet and outlet, somewhat coinciding with the high water content regions.
- The results for the 80 cm tube and the 6 m tube are comparable in the water content, salt content, and consolidation observation.
- No significant increase in the pressure drop has been observed during the CO<sub>2</sub> flooding tests.

## Simulation

Numerous modeling and simulations were carried out to identify the loss in injectivity owing to the salt precipitation associated with CO<sub>2</sub> in-

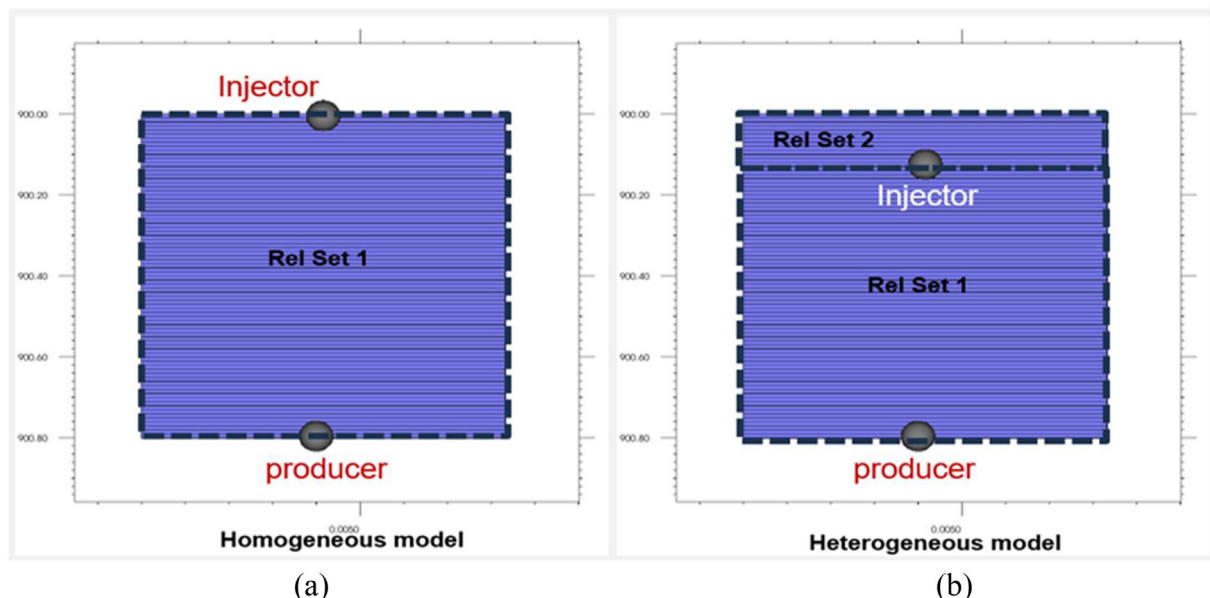
jection near to wells (Giorgis et al., 2007; Muller et al., 2009; Kim et al., 2012; Tambach et al., 2015). In this study, a simulation was performed on vertical long-tube flooding using CMG GEM.

## Simulation models

Two simulation models were built upon Experiment 3. The first simulation model was a 1D **homogeneous model** ( $1 \times 1 \times 80$ ) as shown in Fig. 18(a). The second developed later, sought to rectify the limitations of the first model, by inserting some heterogeneity in different properties such as relative permeability set and well perforation locations (Fig. 18(b)). In the first model, an injection well was introduced at the uppermost grid and a producer at the lowermost grid. Relative permeability endpoints were adjusted such that ultimate remained water saturation at the bottom of the column could be matched. Based on water distribution at the end of experiments 2 and 3, several sensitivity analyses were conducted on the homogeneous model and finally, irreducible water saturation of 0.29 was chosen for the relative permeability curves with a Corey exponent of 3, which was suggested for loose sand packs in CMG-Builder. Table 10 provides detailed model specifications.

