



## Original Paper

# Similarity-based laboratory study of CO<sub>2</sub> huff-n-puff in tight conglomerate cores



Yu-Long Yang<sup>a, b, c, 1</sup>, Yu Hu<sup>a, b, c, 1</sup>, Ya-Ting Zhu<sup>d</sup>, Ji-Gang Zhang<sup>d</sup>, Ping Song<sup>d</sup>, Ming Qin<sup>d</sup>,  
Hai-Rong Wu<sup>a, b, c, \*</sup>, Zhao-Jie Song<sup>a, b, c, \*\*</sup>, Ji-Rui Hou<sup>a, b, c, \*\*\*</sup>

<sup>a</sup> State Key Laboratory of Petroleum Resources and Prospecting, China University of Petroleum-Beijing, Beijing 102249, China

<sup>b</sup> College of Carbon Neutral Energy, China University of Petroleum-Beijing, Beijing 102249, China

<sup>c</sup> Unconventional Petroleum Research Institute, China University of Petroleum-Beijing, Beijing 102249, China

<sup>d</sup> Research Institute of Petroleum Exploration and Development, Xinjiang Oilfield, Karamay 834000, China

## ARTICLE INFO

## Article history:

Received 25 June 2022

Received in revised form

20 September 2022

Accepted 24 September 2022

Available online 30 September 2022

Edited by Yan-Hua Sun

## Keywords:

Tight conglomerate reservoir

CO<sub>2</sub> huff-n-puff

Similarity-based equivalent pressure

Enhanced oil recovery

## ABSTRACT

Tight conglomerate reservoirs are featured with extremely low permeability, strong heterogeneity and poor water injectivity. CO<sub>2</sub> huff-n-puff has been considered a promising candidate to enhance oil recovery in tight reservoirs, owing to its advantages in reducing oil viscosity, improving mobility ratio, quickly replenishing formation pressure, and potentially achieving a miscible state. However, reliable in-house laboratory evaluation of CO<sub>2</sub> huff-n-puff in natural conglomerate cores is challenging due to the inherent high formation pressure. In this study, we put forward an equivalent method based on the similarity of the miscibility index and Grashof number to acquire a lab-controllable pressure that features the flow characteristics of CO<sub>2</sub> injection in a tight conglomerate reservoir. The impacts of depletion degree, pore volume injection of CO<sub>2</sub> and soaking time on ultimate oil recovery in tight cores from the Mahu conglomerate reservoir were successfully tested at an equivalent pressure. Our results showed that oil recovery decreased with increased depletion degree while exhibiting a non-monotonic tendency (first increased and then decreased) with increased CO<sub>2</sub> injection volume and soaking time. The lower oil recoveries under excess CO<sub>2</sub> injection and soaking time were attributed to limited CO<sub>2</sub> dissolution and asphaltene precipitation. This work guides secure and reliable laboratory design of CO<sub>2</sub> huff-n-puff in tight reservoirs with high formation pressure.

© 2022 The Authors. Publishing services by Elsevier B.V. on behalf of KeAi Communications Co. Ltd. This is an open access article under the CC BY-NC-ND license (<http://creativecommons.org/licenses/by-nc-nd/4.0/>).

## 1. Introduction

Despite the worldwide growth of oil production from tight reservoirs, over 90% of tight oil remains underground (Zou et al., 2014; Sheng, 2015, 2017). Oil exploitation in tight reservoirs counts on the natural formation energy at the primary recovery stage. However, the primary oil recovery typically confines to 5%–

15% of original oil in place (OOIP) due to low natural energy and rapidly descending formation pressure, signifying the tremendous potential for enhanced oil recovery (EOR) (Christensen et al., 2001; Zheng et al., 2014; Li et al., 2021a, 2021b; Syed et al., 2022). Conventional water flooding exhibits poor injectivity, potential water sensitivity, and strong water channeling, ascribing to the inherent pressure sensitivity, clay-rich mineral compositions, great specific surface area, and severe heterogeneity in tight reservoirs. This yields difficulties in establishing an effective water-displace-oil system, thus low well-production and undesirable water injection performance (Sun et al., 2021; Zhou and Zhang, 2020).

Great attention has been increasingly paid to evaluating the viability of CO<sub>2</sub> injection for EOR in tight reservoirs (Hoteit and Firoozabadi, 2009; Hawthorne et al., 2013, 2019; Jin et al., 2017), owing to the distinguished advantages of CO<sub>2</sub> and the global goal of achieving carbon neutrality. Mass transfer of CO<sub>2</sub> into reservoir oil generally leads to oil swelling and viscosity reduction, affecting oil

\* Corresponding author. State Key Laboratory of Petroleum Resources and Prospecting, China University of Petroleum-Beijing, Beijing 102249, China.

\*\* Corresponding author. State Key Laboratory of Petroleum Resources and Prospecting, China University of Petroleum-Beijing, Beijing 102249, China.

\*\*\* Corresponding author. State Key Laboratory of Petroleum Resources and Prospecting, China University of Petroleum-Beijing, Beijing 102249, China.

E-mail addresses: [hrwu@cup.edu.cn](mailto:hrwu@cup.edu.cn) (H.-R. Wu), [songz@cup.edu.cn](mailto:songz@cup.edu.cn) (Z.-J. Song), [houjirui@126.com](mailto:houjirui@126.com) (J.-R. Hou).

<sup>1</sup> These authors contributed equally to this work.

mobility and molecular diffusion of CO<sub>2</sub> and oil components. It is also worth mentioning that CO<sub>2</sub> can reach a supercritical state when temperature and pressure are higher than their corresponding critical values (31.2 °C and 7.38 MPa), showcasing dual properties of gas and liquid. Given the high temperature and high pressure of tight reservoirs, the injected CO<sub>2</sub> is anticipated to migrate as a supercritical solvent, yielding a fully miscible state with oil that favors oil recovery (Hou et al., 2017; Chen et al., 2022).

Nevertheless, continuous CO<sub>2</sub> flooding is impractical in a tight reservoir because the remaining oil in the matrix cannot be driven out. Consequently, cyclic injection such as huff-n-puff (HnP) is considered an alternative for the EOR of a single well. HnP comprises three distinct scenarios. CO<sub>2</sub> is injected into the reservoir in the “huff” stage, followed by shutting the injection well to allow sufficient interaction between CO<sub>2</sub> and oil (the so-called “soaking” period). It has been recognized that CO<sub>2</sub> transport in tight reservoirs is likely dominated by diffusion rather than convection due to the ultralow permeability (Hawthorne et al., 2013; Cronin et al., 2018). Therefore, soaking time is crucial to oil recovery enhanced by CO<sub>2</sub> huff-n-puff. Field trials in the Bakken oil reservoir have confirmed that the oil-production rate can significantly increase following a certain soaking period (Alfarge et al., 2018). After a sufficient soaking time, the injection well is reopened at a low pressure to produce CO<sub>2</sub> as well as a CO<sub>2</sub>-rich oil phase. An increase of 30%–50% in ultimate oil recovery compared with primary production was reached by carrying out CO<sub>2</sub> huff-n-puff in the south Texas Eagle Ford reservoir (Rassenfoss, 2017).

