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ORIGINAL ARTICLE

Laboratory study on oil recovery characteristics of carbonated water huff-n-puff process in tight cores under reservoir condition

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Abstract Although CO_2 huff-n-puff process has been widely employed as an efficient oil recovery and carbon storage technique in unconventional reservoirs, it suffers from the disadvantages of lack of availability near a large field and formation leakage due to the high CO_2 mobility. Carbonated water injection (CWI) has been extensively studied as the alternative of CO_2 injection process, but the laboratory studies concerning on CW huff-n-puff process is scarcely reported. In this paper, the oil recovery characteristics of CW huff-n-puff process has been scrutinized under reservoir conditions of 28 MPa and 65 °C together with NMR tests to reveal the oil mobilization rules in the tight cores. Comparative studies on CO_2 huff-n-puff and water huff-n-puff process were also performed. It is found 4 rounds of CW huff-n-puff processes could recover considerable amount of 36.2% of the oil from the tight core. Although the recovery rate is lower than 55.4% for CO_2 process but it is much higher than 14.4% of the water process. NMR T2 results reveal the oil in the large size pores of the tight cores are mobilized and recovered in early huff-n-puff cycles, whereas the oil from small size pores are collected in the later stage of the CW and CO_2 huff-n-puff processes. The research results indicate the CW huff-n-puff process is promising for tight reservoir development.

1. Introduction

Under the contradiction of the fast depletion of oil production from conventional reservoirs and the increasing energy demand, unconventional oil and gas reservoirs, such as tight oil, tight gas and shale gas, have played more and more

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important role in the world's energy supply (Wang et al., 2014; Song et al., 2017). In China, tight reservoir is defined as the oil reservoir with permeability lower than or equal to 0.1mD, apart from the ultra low permeability of 0.1–1 mD, extra low permeability of 1–10 mD and ordinary low permeability of 10–50 mD (Rao et al., 2019; Peng et al., 2019). The economic development of tight oil reservoirs, however, presents a great challenge due to extremely low formation permeability. It is unfavorable to apply water flooding in tight oil reservoir due to low injectivity and poor sweep efficiency, and the horizontal drilling and multi-stage hydraulic fracturing technology also suffers from the steep decline curves of primary production (Zuloaga-molero et al., 2016).

CO_2 has a considerably lower minimum miscibility pressure (MMP) than other gases such as N_2 and CH_4. In a miscible flooding process, the oil swelling and light-hydrocarbon extraction effect could mobilize and produce of the oil in tight reservoirs. (Stalkup, 1983; Martin and Taber, 1992; Abedini et al., 2015; Li and Gu, 2014; Yu et al., 2020; Zheng et al., 2019) Therefore CO_2-based techniques have been well developed as a feasible solution to enhance oil recovery as well as fulfill carbon storage and sequestration in tight oil reservoirs (He et al., 2015; Welkenhuysen et al., 2017; Samara et al., 2019; Zhang et al., 2020; Wu et al., 2019; Myshakin et al., 2019; Zhou et al., 2019; Wu et al., 2020). Among all the CO_2 based unconventional oil production techniques, cyclic CO_2 stimulation, or "CO_2 huff and puff injection', has been effectively applied to improve oil recovery from tight formations. With three stages of huff (injection), soaking, and puff (production), CO_2 huff-n-puff process could assure sufficient contact between CO_2 and crude oil in soaking stage and the oil with improved properties (e.g., viscosity reduction and swelling) will be expelled from tight cores in puff stage by swelling and solution gas drive (Song and Yang, 2013; Ma et al., 2015; Todd and Evans, 2016; Sheng, 2017). Although the huff-n-puff process employing high concentration CO_2 has been proven efficient to recover extra oil from tight formations based on dozens of laboratory works (Rao et al., 2019; Ma et al., 2019; Bai et al., 2019; Zuloaga et al., 2017; Li et al., 2019; Pu et al., 2016), this technique suffers from the following two disadvantages: usually lack of availability near a large field operation and failed CO_2 sequestration because of gas leakage due to the high CO_2 mobility (Sheng, 2017; Shakiba et al., 2016).