The second simulation model was constructed similarly to the first but a **heterogeneous** one (Fig. 18(b)). A new set of relative permeabilities was defined for the upper layers from 1 to 16, with larger irreducible water saturation. The injector well was perforated in the 17th grid block to mimic the channeling of CO<sub>2</sub> in the bypassed area in the inlet segment. Such treatment prevents CO<sub>2</sub> from entering the upper neighboring grids and thus keeps a relatively high water saturation in the area. The existence of such an area has been confirmed in experiments 1, 2, and 3. A simulation was then carried out on a homogenous model to set the appropriate permeability and capillary pressure, then switched to the heterogeneous model for detailed simulations.

To tune the interaction of water and CO<sub>2</sub>, a binary interaction coefficient of 0.165 was selected. This choice allowed us to precisely align the



**Fig. 18.** Simulation models constructed for modeling CO<sub>2</sub> injection into a long vertical tube, (a) homogeneous model & (b) heterogeneous model.

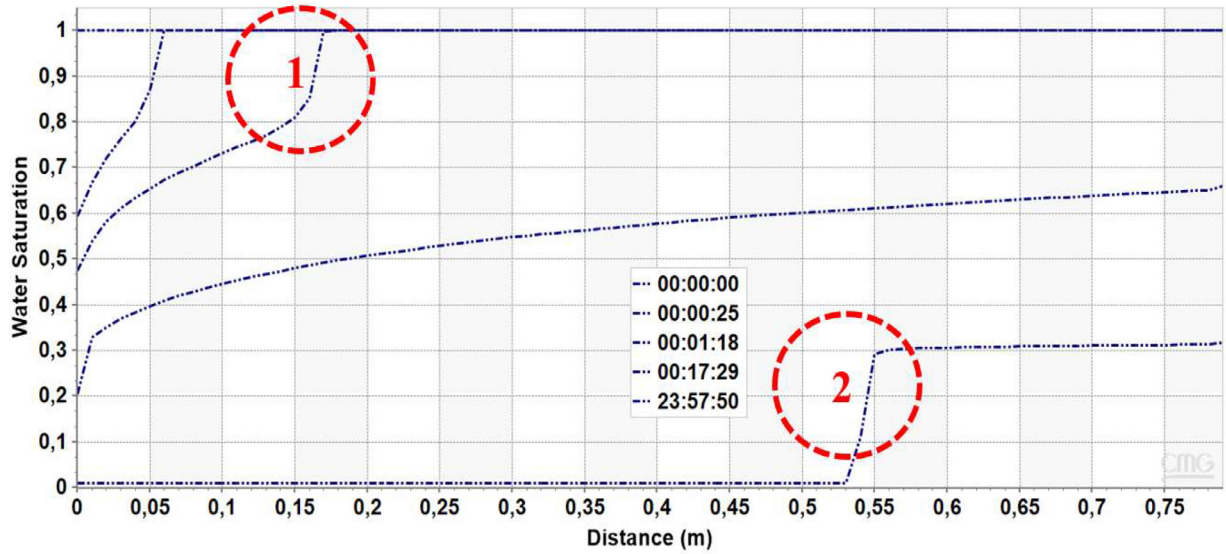


Fig. 19. Predicted water saturation distribution at different times within the linear path between the injector and producer in the homogeneous model.

**Table 10**  
Simulation model specifications.

| Description   | Value  | Unit                |
|---|--------|---------------------|
| Length in x and y direction   | 0.0083 | M                   |
| Length in z-direction   | 0.8    | M                   |
| Porosity  | 52     | %                   |
| Permeability  | 1578   | mD                  |
| Temperature   | 60     | °C                  |
| Pressure  | 200    | Bar                 |
| Initial water saturation  | 100    | %                   |
| CO <sub>2</sub> injection rate  | 0.0021 | m <sup>3</sup> /day |
| Irreducible water saturation in set number 1                                | 0.29   |                     |
| Irreducible water saturation in set number 2                                | 0.45   |                     |
| Water relative permeability at irreducible gas saturation                   | 1      |                     |
| Gas relative permeability at irreducible water saturation                   | 1      |                     |
| Exponent for calculation of $k_r$ at different saturations of water and gas | 3      |                     |

extension of the dried-out region with segment 6, ensuring a seamless match. Furthermore, to achieve the desired maximum salt deposition in segment 2, the diffusion coefficient of Na<sup>+</sup> and Cl<sup>-</sup> was fine-tuned to a value of  $4 \times 10^{-6}$  cm<sup>2</sup>/sec. Ion diffusion tends to homogenize the amount of salt deposition within the dried-out zone.