Mahu tight conglomerate reservoir, located at Junggar Basin, is the world's largest conglomerate reservoir with abundant reserves, discovered in 2017 (Zhang et al., 2022). Nevertheless, due to its complexities in lithology and pore structures, the exploitation of tight conglomerate reservoirs is still in its infancy. Therefore, CO<sub>2</sub> HnP in Mahu tight conglomerate reservoir is a relatively new challenge, with fundamental questions remaining, such as an appropriate primary recovery period, optimal soaking time and injection volume. Albeit laboratory study provides an effective way to examine the effect of CO<sub>2</sub> HnP, the high pressure (over 50 MPa) of Mahu tight conglomerate reservoir restrains the reliability of core-scale laboratory due to serious security concerns. Table 1 summarizes experimental conditions of CO<sub>2</sub> injection in tight cores that have been reported in recent literature. As observed, the highest experimental pressure available for reference is 43 MPa, and in-house CO<sub>2</sub> injection under pressures representative of the Mahu tight conglomerate reservoir has not been reported. This also impedes creating and calibrating numerical representation of core samples that allows one to assess the process efficiency (Zuloaga et al., 2017; Janiga et al., 2018). Accordingly, it is urgent to figure out an appropriate method to address high-pressure CO<sub>2</sub> injection in the laboratory, balancing lab security and confidence level.

In the current work, we proposed a similarity-based approach to determining an equivalent experimental pressure, enabling one to obtain a lab-controllable pressure representing the flow characteristics of CO<sub>2</sub> injection in high-pressure tight reservoirs. The impacts of depletion degree, injection volume of CO<sub>2</sub> and soaking time on ultimate oil recovery in tight cores of the Mahu conglomerate reservoir were successfully tested at an equivalent pressure. This work evaluated the feasibility of CO<sub>2</sub> HnP in the Mahu conglomerate reservoir and guided the designing of a secure and credible laboratory study of CO<sub>2</sub> injection in tight reservoirs with high formation pressure.

## 2. Experimental study

### 2.1. Materials and preparations

#### 2.1.1. Simulated oil

The degassed crude oil with a density of 0.7444 kg/m<sup>3</sup> and a viscosity of 0.2778 cP, taken from the Mahu tight conglomerate reservoir (Xinjiang Oilfield, China), was used to prepare the simulated oil. Light components are added to the crude oil to simulate the formation oil and acquire a manageable test pressure representing the reservoir condition (the method to determine the content of light gas is detailed in Section 2.2.2). Oil compositions were determined using a gas chromatography spectrometry (Agilent 7890A). The obtained total hydrocarbon chromatography of the simulated oil is presented in Fig. 1.

#### 2.1.2. CO<sub>2</sub>

CO<sub>2</sub> with a purity of 99.99% used in our experiments was purchased from Beijing Jinggao Gas Manufacturing Co., Ltd.

#### 2.1.3. Artificial formation water

The artificial formation water (AFW) with the same ionic compositions as that in the studied reservoir was prepared using deionized ultrapure water (Millipore Corporation, USA) and high-purity salt. The main components of AFW are demonstrated in Table 2. The brine was degassed to prevent dissolved air from damaging the core permeability (Russell et al., 2017).

#### 2.1.4. Cores

The cores used to conduct experiments were taken from Mahu oil field (Xinjiang Oilfield Company, China). The basic parameters of the cores are shown in Table 3. The cores were cleaned before experiments by injecting petroleum ether due to the presence of formation oil inside and then dried in an oven at 80 °C for more than 24 h.

### 2.2. Methodologies

#### 2.2.1. Determination of minimum miscibility pressure (MMP)

The MMP of CO<sub>2</sub> in the degassed crude oil was determined using the slim tube method. The experimental procedure follows Li et al. (2021b). The temperature was maintained at 80 °C, consistent with the targeted reservoir. Since the light components of the crude oil were volatilized as soon as they were taken to the ground, we are ignorant of the original oil components. Despite the deviation between the measured MMP and the practical MMP underground, it is worth noting that our measurement provides an upper limit of MMP that could be considered as a reference for the pilot tests of CO<sub>2</sub> huff-n-puff.

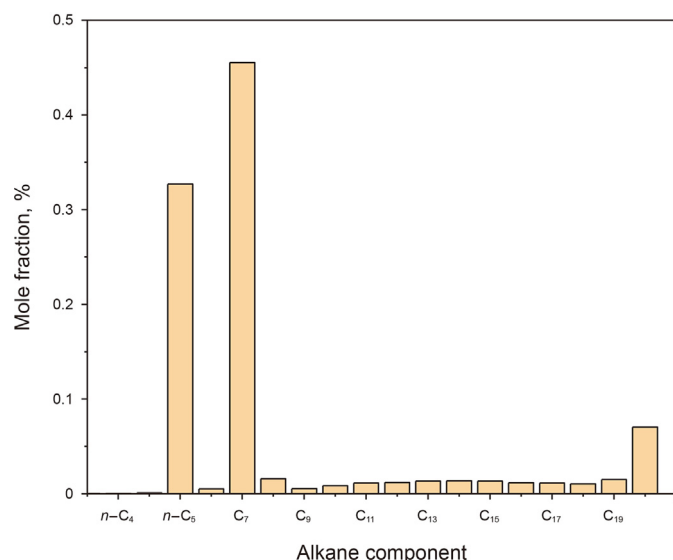
#### 2.2.2. Determination of equivalent pressure

Due to the high formation pressure of the targeted reservoir, we propose to conduct the CO<sub>2</sub> huff-n-puff experiments under an equivalent safe pressure in the laboratory, determined by the similarity of miscibility index  $\lambda$  and Grashof number  $Gr$ . The miscibility index, defined as the ratio of formation pressure to the MMP obtained via the slim tube method, denotes the degree of miscibility. The Grashof number, defined as the square of the ratio of density to viscosity multiplied by a constant, features the degree of natural convection. The specific procedure for acquiring the equivalent pressure is as follows.

- (1) Establish a PVT model using the WinProp mode of CMG (a reservoir simulation software) to calculate MMP based on experimental parameters, such as crude oil compositions

**Table 1**  
Experimental parameters of CO<sub>2</sub> injection from the literature.

References	Core types	Permeability, mD	Methods	Pressure, MPa	Temperature, °C
Du et al., 2020	Sandstone	0.182–0.319	Flooding/HnP	13	60
Yu et al., 2021	NA	0.028–0.193	Flooding	20	90
Huq et al., 2015	Sandstone	11.73	Flooding	5	125
Smith et al., 2013	Caprock	0.0005	Flooding	24.8	60
Wigand et al., 2008	Sandstone	0.5	Flooding	15	60
Zou et al., 2018	Shale	0.000061 and 0.000232	Soaking	10–30	40–120
Pu et al., 2016	Sandstone	0.30	HnP	4–26	75
Wei et al., 2019	Sandstone	0.95	Flooding	35	80
Li and Gu, 2014	Sandstone	0.28–2.78	Flooding	12	53
Dong et al., 2020	Sandstone	0.1	HnP	25	25
Bai et al., 2019b	Sandstone	0.03	HnP	40	60
Wang et al., 2017	Sandstone	0.218	Soaking	12	40
Zhou et al., 2020	NA	1.33–3.25	Flooding	12.18	50
Zhu et al., 2020	Shale/sandstone	0.0053–0.018	HnP	16	60
Wang et al., 2021	Sandstone	0.1–1.0	Flooding	18	60
Pu et al., 2021	Conglomerate	0.88	HnP	9.8	69
Hu et al., 2020	NA	0.2–0.4	Flooding	0.69	60
Syah et al., 2021	Sandstone	0.05–0.9	Flooding/HnP	13–16	80
Zhou et al., 2019	Sandstone	0.98–11.6	Flooding	12.9	44
Bai et al., 2019a	NA	0.89–9.10	HnP	14	65
Qian et al., 2018	Sandstone	0.36–0.81	HnP	5–16	61
Wei et al., 2017	Sandstone	0.63	HnP	34	75
Ding et al., 2021	Artificial sandpacks	0.2–300	HnP	23	108
Fernø et al., 2015	Shale	0.00074–0.0017	Flooding	22.1	60
Gao and Pu, 2021	Conglomerate	0.16	HnP	37	89
Ma et al., 2019	Sandstone	0.03–0.47	HnP	43	81
Ma et al., 2015	NA	2.3	HnP	12.9	44
Wei et al., 2020a	Sandstone	0.813	HnP	35	80
Wei et al., 2020b	Sandstone	0.79–2.43	Flooding/HnP	25	75



**Fig. 1.** Oil compositions determined using total hydrocarbon chromatography.

based on total hydrocarbon chromatography, temperature, pressure, etc. Unknown parameters, e.g., interaction coefficients, are calibrated by the slim-tube-measured MMP corresponding to reservoir conditions (MMP<sub>res</sub>).