An alternative method that alleviates these problems is to substitute pure CO_2 with Carbonated Water (CW). CW is a single phase CO_2-enriched water at the reservoir conditions and are commonly used with the favorable mechanisms such as reducing the IFT, altering the reservoir's rocks wettability, reducing viscosity and oil swelling through the transfer of carbon dioxide from CW to adjacent oil, and increasing the gas viscosity to improve the sweep efficiency (Khaksar et al., 2017; Nowrouzi et al., 2019; Riazi and Golkari, 2016; Esene et al., 2019). Compared to the technique employing pure CO_2, the CW method consumes much less CO_2 therefore is more suitable for the oil reservoirs far from major CO_2 resources. In addition, EOR process employing CW could be a more secured method to store CO_2 in the underground formations as CO_2 is more stably dissolved in water rather than in oil (Sohrabi et al., 2011; Sohrabi et al., 2012).

Extensive research works have been carried out at laboratory scales concerning the oil recovery characteristics with Carbonated Water Injection (CWI) process. Shu et al. (Shu et al., 2014) experimentally investigated a pre-flushing method of the reservoir using Active Carbonated Water (ACW) before a CO_2 flood and they found the oil recovery increased by 35.5%, compared to 16.6% from injecting CO_2 alone. Shakiba et al. (Shakiba et al., 2016) investigated the oil recovery during secondary and tertiary CW injection in a low permeable carbonate oil reservoir and showed 40.54% and 56.74% more oil recovery during tertiary CWI (TCWI) and secondary CWI (SCWI) compared to the corresponding water flooding, respectively. Bakhshi et al. (Bakhshi et al., 2017) evaluated the CW flooding process as a secondary and a tertiary recovery technique based on core experiments and reported higher oil recovery rate for CW flooding as compared to water flooding results. Honarvar et al. (Honarvar et al., 2017) investigated the CWI potential on an Iranian carbonate reservoir and showed that oil recovery increased with CWI as compared to conventional water flooding. Seyyedi et al. (Seyyedi et al., 2018) comparatively studied the oil compositional variations during CO_2 and carbonated water injection scenarios and revealed the remaining oil after CO_2 flooding was heavier than the original oil and its production was even more difficult. While during CWI the resident oil became lighter (lower viscosity) and no evidence of substantial extraction or oil downgrading was observed. Seyyedi et al. (Seyyedi et al., 2018) carried out a series of high-pressure and high-temperature coreflood experiments with a 98.7 mD heterogeneous sandstone core. They found the ultimate oil recovery by CWI, either as the secondary or tertiary injection scenario, was higher than that of conventional water flooding. Mahzari et al. (Mahzari et al., 2018) investigated CWI process under reservoir conditions and observed based on the core displacement tests that secondary CWI could recover additional 26% oil compared to conventional seawater injection. Jia (Jia, 2019) reported the encouraging results of CWI process in terms of the improved oil recovery and carbon storage based on the micromodel results. They found a secondary water flooding after the CWI could decrease the residual oil recovery factor from 67% to 32.7%.

It could be deduced from above literature reviews that up to now, most laboratory works focus on CW injection process in low permeable or conventional porous medium, the investigation on CW huff-n-puff process in tight cores is scarcely reported. As an effective alternative of CO_2, the CW huff-n-puff process could also be potentially applied in tight oil recovery practices. To shed the light on applying CW huff-n-puff technique in tight formations, we experimentally studied the oil recovery characteristics of the CW huff-n-puff process in a tight core. In addition, Nuclear Magnetic Resonance (NMR) measurements, which has been widely employed for the determination of pore distribution and fluid saturation in porous media (Vinegar, 1986; Chen et al., 1993; Qun et al., 2019; Liu et al., 2020); were conducted to reveal the oil production rules from different size pores in the tight cores. To show clearly the tight oil recovery efficiency of the CW huff-n-puff process, comparative studies were also carried out on CO_2 huff-n-puff and water huff-n-puff processes.

2. Experimental section

2.1. Materials

Three 5 cm diameter tight cores retrieved from the oil field, as shown in Fig. 1 and numbered as 1, 2, 3 with length of 4.8–5.0 cm, were employed in this study. The physical properties of the three tight cores are summarized in Table 1, showing the core porosity and permeability locating in the range 13.9–15.1% and 0.0085–0.034 mD respectively.