The PVT section of the model incorporates Peng-Robinson EOS with a 3-component system of components (CO<sub>2</sub>, H<sub>2</sub>O and a tracer of CH<sub>4</sub> for accuracy of calculations). The model accounted for both CO<sub>2</sub> dissolution (Harvey, 1996) and water vaporization (Canjar and Manning, 1967; Saul and Wagner, 1987).

The mineral dissolution and the precipitation reaction rate are calculated from Transient State Theory (TST) (Lasaga and Kirkpatrick, 1981). The equation for the TST rate is:

$$r = \text{sgn} \left[ 1 - \left( \frac{Q}{K_{eq}} \right) \right] \hat{A} S_w \left[ k_0 + \sum_{i=1}^n k_i a_i^{wi} \right] \left| 1 - \left( \frac{Q}{K_{eq}} \right)^\xi \right|^\zeta \quad (2)$$

$$\hat{A} = \hat{A}_0 \cdot \frac{N_m}{N_{mo}} \quad (3)$$

where  $Q$  is the activity product and  $K_{eq}$  is the chemical equilibrium constant. The  $\hat{A}$  and  $\hat{A}_0$  are the reactive surface area at the current time and at zero time respectively.  $N_m$  and  $N_{mo}$  are respectively the current moles of mineral and the moles of mineral at time zero.  $\xi$  and  $\zeta$  are matching parameters. The rate constant  $k_0$  at the current temperature,  $T[K]$ , can be calculated from rate constant  $k_0^*$ .

$$k_0 = k_0^* \exp \left[ -\frac{E_a}{R} \left( \frac{1}{T} - \frac{1}{T^*} \right) \right] \quad (4)$$

where  $E_a$  is activation energy [J/mol] and  $R$  is the gas constant [8.314 J/mol K]. Halite equilibrium reaction with its ions was modeled using the CMG-GEM database (GEOCHEM\_V2):



For this reaction, a reactive surface area of 2163 m<sup>2</sup>/m<sup>3</sup> was used with an activation energy of 7400 J/mol, and a Log10 reaction rate [mol/(m<sup>2</sup>s)] of -0.21. These values were chosen based on the default recommendations of the CMG-GEM software. They can be derived from literature or used as matching parameters. However, the reported values in the literature can be accompanied by considerable uncertainties. In our simulations, the default values from CMG-GEM provided satisfactory results, thus we did not adjust them.

#### Typical water saturation profile

Fig. 19 shows the variation of water saturation along the porous media at different times. The displacement was primarily controlled by the Buckley-Leveret theory and gravity drainage, with the water at the top being displaced quickly in the beginning and the water saturation at the bottom staying high before the breakthrough. Two fronts could be identified by inspecting the water saturation profile. The first front (marked by "1") was the flooding front due to the viscous and gravity forces, and its movement was controlled by the predefined relative permeabilities. There was another dried-out front (marked by "2") lagging behind, with