- (2) Calculate the miscibility index of reservoir,  $\lambda_{res}$ , using formation pressure  $P_{res}$  and the slim-tube-measured MMP<sub>res</sub>.

**Table 2**  
Main components of AFW.

Salt	NaCl	KCl	Na <sub>2</sub> SO <sub>4</sub>	CaCl <sub>2</sub>	MgCl <sub>2</sub>	NaHCO <sub>3</sub>	Total
Concentration, mg/L	978.14	2249.18	286.4	309.23	31.92	989.73	8246.36

**Table 3**  
Core parameters.

No.	Porosity, %	Diameter, mm	Length, mm	Mass, g	Permeability, mD
K4	21.91	2.600	6.214	78.554	0.2187
K10	22.92	2.516	7.239	80.403	0.0664
K11	22.94	2.529	6.716	80.058	0.1425
K12	20.20	2.525	7.168	87.539	0.1004
K13	20.04	2.514	7.287	87.856	0.0361
K14	18.66	2.520	7.797	92.343	0.0607
K15	25.48	2.510	4.973	58.103	0.2024

- (3) Change oil compositions by adding light components (*n*-heptane and *n*-pentane) and obtain the corresponding minimum miscibility pressure MMP<sub>lab</sub>.
- (4) Obtain the experimental pressure  $P_{exp}$  via the similarity of miscibility index of reservoir and experiment, i.e., setting  $\lambda_{exp} = \lambda_{res}$ . Adding light components generally yields a lower MMP (Abedini and Torabi, 2014) and thus, leads to lower experimental pressure.
- (5) Calculate the viscosities and densities of oil and CO<sub>2</sub> correspond to  $P_{res}$  and  $P_{exp}$  using the flash mode of the established PVT model.
- (6) Obtain the Grashof numbers corresponding to the reservoir and experimental conditions, and check the similarity of *Gr*. If  $|Gr_{exp} - Gr_{res}| \leq 0.001$ , the acquired  $P_{exp}$  in step (4) is selected as long as its value meets the laboratory safety requirement. Otherwise, repeat step (3) and adjust the amount of added light components until

$|Gr_{\text{exp}} - Gr_{\text{res}}| \leq 0.001$  is satisfied, and the corresponding experimental pressure falls within a safety zone.

### 2.2.3. Experimental procedures of CO<sub>2</sub> huff-n-puff

A schematic diagram of the laboratory setup for CO<sub>2</sub> huff-n-puff is illustrated in Fig. 2. The CO<sub>2</sub> huff-n-puff tests were carried out after depletion. The experimental procedure is given as follows.

- (1) The core was placed within the core holder. The permeability was measured while setting the confining pressure as 10 MPa. Then the core holder was connected to the vacuum pump and vacuumized for over 5 h.
- (2) The core was saturated with the prepared artificial formation water. The injected AFW was recorded, and the pore volume and porosity were calculated. The temperature was set at 80 °C. The pressure of the back-pressure valve was set as 23 MPa.
- (3) The simulated oil aged for three days was injected into the core at 0.1 mL/min until the oil was produced and water was not observed at the core outlet. The volume of saturated oil was recorded.
- (4) Open the valve at the outlet, and the pressure of the check valve was gradually lowered to a target value. The variations in pressure and produced oil were recorded.
- (5) CO<sub>2</sub> was injected at a constant rate of 0.1 mL/min. All valves were closed to soak after injecting a certain amount of CO<sub>2</sub>.
- (6) The valve at the outlet was open, and the back-pressure valve was regulated to produce oil after a target soaking time. CO<sub>2</sub> huff-n-puff was processed until oil was no longer produced. The variations in pressure and oil production were recorded.
- (7) Steps (1–6) were repeated to explore the impact of depletion degree, CO<sub>2</sub> injection volume and soaking time on enhanced oil recovery.

## 3. Results and discussion

### 3.1. MMP of CO<sub>2</sub> in crude oil

The minimum miscibility pressure of CO<sub>2</sub> in crude oil was determined by the slim tube method at 80 °C. The relationships of

oil-displacement efficiency and injection volume of CO<sub>2</sub> under various pressures (7, 15, 25, 32, 40, and 50 MPa) were plotted in Fig. 3a. Generally, the oil-displacement efficiency after 1.2 pore volume (PV) of CO<sub>2</sub> injection is considered the oil-displacement efficiency corresponding to the experimental pressure, and the turning point of the curve showing the relationship between the oil-displacement efficiency and the driving pressure denotes MMP. Therefore, as seen in Fig. 3b, the MMP of CO<sub>2</sub> in crude oil is 28.5 MPa approximately.

Despite its long-lasting measurements, the slim tube test is an ideal one-dimensional model of the reservoir, bringing about multiple equilibrium contacts between simultaneous flowing fluids, widely accepted as the “industry standard” for determining MMP (Elsharkawy et al., 1996; Dong et al., 2001; Zhang and Gu, 2015). The long slim tube yields a minimized effect of transition zone length, and the small tube diameter mitigates viscous fingering. Unfortunately, a standard for test design, operating procedure, and criteria for determining MMP with slim tubes is absent, which may result in discrepancies from test to test. Moreover, the impacts of packing material and porosity of a packed slim tube on MMP remain controversial (Elsharkawy et al., 1996), giving rise to difficulties in comparing obtained data with those from other time-saving techniques (e.g., the pressure rising bubble apparatus, the pressure-density diagram, the vanishing interfacial tension, the sonic response method, the rapid pressure increase method, etc.) (Hawthorne et al., 2013; Liu et al., 2016; Czarnota et al., 2017a, 2017b).

### 3.2. Equivalent pressure

Following the procedure introduced in Section 2.2.2, the obtained equivalent pressure was 22.82 MPa. The contents of added light components and relevant parameters are listed in Table 4. Accordingly, the CO<sub>2</sub> huff-n-puff experiments using the simulated oil were performed under a back pressure of 22.82 MPa.

### 3.3. CO<sub>2</sub> huff-n-puff experiments

#### 3.3.1. Impact of depletion degree on EOR of CO<sub>2</sub> huff-n-puff

Greater depletion commonly leads to lower formation pressure and a weaker effect of pressure replenishment by gas injection.