Core mineralogy composition is listed in Table 2, showing the main mineral components of quartz (mass fraction 33%), plagioclase (mass fraction 42%), and potassium feldspar (mass fraction 18%), together with minor concentration of ferrodolomite (mass fraction 3%), clay minerals (mass fraction 3%) and calcite (mass fraction 1%).

Simulated oil with the density of 0.80 g/cm^3 is employed in this study. The oil viscosity-temperature relationship and the Minimum Miscibility Pressure (MMP) of the oil / CO_2 system were measured and depicted in Fig. 2(a) and Fig. 2(b) respectively. It is clearly observed from Fig. 2 (a) that the oil viscosity decreases with temperature from 5.2 mPas at 30 °C to 2.0 mP′s at 80 °C. As the important criterion to judge a miscible or immiscible system and to distinguish oil recovery mechanism in CO_2 injection processes (Du et al., 2019); MMP of oil/CO_2 system is determined with the Vanish Interfacial Tension (VIT) method (Rao, 1997) through measuring the interfacial tension (IFT) between the pendant oil drop and the surrounding CO_2 fluid in a high temperature high pressure view cell. Fig. 2(b) shows the IFT values under different system pressures at 65 °C in accordance with the reservoir temperature. It is clearly observed the IFT decreases linearly with the increasing pressure and approaches to 0 under the system pressure of 13.2 MPa, indicating the MMP for the oil/CO_2 system is 13.2 MPa.

2.2. Carbonated water

In this study, three huff-n-puff processes respectively employing CO_2, brine and carbonated water are comparatively studied. Supercritical CO_2 with purity of 99.9% is employed in the CO_2 huff-n-puff process. While the brine with salinity of 1.4 wt%, prepared by dissolving 14 g analytical NaCl in 986 g deionized water, is employed in the water huff-n-puff processes.

The carbonated water employed in the CW huff-n-puff process is prepared by continuous injection CO_2 in the 1.4 wt% salinity brine under constant pressure of 20 MPa and temperature of 45 °C. At first, pour 950 mL brine in the 1L stainless steel container under atmospheric condition, then seal the container and put it in an incubator of 45 °C. Pressurize the container through CO_2 injection until the internal pressure approaches 20 MPa. Maintain the pressure of 20 MPa through continuous CO_2 injection 72 h to achieve the phase equilibrium between CO_2 and brine. Based on the thermodynamic data of CO_2 solubility in brine (Zhao et al., 2015), it is assured the prepared CO_2-enriched solution is a stable one phase fluid and has the same amount of dissolved CO_2 as under the reservoir condition of 28 MPa & 65 °C.

2.3. Experimental apparatus

Fig. 3 depicts the setup components schematically for performing the high pressure and high temperature huff-n-puff processes.

As shown in the figure, the oil and three injection fluids of carbonated water, brine and liquid CO_2, are stored in four stainless steel containers marked of No.2, 3, 4 and 8, respectively. The stainless core holder (No.17), which could stand pressure up to 70 MPa and temperature up to 150 °C, is put in an incubator (No. 25) with upper limit of 150 °C. Together with the backpressure regulation system (No. 22–24) and confining pressure pump No.18, the core could maintain the reservoir pressure and temperature in the core holder without any side leakage. With the opened valve No.15, No.19 and the closed valve No. 16, oil is pushed through the tight core with high precision syringe pump to fulfill the oil saturation process. With opened valve No.15 and closed valve No. 16 and No.19, the fluid injection (huff) and soaking process is performed. Then with opened valve No.16 and closed valve No. 15 and No.19, the production (puff) process is performed and the produced oil is collected and measured with the effluent collection device (No.21).