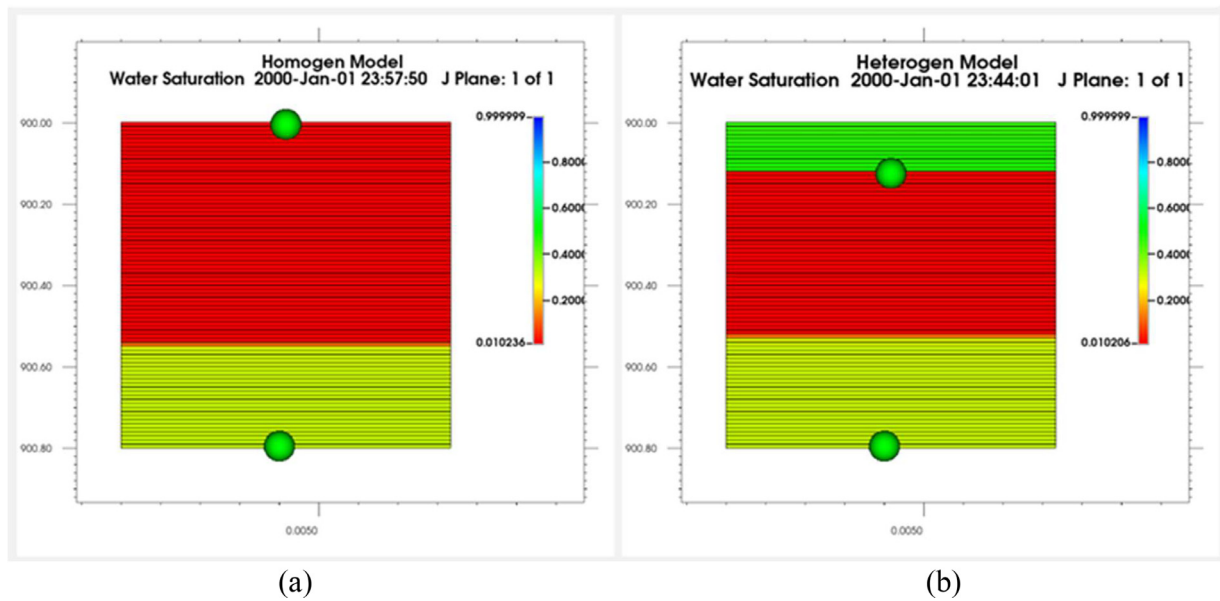


Fig. 20. Water saturation distribution in (a) homogeneous model and (b) heterogeneous model after 80 PV of  $\text{CO}_2$  injection.

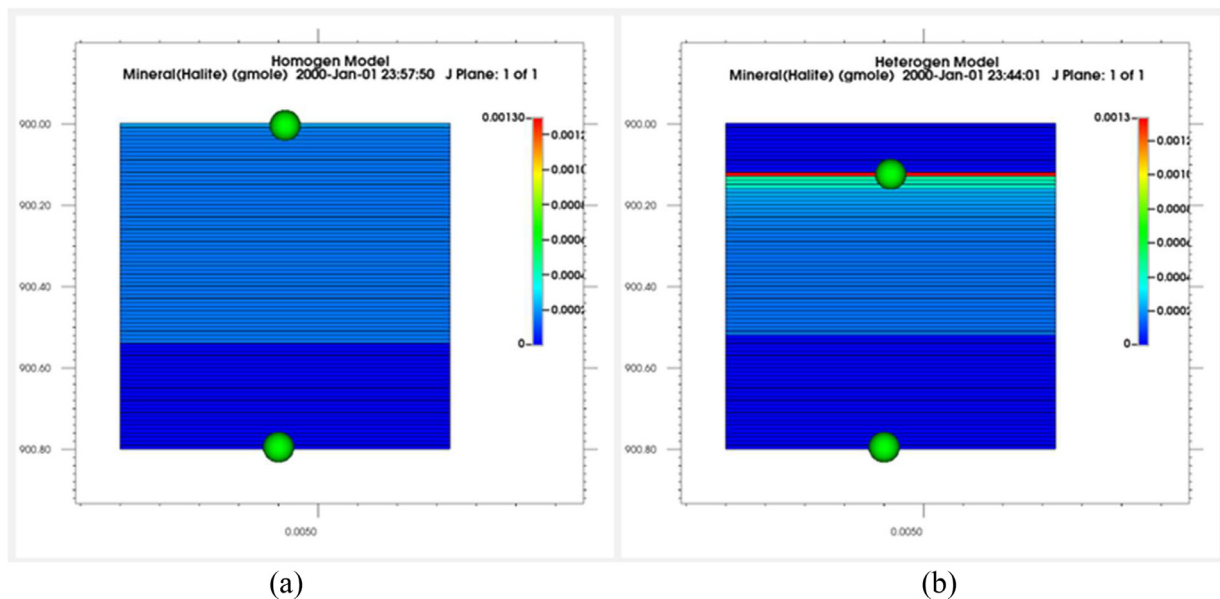


Fig. 21. Halite deposition distribution in (a) homogeneous model and (b) heterogeneous model after 80 PV of  $\text{CO}_2$  injection.

the water saturation reduced to about 0.01 by vaporization. The flooding front moves much faster than the dried-out front. The brine phase between the two fronts appears stationary but was actually under the influence of two-phase flow and vaporization. The net outcome was an enrichment of the brine solution which led to salt precipitation at a later stage.