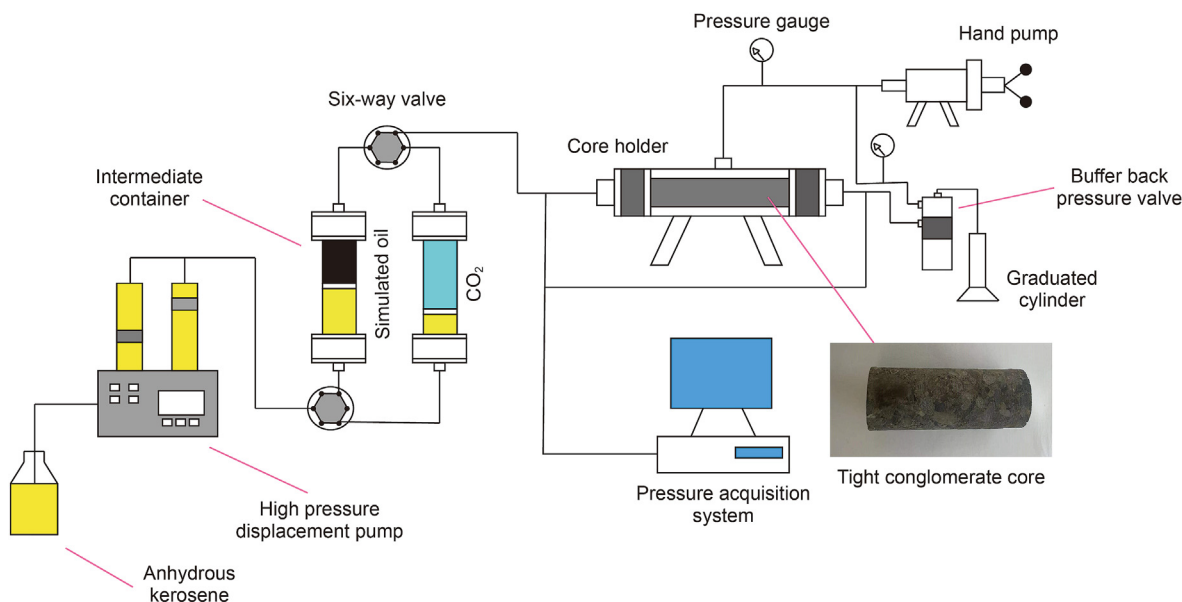


Fig. 2. Schematic laboratory setup for CO<sub>2</sub> huff-n-puff.

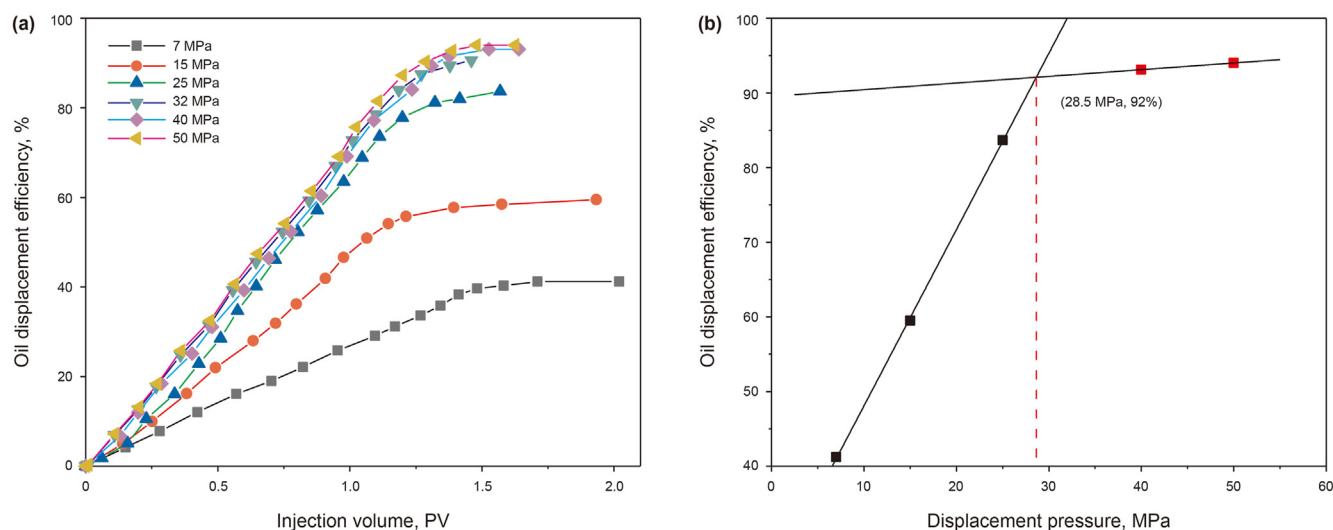


Fig. 3. Results of slim tube tests: (a) Oil-displacement efficiency vs. pore volume of CO<sub>2</sub> injection; (b) Steady oil-displacement efficiency vs. displacement pressure.

Table 4

Oil components and relevant parameters corresponding to reservoir and experimental conditions.

Oil component, g		MMP, MPa	<i>P</i> , MPa	Density, g/cm <sup>3</sup>	Viscosity, cP	<i>Gr</i>	$\gamma$	
Crude oil	C <sub>7</sub>	C <sub>5</sub>						
15.584	0	0	27.925	53	0.7444	0.2778	7.180394	1.8974
15.584	13.032	6.9	12.025	22.8227	0.7077	0.2641	7.180817	1.8974

Consequently, an appropriate depletion degree is crucial to CO<sub>2</sub> huff-n-puff. We tested the impacts of different depletion degrees (30%, 20%, and 10%) on the recovery rate of CO<sub>2</sub> huff-n-puff. The corresponding CO<sub>2</sub> injection volume and soaking time are 0.5 PV and 10 h, respectively. The physical parameters of cores used in these experiments are presented in Table 3 (rows 2–4). All experiments were conducted under the equivalent pressure acquired in Section 3.2. Fig. 4 shows experimental results of CO<sub>2</sub> huff-n-puff under various depletion degrees.

The depletion stage simulates oil recovery under natural reservoir energy. Fig. 4a demonstrates the inlet and outlet pressures and recovery factors of CO<sub>2</sub> HnP when the outlet pressure was depleted to 30% of the initial backpressure. The inlet and outlet pressures at the beginning of depletion were 23.56 and 22.88 MPa, respectively, and then declined downward to 16.86 and 16.03 MPa at the end of the depletion scenario. The recovery factor was approximately 3.03% during depletion. The internal pressure rose slowly with CO<sub>2</sub> injection at the 'huff' scenario, suggesting effective pressure replenishment. At the stage of soaking, CO<sub>2</sub> injection ended while the internal pressure continued to increase gradually, owing to the increased elastic energy induced by dissolved CO<sub>2</sub> in oil that leads to oil swelling. Produced oil was collected at the 'puff' stage. The internal pressure continuously declined until oil production ceased. The oil recovery factor increased by 5.79% during CO<sub>2</sub> huff-n-puff.

In comparison, despite the similar tendency at lower depletion degrees of 20% (Fig. 4b) and 10% (Fig. 4c), higher remaining oil at lower depletion degrees led to increased internal pressure as well as a higher recovery factor. Oil recovery factors corresponding to 20% and 10% depletions were 6.68% and 7.65%, respectively. This might be ascribed to a greater amount of dissolved CO<sub>2</sub>, resulting in more significant gas–oil–rock interactions and higher elastic energy.

Fig. 5a compares the recovery factors under different depletion degrees. The recovery factor decreases with increased depletion

degree, demonstrating that CO<sub>2</sub> can more effectively interact with more oil under higher core pressure, yielding more significant oil swelling and viscosity reduction. Moreover, the diffusion of CO<sub>2</sub> molecules leads to higher oil recovery from small pores that cannot be accessed during depletion.

### 3.3.2. Impact of CO<sub>2</sub> injection volume on EOR of CO<sub>2</sub> huff-n-puff

Injection volume of CO<sub>2</sub> can significantly affect oil recovery. However, an excess of gas injection may not be economical as the mutual interactions between CO<sub>2</sub> and oil are limited when CO<sub>2</sub> injection is over a critical value. Thus, it is important to determine an optimized volume of CO<sub>2</sub> injected. We compared oil recoveries under 0.5, 0.75, and 1.0 PV at 10% depletion. The physical parameters of cores used in these experiments are presented in Table 3 (rows 5–6).