Fig. 4 schematically illustrate the tight core when loaded in the core holder. Two tubes, one is for fluid injection and the other for the puff liquid extraction, are fabricated in the

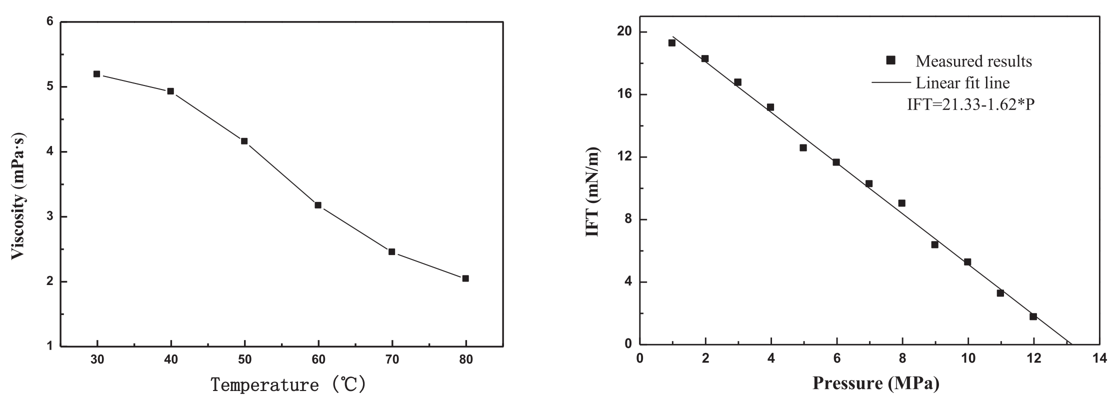
Fig. 1 Tight core samples employed in the study.

Table 1 Physical properties of the tight core samples.

Core No.	Length/cm	Diameter/cm	Permeability/mD	Porosity/%
1	5.0	5.0	0.009	14.0
2	5.0	5.0	0.022	13.9
3	4.8	5.0	0.034	15.1

Table 2 Core mineralogy compositions (wt%).

Quartz	Potassium feldspar	Plagioclase	calcite	Ferrodolomite	clay mineral
33	18	42	1	3	3



(a) **viscosity-temperature relationship** (b) **MMP determined on VIT method**

Fig. 2 Properties of oil (a) viscosity and (b) miscibility with CO_2.

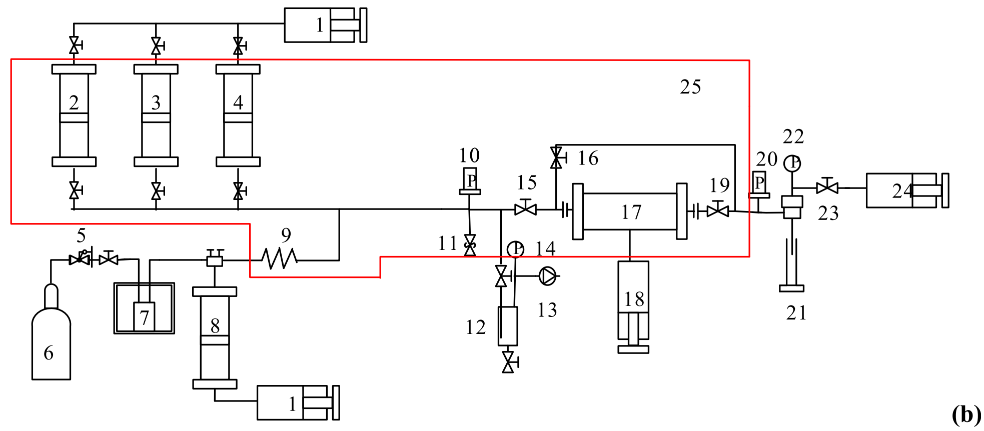
sealing bolt with fluid distributor at the front surface touching the core. The core is sealed tightly with the rubber jacket with the confine pressure water. With other side of the core blocked with stainless steel blocker, the injection fluid in the huff process is introduced into the tight core through the injection tube. Whereas the extracted fluid, such as produced oil together with water or CO_2 in the puff process, is guided out of the core through the outflow tube.

After each cycle of huff-n-puff process, the tight core sample will be taken out of the core holder for NMR measurement (NMR model: MesoMR23-060H-I, Suzhou Niumag Analytical Instrument Co. China). The T2 spectrum obtained by NMR reflects the spatial distribution of ^{1}H-proton-bearing fluid in the rock sample with longer the relaxation time standing for larger pore diameter (Qun et al., 2019; Liu et al., 2020). Therefore, the NMR T2 results could reveal the oil mobilization rules in different size pores of the tight media after the huff-h-puff process.