#### Comparison between experiment and simulation results

The experimental results are compared with the simulation results using two different models. Table 11 provides the water content, the salt content, and the deposited salt amount in each segment in experiment 3. Although the homogeneous model could capture the general production and deposition values, the detailed variations of saturation and deposition were described better with the heterogeneous model. Still, there

was a gap between the predicted extent of dried-out regions and the experimental observation. The experimental dried-out front moves faster toward the producer, indicating a more significant amount of vaporized water and salt deposition. Previously, Khosravi et al. (2015) reported a similar scenario where the actual experimental vaporization rate was higher than the simulated results. It was attributed to spreading and Marangoni-induced turbulence, which increased the interfacial area between phases, and a similar phenomenon was evident for the long tube experiment.

Figs. 20 and 21 show the water and deposited salt distribution at the end of the flooding (after 80 PV injection), respectively. For the homogeneous model, salt deposition mainly occurred at the top, close to the injector, where the water saturation was low, and this contradicts the observation of high-water saturation close to the inlet. For the heterogeneous model, where a water region close to the inlet was deliberately

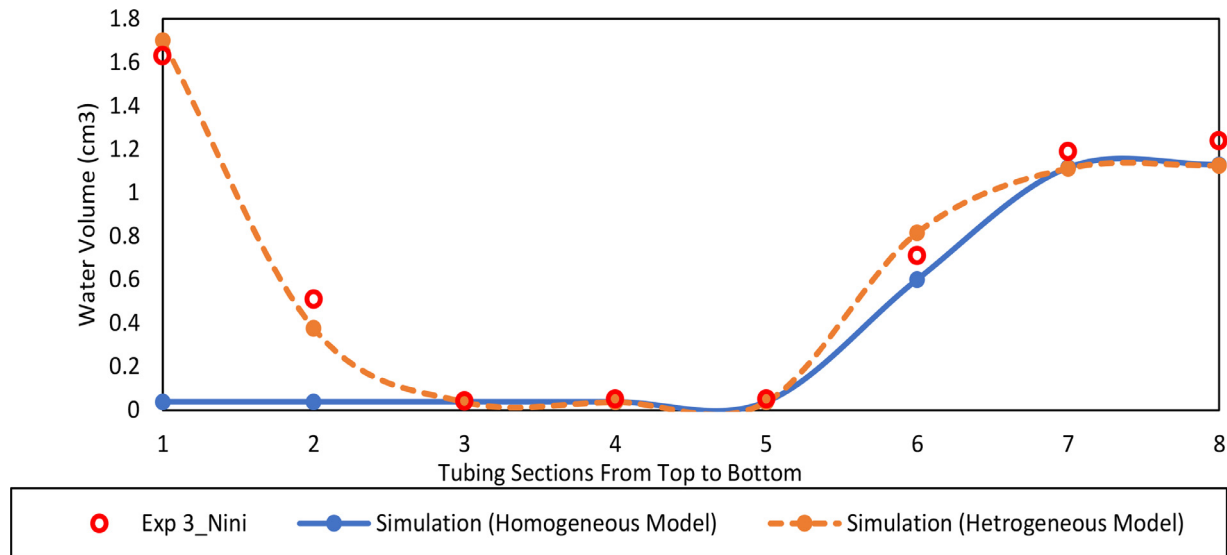


Fig. 22. Comparison of the experimental and simulated salt deposition along different segments of the sand-pack column, after 80 PV of CO<sub>2</sub> injection.