Fig. 5b shows the recovery factor under different CO<sub>2</sub> injection volumes. The recovery factor presents a non-monotonic variation with increased CO<sub>2</sub> injection volume, indicating the existence of an optimal value around 0.75 PV injection of CO<sub>2</sub>. As more CO<sub>2</sub> dissolves into oil, the oil swells with a remarkable viscosity decreasing. As oil production goes on, the core pressure declines gradually. When the pressure is lower than the bubble point pressure, CO<sub>2</sub> separates from oil, driving a distinguished rising in oil production at the late production stage. However, the recovery factor at 1 PV CO<sub>2</sub> injection is lower than that at 0.75 PV, which may be restricted by limited CO<sub>2</sub> dissolution and asphaltene precipitation. The combination of asphaltene and resin components at high pressure and temperature is inhibited, leading to severe asphaltene aggregation and precipitation. This phenomenon is more prominent at high pressure due to the shortened distance between asphaltene molecules (Cao and Gu, 2013). The precipitated asphaltene adsorbs at solid surfaces or blocks pore throats. Shen and Sheng (2018) experimentally reported that matrix permeability could be reduced by 25%–50% due to asphaltene deposition during CO<sub>2</sub> huff-



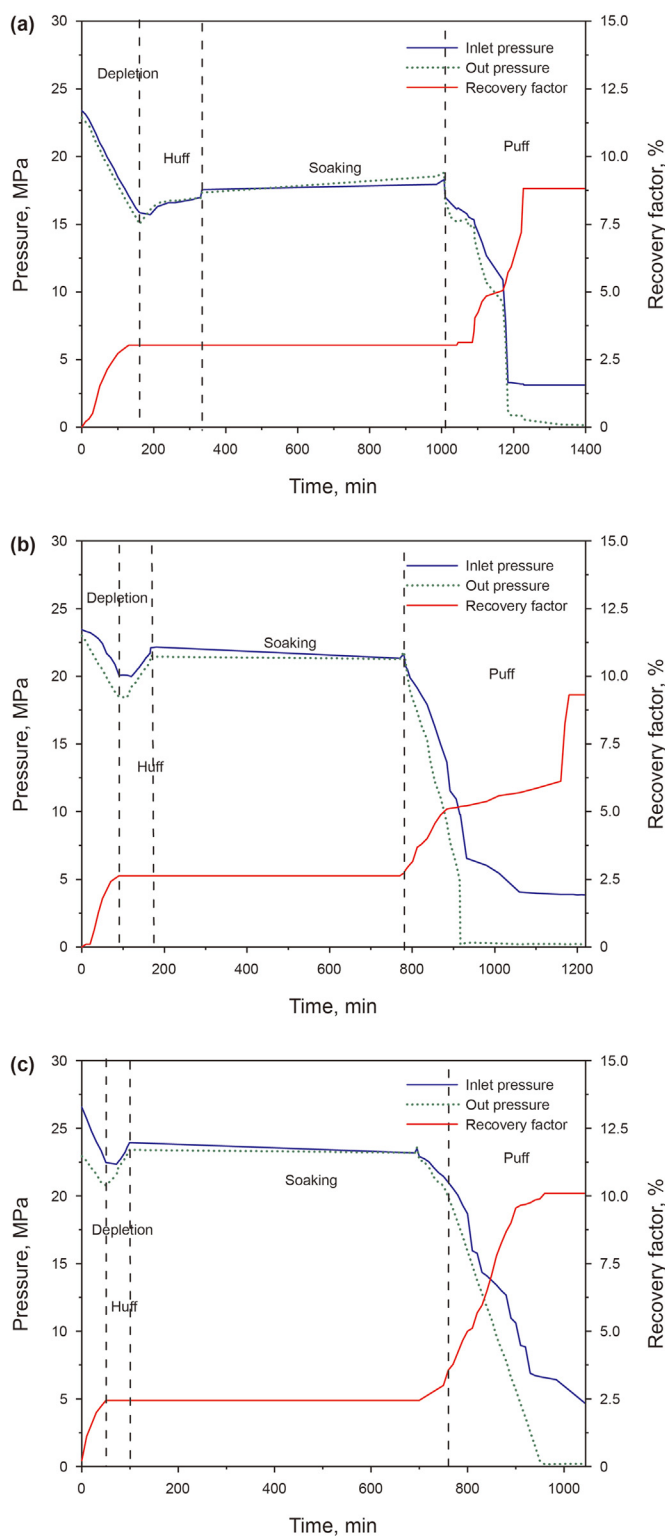


Fig. 4. Inlet and outlet pressures and recovery factors of CO<sub>2</sub> huff-n-puff under various depletion degrees: (a) 30% depletion; (b) 20% depletion; (c) 10% depletion.

n-puff using Eagle Ford outcrop cores saturated with dead oil. Therefore, formation damage caused by excess CO<sub>2</sub> injection may decrease oil recovery. Nevertheless, it is worth noting that the permeability of the core used for 0.75 PV test is nearly one order of magnitude lower than that used for 1.0 PV test, which may have an

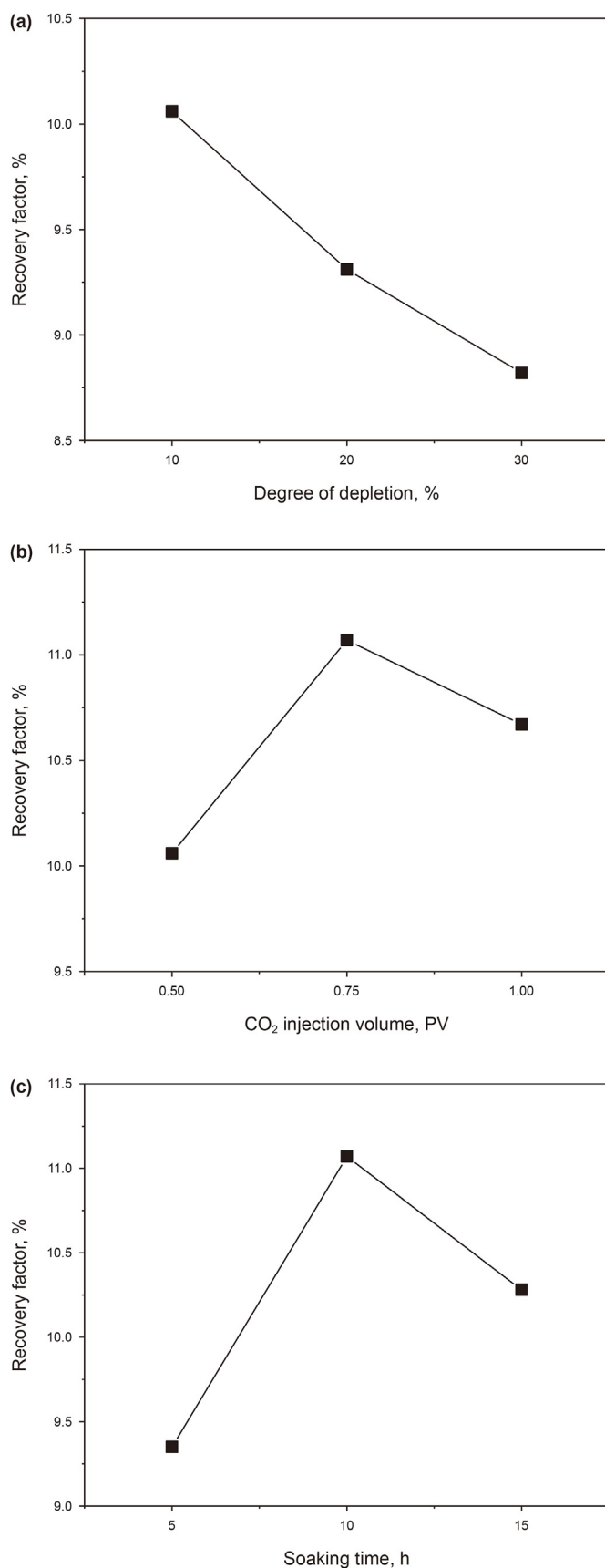


Fig. 5. Recovery factors under different depletion degrees (a), CO<sub>2</sub> injection volumes (b), and soaking times (c).

interference on the final oil recovery.

### 3.3.3. Impact of soaking time on EOR of CO<sub>2</sub> huff-n-puff

Soaking time determines whether CO<sub>2</sub> and oil interact adequately to achieve effective oil swelling and viscosity reduction, light component extraction, and surface wettability alteration. Accordingly, soaking time is also crucial to the EOR of CO<sub>2</sub> huff-n-puff. We tested oil recoveries under three soaking periods (5, 10, and 15 h) at 10% depletion and 0.75 PV injection of CO<sub>2</sub>. The physical parameters of cores used in these experiments are presented in Table 3 (rows 7–8).

Similar to the impact of CO<sub>2</sub> injection volume, the recovery factor shows a non-monotonic tendency, i.e., it increases first and then decreases with increased soaking time (Fig. 5c). The internal core pressure is higher than the MMP (12.025 MPa) of CO<sub>2</sub> in the simulated oil, inferring a miscible state. Molecule diffusion dominates mass transfer between CO<sub>2</sub> and oil due to the absence of convection during soaking in tight cores. The dissolution of CO<sub>2</sub> increases with soaking time because diffusion is time-dependent (Czarnota et al., 2018; Rezk and Foroozesh, 2019; Janiga et al., 2020). Increased CO<sub>2</sub> dissolution results in increased oil swelling, viscosity reduction and interfacial tension change, yielding rising oil recovery. The oil recovery reaches a peak after a 10-h soaking. This is consistent with experimental results of static tests acquired by Rezk and Foroozesh (2019), which showed that oil swelling, viscosity reduction and interfacial tension arrive at an equilibrium state after about 10-h interaction between CO<sub>2</sub> and oil. However, excess contacting time may give rise to asphaltene aggregation and precipitation, leading to decreased oil recovery.

## 4. Conclusions

We conducted CO<sub>2</sub> huff-n-puff experiments in natural conglomerate cores to examine its feasibility in enhancing oil recovery in tight conglomerate reservoirs. The miscibility index was proposed to characterize the miscible degree of CO<sub>2</sub> in oil. The similarity of miscibility index and Grashof number was used to obtain a lab-controllable pressure that was equivalent to the formation pressure. The equivalent pressure was calculated using the WinProp mode in CMG. Our results allow drawing the following conclusions.

- (1) The experimental pressure could be equivalently reduced to 23 MPa to ensure lab security and CO<sub>2</sub> flow similarity. The absence of light components in the crude oil used in the slim-tube test inferred that the MMP of CO<sub>2</sub> in formation oil was lower than the measured value of 28.5 MPa. Moreover, CO<sub>2</sub> MMP in the simulated oil was 12.025 MPa, indicating a miscible state during CO<sub>2</sub> huff and soaking experiments.
- (2) Oil recovery decreased with increased depletion degree. The lower the depletion degree, the higher the core pressure, and the greater the remaining oil. Therefore, CO<sub>2</sub> can more effectively interact with more oil, yielding more significant oil swelling and viscosity reduction and thus higher oil recovery.
- (3) Oil recovery first raised and then declined with increased CO<sub>2</sub> injection volume and soaking time. Despite the more effective interaction between CO<sub>2</sub> and remaining oil with increasingly dissolved CO<sub>2</sub> and longer soaking time, the recovery factor may be restricted by limited CO<sub>2</sub> dissolution and asphaltene precipitation, yielding an optimal value of CO<sub>2</sub> injection as well as soaking time.

## Acknowledgments

This study is financially supported by CNPC Innovation Foundation (2020D-5007-0214), Major Strategic Project of CNPC (ZLZX2020-01-04) and Beijing Municipal Excellent Talent Training Funds Youth Advanced Individual Project (2018000020124G163). The authors would like to acknowledge the full support from CMG-CUP Joint Numerical Reservoir Simulation Laboratory.

## References

- Abedini, A., Torabi, F., 2014. Oil recovery performance of immiscible and miscible CO<sub>2</sub> huff-and-puff processes. *Energy Fuel*. 28 (2), 774–784. <https://doi.org/10.1021/ef401363b>.
- Alfarge, D., Wei, M., Bai, B., 2018. Data analysis for CO<sub>2</sub>-EOR in shale-oil reservoirs based on a laboratory database. *J. Petrol. Sci. Eng.* 162, 697–711. <https://doi.org/10.1016/j.petrol.2017.10.087>.
- Bai, H., Zhang, Q., Li, Z., et al., 2019a. Effect of fracture on production characteristics and oil distribution during CO<sub>2</sub> huff-n-puff under tight and low-permeability conditions. *Fuel* 246, 117–125. <https://doi.org/10.1016/j.fuel.2019.02.107>.
- Bai, J., Liu, H., Wang, J., et al., 2019b. CO<sub>2</sub> water and N<sub>2</sub> injection for enhanced oil recovery with spatial arrangement of fractures in tight-oil reservoirs using huff-n-puff. *Energies* 12 (5). <https://doi.org/10.3390/en12050823>.
- Cao, M., Gu, Y., 2013. Oil recovery mechanisms and asphaltene precipitation phenomenon in immiscible and miscible CO<sub>2</sub> flooding processes. *Fuel* 109, 157–166. <https://doi.org/10.1016/j.fuel.2013.01.018>.
- Chen, Z., Su, Y., Li, L., et al., 2022. Characteristics and mechanisms of supercritical CO<sub>2</sub> flooding under different factors in low-permeability reservoirs. *Petrol. Sci.* 19 (3), 1174–1184. <https://doi.org/10.1016/j.petsci.2022.01.016>.
- Christensen, J.R., Stenby, E.H., Skauge, A., 2001. Review of WAG field experience. *SPE Reservoir Eval. Eng.* 4 (2), 97–106. <https://doi.org/10.2118/71203-PA>.
- Cronin, M., Emami-Meybodi, H., Johns, R.T., 2018. Diffusion-dominated proxy model for solvent injection in ultratight oil reservoirs. *SPE J.* 24 (2), 660–680. <https://doi.org/10.2118/190305-PA>.
- Czarnota, R., Janiga, D., Stopa, J., et al., 2017a. Determination of minimum miscibility pressure for CO<sub>2</sub> and oil system using acoustically monitored separator. *J. CO<sub>2</sub> Util.* 17, 32–36. <https://doi.org/10.1016/j.jcou.2016.11.004>.
- Czarnota, R., Janiga, D., Stopa, J., et al., 2017b. Minimum miscibility pressure measurement for CO<sub>2</sub> and oil using rapid pressure increase method. *J. CO<sub>2</sub> Util.* 21, 156–161.
- Czarnota, R., Janiga, D., Stopa, J., Wojnarowski, P., 2018. Acoustic investigation of CO<sub>2</sub> mass transfer into oil phase for vapor extraction process under reservoir conditions. *Int. J. Heat Mass Tran.* 127, 430–437. <https://doi.org/10.1016/j.ijheatmasstransfer.2018.06.098>.
- Ding, M., Wang, Y., Liu, D., et al., 2021. Enhancing tight oil recovery using CO<sub>2</sub> huff and puff injection: an experimental study of the influencing factors. *J. Nat. Gas Sci. Eng.* 90. <https://doi.org/10.1016/j.jngse.2021.103931>.
- Dong, M., Huang, S., Dyer, S.B., et al., 2001. A comparison of CO<sub>2</sub> minimum miscibility pressure determinations for Weyburn crude oil. *J. Petrol. Sci. Eng.* 65431 (1), 13–22. [https://doi.org/10.1016/S0920-4105\(01\)00135-8](https://doi.org/10.1016/S0920-4105(01)00135-8).
- Dong, X., Shen, L., Liu, X., et al., 2020. NMR characterization of a tight sand's pore structures and fluid mobility: an experimental investigation for CO<sub>2</sub> EOR potential. *Mar. Petrol. Geol.* 118. <https://doi.org/10.1016/j.marpetgeo.2020.104460>.
- Du, D., Pu, W., Jin, F., et al., 2020. Experimental study on EOR by CO<sub>2</sub> huff-n-puff and CO<sub>2</sub> flooding in tight conglomerate reservoirs with pore scale. *Chem. Eng. Res. Des.* 156, 425–432. <https://doi.org/10.1016/j.cherd.2020.02.018>.
- Elsharkawy, A.M., Poettmann, F.H., Christiansen, R.L., 1996. Measuring CO<sub>2</sub> minimum miscibility pressures: slim-tube or rising-bubble method? *Energy Fuel*. 10 (2), 443–449. <https://doi.org/10.1021/ef940212f>.
- Ferno, M.A., Hauge, L.P., Uno Rognmo, A., et al., 2015. Flow visualization of CO<sub>2</sub> in tight shale formations at reservoir conditions. *Geophys. Res. Lett.* 42 (18), 7414–7419. <https://doi.org/10.1002/2015gl065100>.
- Gao, H., Pu, W., 2021. Experimental study on supercritical CO<sub>2</sub> huff and puff in tight conglomerate reservoirs. *ACS Omega* 6 (38), 24545–24552. <https://doi.org/10.1021/acsomega.1c03130>.
- Hawthorne, S.B., Gorecki, C.D., Sorensen, J.A., et al., 2013. Hydrocarbon mobilization mechanisms from Upper, Middle, and Lower Bakken reservoir rocks exposed to CO<sub>2</sub>. *SPE Unconv. Resour. Conf. Canada*. <https://doi.org/10.2118/167200-MS>.
- Hawthorne, S.B., Grabanski, C.B., Miller, D.J., et al., 2019. Hydrocarbon recovery from Williston Basin shale and mudrock cores with supercritical CO<sub>2</sub>: part 2. Mechanisms that control oil recovery rates and CO<sub>2</sub> Permeation. *Energy Fuel*. 33 (8), 6867–6877. <https://doi.org/10.1021/acs.energyfuels.9b01180>.
- Hoteit, H., Firoozabadi, A., 2009. Numerical modeling of diffusion in fractured media for gas-injection and-recycling schemes. *SPE J.* 14 (2), 323–337. <https://doi.org/10.2118/103292-PA>.
- Hou, P., Ju, Y., Gao, F., et al., 2017. Simulation and visualization of the displacement between CO<sub>2</sub> and formation fluids at pore-scale levels and its application to the recovery of shale gas. *Int. J. Coal Sci. Technol.* 3 (4), 351–369. <https://doi.org/10.1007/s40789-016-0155-9>.
- Hu, X., Li, M., Peng, C., et al., 2020. Hybrid thermal-chemical enhanced oil recovery methods; an experimental study for tight reservoirs. *Symmetry* 12 (6). <https://doi.org/10.3390/s12060155>.