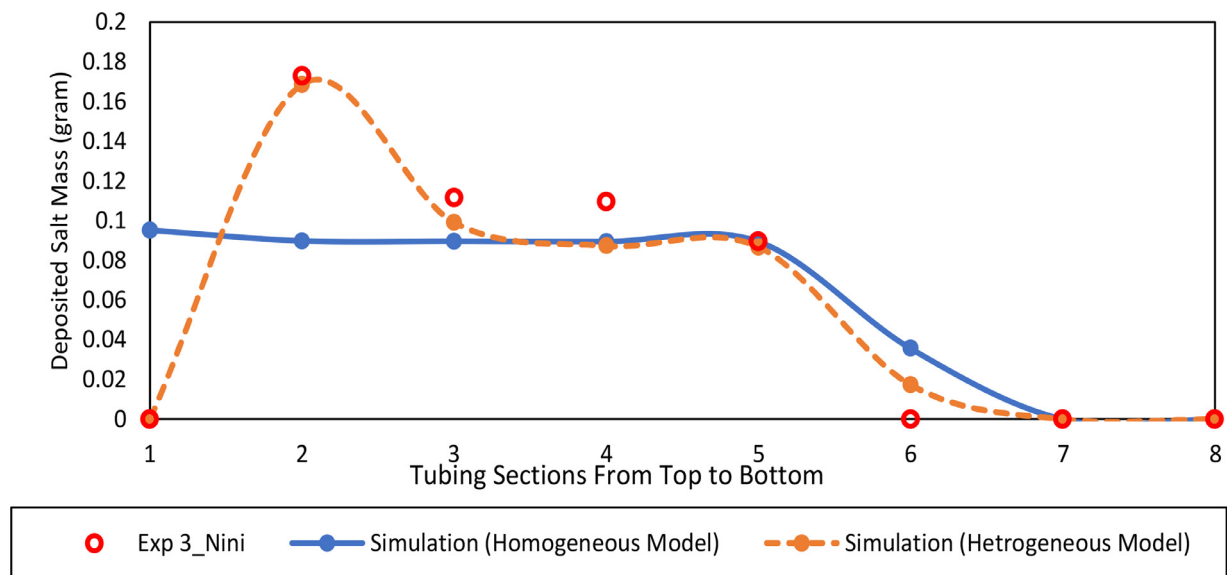


Fig. 23. Comparison of the experimental and simulated water content along different segments of the sand-pack column, after 80 PV of CO<sub>2</sub> injection.

**Table 11**

Comparison between the experimental and simulation results.

| Long Tube Sections | Results of Experiment 3     |                  |                 |                            |                            |                            | Simulation (Homogeneous Model) |                 | Simulation (Heterogeneous Model) |                 |
|--------------------|-----------------------------|------------------|-----------------|----------------------------|----------------------------|----------------------------|--------------------------------|-----------------|----------------------------------|-----------------|
|                    | Water Cont. cm <sup>3</sup> | Salt Cont. grams | Dep. Salt grams | Vap. Water cm <sup>3</sup> | Red. Water cm <sup>3</sup> | Dis. Water cm <sup>3</sup> | Water Cont. cm <sup>3</sup>    | Dep. Salt grams | Water Cont. cm <sup>3</sup>      | Dep. Salt grams |
| 1                  | 1.63                        | 0.21             | -               | -                          | 2.00                       | 2.00                       | 0.04                           | 0.0952          | 1.70                             | 0.0000          |
| 2                  | 0.51                        | 0.28             | 0.1729          | 1.86                       | 3.12                       | 1.26                       | 0.04                           | 0.0898          | 0.38                             | 0.2031          |
| 3                  | 0.04                        | 0.12             | 0.1116          | 1.2                        | 3.59                       | 2.39                       | 0.04                           | 0.0895          | 0.04                             | 0.1018          |
| 4                  | 0.05                        | 0.12             | 0.1095          | 1.18                       | 3.58                       | 2.4                        | 0.04                           | 0.0894          | 0.04                             | 0.0897          |
| 5                  | 0.05                        | 0.10             | 0.0895          | 0.96                       | 3.58                       | 2.61                       | 0.04                           | 0.0894          | 0.04                             | 0.0892          |
| 6                  | 0.71                        | 0.13             | -               | -                          | 2.92                       | 2.92                       | 0.60                           | 0.0357          | 0.82                             | 0.0178          |
| 7                  | 1.19                        | 0.15             | -               | -                          | 2.44                       | 2.44                       | 1.12                           | 0.0000          | 1.11                             | 0.0000          |
| 8                  | 1.24                        | 0.14             | -               | -                          | 2.39                       | 2.39                       | 1.13                           | 0.0000          | 1.13                             | 0.0000          |
| Total              | 5.42                        | 1.25             | 0.4834          | 5.19                       | 23.58                      | 18.39                      | 0.04                           | 0.0952          | 1.70                             | 0.0000          |
| % of Total Model   |                             |                  |                 | 17.91                      | 81.31                      | 63.4                       |                                |                 |                                  |                 |

bypassed, the simulation captures the high-water saturation in the upper segment as well as a high deposited salt content in the middle of the column.