- doi.org/10.3390/sym12060947.
- Huq, F., Haderlein, S.B., Cirkpa, O.A., et al., 2015. Flow-through experiments on water–rock interactions in a sandstone caused by CO<sub>2</sub> injection at pressures and temperatures mimicking reservoir conditions. *Appl. Geochem.* 58, 136–146. <https://doi.org/10.1016/j.apgeochem.2015.04.006>.
- Janiga, D., Czarnota, R., Kuk, E., Stopa, J., Wójnarowski, P., 2020. Measurement of oil–CO<sub>2</sub> diffusion coefficient using pulse-echo method for pressure-volume decay approach under reservoir conditions. *J. Petrol. Sci. Eng.* 185, 106636. <https://doi.org/10.1016/j.petrol.2019.106636>.
- Janiga, D., Czarnota, R., Stopa, J., et al., 2018. Huff and puff process optimization in micro scale by coupling laboratory experiment and numerical simulation. *Fuel* 224, 289–301. <https://doi.org/10.1016/j.fuel.2018.03.085>.
- Jin, L., Hawthorne, S., Sorensen, J., et al., 2017. Advancing CO<sub>2</sub> enhanced oil recovery and storage in unconventional oil play—experimental studies on Bakken shales. *Appl. Energy* 208, 171–183. <https://doi.org/10.1016/j.apenergy.2017.10.054>.
- Li, D., Saraji, S., Jiao, Z., et al., 2021a. CO<sub>2</sub> injection strategies for enhanced oil recovery and geological sequestration in a tight reservoir: an experimental study. *Fuel* 284. <https://doi.org/10.1016/j.fuel.2020.119013>.
- Li, H., Yang, Z., Li, R., et al., 2021b. Mechanism of CO<sub>2</sub> enhanced oil recovery in shale reservoirs. *Petrol. Sci.* 18 (6), 1788–1796. <https://doi.org/10.1016/j.petsci.2021.09.040>.
- Li, Z., Gu, Y., 2014. Optimum timing for miscible CO<sub>2</sub>-EOR after waterflooding in a tight sandstone formation. *Energy Fuel*. 28 (1), 488–499. <https://doi.org/10.1021/ef402003r>.
- Liu, Y., Jiang, L., Song, Y., et al., 2016. Estimation of minimum miscibility pressure (MMP) of CO<sub>2</sub> and liquid n-alkane systems using an improved MRI technique. *Magn. Reson. Imag.* 34 (2), 97–104. <https://doi.org/10.1016/j.mri.2015.10.035>.
- Ma, J., Wang, X., Gao, R., et al., 2015. Enhanced light oil recovery from tight formations through CO<sub>2</sub> huff 'n'puff processes. *Fuel* 154, 35–44. <https://doi.org/10.1016/j.fuel.2015.03.029>.
- Ma, Q., Yang, S., Lv, D., et al., 2019. Experimental investigation on the influence factors and oil production distribution in different pore sizes during CO<sub>2</sub> huff-n-puff in an ultra-high-pressure tight oil reservoir. *J. Petrol. Sci. Eng.* 178, 1155–1163. <https://doi.org/10.1016/j.petrol.2019.04.012>.
- Pu, W., Du, D., Wang, S., et al., 2021. Experimental study of CO<sub>2</sub> huff-n-puff in a tight conglomerate reservoir using true triaxial stress cell core fracturing and displacement system: a case study. *J. Petrol. Sci. Eng.* 199. <https://doi.org/10.1016/j.petrol.2020.108298>.
- Pu, W., Wei, B., Jin, F., et al., 2016. Experimental investigation of CO<sub>2</sub> huff-n-puff process for enhancing oil recovery in tight reservoirs. *Chem. Eng. Res. Des.* 111, 269–276. <https://doi.org/10.1016/j.cherd.2016.05.012>.
- Qian, K., Yang, S., Dou, H., et al., 2018. Experimental investigation on microscopic residual oil distribution during CO<sub>2</sub> Huff-and-Puff process in tight oil reservoirs. *Energies* 11 (10), 2843. <https://doi.org/10.3390/en11102843>.
- Rassenfoss, S., 2017. Shale EOR works, but will it make a difference? *J. Petrol. Technol.* 69 (10), 34–40. <https://doi.org/10.2118/1017-0034-JPT>.
- Rezk, M.G., Foroozesh, J., 2019. Effect of CO<sub>2</sub> mass transfer on rate of oil properties changes: application to CO<sub>2</sub>-EOR projects. *J. Petrol. Sci. Eng.* 180, 298–309. <https://doi.org/10.1016/j.petrol.2019.05.053>.
- Russell, T., Pham, D., Neishaboor, M.T., et al., 2017. Effects of kaolinite in rocks on fines migration. *J. Nat. Gas Sci. Eng.* 45, 243–255. <https://doi.org/10.1016/j.jngse.2017.05.020>.
- Shen, Z., Sheng, J.J., 2018. Experimental and numerical study of permeability reduction caused by asphaltene precipitation and deposition during CO<sub>2</sub> huff and puff injection in Eagle Ford shale. *Fuel* 211, 432–445. <https://doi.org/10.1016/j.fuel.2017.09.047>.
- Sheng, J., 2015. Increase liquid oil production by huff-n-puff of produced gas in shale gas condensate reservoirs. *J. Unconvent. Oil and Gas Resour.* 11, 19–26. <https://doi.org/10.1016/j.juogr.2015.04.004>.
- Sheng, J., 2017. Critical review of field EOR projects in shale and tight reservoirs. *J. Petrol. Sci. Eng.* 159, 654–665. <https://doi.org/10.1016/j.petrol.2017.09.022>.
- Smith, M.M., Sholokhova, Y., Hao, Y., et al., 2013. Evaporite caprock integrity: an experimental study of reactive mineralogy and pore-scale heterogeneity during brine–CO<sub>2</sub> exposure. *Environ. Sci. Technol.* 47 (1), 262–268. <https://doi.org/10.1021/es3012723>.
- Sun, Y., Xin, Y., Lyu, F., et al., 2021. Experimental study on the mechanism of adsorption-improved imbibition in oil-wet tight sandstone by a nonionic surfactant for enhanced oil recovery. *Petrol. Sci.* 18 (4), 1115–1126. <https://doi.org/10.1016/j.petsci.2021.07.005>.