The results of the heterogeneous model are in better agreement with the experimental observation. It can be hypothesized that certain compaction happened in the upper segments (the first segment and a part of the second one), which caused a change in the relative permeability curves and an increase in the irreducible water saturation. The injected gas bypassed this zone via a small channel and dried the segments below. Eventually, brine drained from the bypassed region under gravity into the dried-out segments just beneath it. The brine entering the new zone was quickly vaporized, resulting in salt deposition in the entry region. In the simulation with the heterogeneous model, the highest amount of salt deposition was obtained in the second segment. Fig. 21 also shows that the maximum salt deposition always happens in the first grid of the dried-out region no matter which model was used. The channeling in the heterogeneous model mainly moves the dried-out region deeper into the tube. In the dried-out region, the upper part had a higher salt deposition due to more exposure to dry injection gas.

Figs. 22 and 23 compare the experimental and simulated water content and deposited salt amount in each segment. The deposited salt amount was estimated using the total salt and saturated brine salinity. Fig. 22 shows that both models reasonably described the water content for segments 3 to 8. The major difference was that only the heterogeneous model could capture the high-water content close to the inlet. Similarly, Fig. 23 shows that the two models give similar deposited salt for segments 3 to 8, but only the heterogeneous model can capture the salt precipitation in the upper part (segments 1 and 2). In the homogeneous model, the salt precipitation in the inlet was relatively high. But a large amount of brine was bypassed and not vaporized close to the inlet in the heterogeneous model, which suppresses the salt precipitation.

## Conclusions

Water vaporization associated with the huge amount of injected CO<sub>2</sub> can result in salt precipitation and even injectivity impairment in the near wellbore region. In this work, a new experimental method using a long tube with packed sand was developed to study salt precipitation on a larger scale as compared with the shorter lengths in conventional core floodings. After around 80 pore volumes of CO<sub>2</sub> injection, the tube was cut into 8 equal segments for different analyses, such as the water content, the salt content, and the SEM analysis. Compared to conventional flooding, the long-tube test provides more insight on the water and salt distribution in the porous media after the flooding. The SEM analysis clearly indicated the presence of halite in each section of the tubing. Only the inlet and outlet showed relatively high-water saturation, and the water in the middle part was largely evaporated. A salt content larger than 1 wt% was observed throughout the tubing, but only the middle segments deposited salt. Consolidation of the packed sand was found in some segments close to the inlet and outlet. No significant increase in the pressure drop has been observed after the CO<sub>2</sub> injection.

After the completion of experimental studies, a numerical simulation was carried out on the long-tube flooding results of experiment 3 using both a homogeneous model and a heterogeneous one. The typical salt-precipitation solution consists of two fronts: a leading front of immiscible displacement and a trailing front of vaporization, with the latter giving a dried-out zone, reflected in the simulation of the homogeneous model. In the homogeneous model, the salt deposition occurs mainly at the top, close to the injector where the water saturation was low, and this phenomenon contradicts our observation of high-water saturation close to the inlet. Hence, it was necessary to use a heterogeneous model, which can account for the bypassing of the brine close to the inlet, to capture the observed water and salt distribution. The heterogeneous model produces a dried-out zone in the middle of the tube and the highest amount of deposited salt in the upper part of this zone, which was in agreement with experimental results.

The long-tube flooding test provides a complementary approach to the conventional core flooding. It is recommended to adopt such a test in the laboratory investigation on salt precipitation. The current study is limited to one salinity and two flow rates. A wider range of conditions (temperature, pressure, salinity, and flow rate) should be investigated in the future. More tests with longer tubes can be conducted to determine the length effect and the optimal length. In the current long-tube flooding design as well as the conventional flooding design, the cross flow (perpendicular to the main displacement direction) from the neighboring region is not reflected. Since the capillary-driven cross flow is important to salt precipitation, it should be considered how to extend both flooding designs, which are essentially for one dimensional displacement, to honor this effect in a second dimension.

## Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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