- Syah, R., Alizadeh, S.M., Nasution, M.K.M., et al., 2021. Carbon dioxide-based enhanced oil recovery methods to evaluate tight oil reservoirs productivity: a laboratory perspective coupled with geo-sequestration feature. *Energy Rep.* 7, 4697–4704. <https://doi.org/10.1016/j.egy.2021.07.043>.
- Syed, F.I., Dahaghi, A.K., Muther, T., 2022. Laboratory to field scale assessment for EOR applicability in tight oil reservoirs. *Petrol. Sci.* 19 (5). <https://doi.org/10.1016/j.petsci.2022.04.014>.
- Wang, H., Lun, Z., Lv, C., et al., 2017. Measurement and visualization of tight rock exposed to CO<sub>2</sub> using NMR relaxometry and MRI. *Sci. Rep.* 7, 44354. <https://doi.org/10.1038/srep44354>.
- Wang, Y., Shang, Q., Zhou, L., et al., 2021. Utilizing macroscopic areal permeability heterogeneity to enhance the effect of CO<sub>2</sub> flooding in tight sandstone reservoirs in the Ordos Basin. *J. Petrol. Sci. Eng.* 196. <https://doi.org/10.1016/j.petrol.2020.107633>.
- Wei, B., Gao, K., Song, T., et al., 2020a. Nuclear-magnetic-resonance monitoring of mass exchange in a low-permeability matrix/fracture model during CO<sub>2</sub> cyclic injection: a mechanistic study. *SPE J.* 25 (1), 440–450. <https://doi.org/10.2118/199345-PA>.
- Wei, B., Lu, L., Pu, W., et al., 2017. Production dynamics of CO<sub>2</sub> cyclic injection and CO<sub>2</sub> sequestration in tight porous media of Lucaogou formation in Jimsar sag. *J. Petrol. Sci. Eng.* 157, 1084–1094. <https://doi.org/10.1016/j.petrol.2017.08.023>.
- Wei, B., Zhang, X., Liu, J., et al., 2020b. Adsorptive behaviors of supercritical CO<sub>2</sub> in tight porous media and triggered chemical reactions with rock minerals during CO<sub>2</sub>-EOR and sequestration. *Chem. Eng. J.* 381. <https://doi.org/10.1016/j.cej.2019.122577>.
- Wei, B., Zhang, X., Wu, R., et al., 2019. Pore-scale monitoring of CO<sub>2</sub> and N<sub>2</sub> flooding processes in a tight formation under reservoir conditions using nuclear magnetic resonance (NMR): a case study. *Fuel* 246, 34–41. <https://doi.org/10.1016/j.fuel.2019.02.103>.
- Wigand, M., Carey, J.W., Schütt, H., et al., 2008. Geochemical effects of CO<sub>2</sub> sequestration in sandstones under simulated in situ conditions of deep saline aquifers. *Appl. Geochem.* 23 (9), 2735–2745. <https://doi.org/10.1016/j.apgeochem.2008.06.006>.
- Yu, H., Fu, W., Zhang, Y., et al., 2021. Experimental study on EOR performance of CO<sub>2</sub>-based flooding methods on tight oil. *Fuel* 290. <https://doi.org/10.1016/j.fuel.2020.119988>.
- Zhang, D., Ma, S., Zhang, J., et al., 2022. Field experiments of different fracturing designs in tight conglomerate oil reservoirs. *Sci. Rep.* 12 (1), 3220. <https://doi.org/10.1038/s41598-022-07162-y>.
- Zhang, K., Gu, Y., 2015. Two different technical criteria for determining the minimum miscibility pressures (MMPs) from the slim-tube and coreflood tests. *Fuel* 161, 146–156. <https://doi.org/10.1016/j.fuel.2015.08.039>.
- Zheng, J., Ju, Y., Zhao, X., 2014. Influence of pore structures on the mechanical behavior of low-permeability sandstones: numerical reconstruction and analysis. *Int. J. Coal Sci. Technol.* 1 (3), 329–337. <https://doi.org/10.1007/s40789-014-0020-7>.
- Zhou, D., Zhang, G., 2020. A review of mechanisms of induced fractures in SC-CO<sub>2</sub> fracturing. *Petrol. Sci. Bull.* 2, 239–253 (in Chinese).
- Zhou, X., Wang, Y., Zhang, L., et al., 2020. Evaluation of enhanced oil recovery potential using gas/water flooding in a tight oil reservoir. *Fuel* 272. <https://doi.org/10.1016/j.fuel.2020.117706>.
- Zhou, X., Yuan, Q., Zhang, Y., et al., 2019. Performance evaluation of CO<sub>2</sub> flooding process in tight oil reservoir via experimental and numerical simulation studies. *Fuel* 236, 730–746. <https://doi.org/10.1016/j.fuel.2018.09.035>.
- Zhu, C., Guo, W., Wang, Y., et al., 2020. Experimental study of enhanced oil recovery by CO<sub>2</sub> huff-n-puff in shales and tight sandstones with fractures. *Petrol. Sci.* 18, 852–869. <https://doi.org/10.1007/s12182-020-00538-7>.
- Zou, C., Yang, Z., Zhang, G., et al., 2014. Conventional and unconventional petroleum “orderly accumulation”: concept and practical significance. *Petrol. Explor. Dev.* 41 (1), 14–30. [https://doi.org/10.1016/s1876-3804\(14\)60002-1](https://doi.org/10.1016/s1876-3804(14)60002-1).
- Zou, Y., Li, S., Ma, X., et al., 2018. Effects of CO<sub>2</sub>-brine-rock interaction on porosity/permeability and mechanical properties during supercritical-CO<sub>2</sub> fracturing in shale reservoirs. *J. Nat. Gas Sci. Eng.* 49, 157–168. <https://doi.org/10.1016/j.jngse.2017.11.004>.
- Zuloaga, P., Yu, W., Miao, J., et al., 2017. Performance evaluation of CO<sub>2</sub> huff-n-puff and continuous CO<sub>2</sub> injection in tight oil reservoirs. *Energy* 134, 181–192. <https://doi.org/10.1016/j.energy.2017.06.028>.