2.4. Experimental procedure

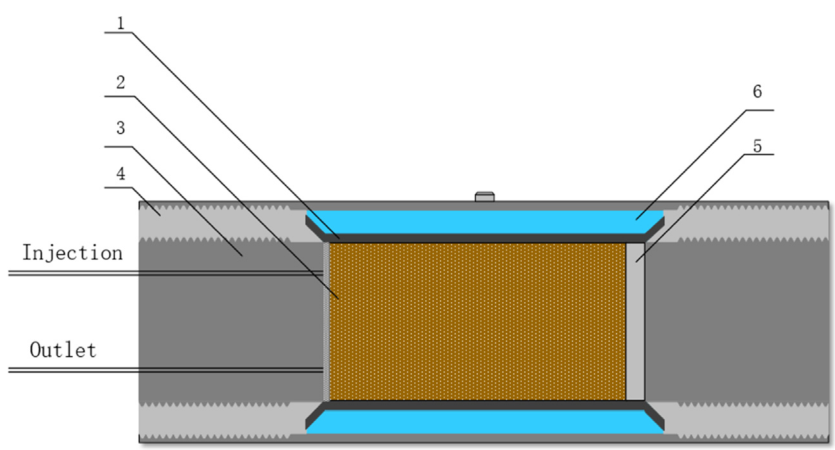
The experimental procedure for CW huff-n-puff process is listed as follows,

(1) Dry the core for 72 h, weigh and measure the T2 spectrum of the dry core with NMR equipment.
(2) Put the tight core in the core holder and vacuum the core for 24 h.
(3) Flood the core with oil at elevated temperature of 65 °C and elevated backpressure around 10 MPa. When the oil makes breakthrough from the tight core, the oil flood rate is set to 0.05 mL/min. Record the pressure drop between inlet and outlet of core to obtain the oil phase permeability.
(4) Keep oil injection for another 12 h with closed outlet and bypass valve at fixed system pressure of 20 MPa and temperature of 65 °C to fulfill satisfactory oil saturation of the core.
(5) Release the system pressure at the depletion speed of 0.5 MPa/min and take out the oil saturated core from the core holder. Weigh and measure the T2 spectrum of the oil saturated core with NMR equipment. It has to be mentioned setting up the on-line NMR equipment for such a high pressure and temperature system is too complicated to be easily fulfilled, therefore the off-line NMR measurement for the tight cores are performed on not only the oil saturated cores but the core samples after each cycle of huff-n-puff process.



1. High precision syringe pump; 2.Container (oil); 3.Container (CW); 4.Container (brine); 5. Pressure relief valve; 6.CO_2 cylinders; 7. Refrigeration unit; 8. Container (liquid CO_2); 9.Preheating coil; 10.inlet pressure transducer; 11.Relief valve; 12.Buffer tank; 13.Vacuum pump; 14.Vacuum gauge; 15.Inlet valve 16.Bypass tube valve; 17.Core holder; 18. Confining pressure pump; 19.Outlet valve; 20.Outlet pressure transducer; 21.Effluent collection device; 22.back pressure gauge; 23.back pressure valve; 24.Back pressure of pump; 25. Incubator

Fig. 3 Schematic diagram of the experimental setup.



1. Rubber jacket; 2. Tight core; 3. Sealing bolt of the core (with fluid distributor at the front surface and fabricated injection and outlet tubes); 4. Sealing bolt of the rubber jacket; 5. Blocker; 6; Confine pressure water

Fig. 4 Schematic illustration of the core when loaded in the core holder.

(6) Perform the 1st cycle huff-n-puff procedure. Put back the oil-saturated core in the core holder, then keep continuous injection of CW to elevate the system pressure to 28 MPa and temperature to 65 °C in accordance with the designated reservoir condition. Maintain the system condition for 24 h and then release the system pressure at the depletion speed of 0.5 MPa/min. Measure the collected oil volume and measure the T2 spectrum of the core with NMR equipment.

(7) In the same procedure, perform totally four cycles of CW huff-n-puff process and record the oil recovery rate and measure the NMR T2 spectrum of the core after each cycle of the process.

As the comparative studies, CO_2 huff-n-puff process and water huff-n-puff process are also carried out. Fig. 5 depicts the experimental schemes and corresponding measurements for all the three huff-n-puff procedures. Besides oil recovery

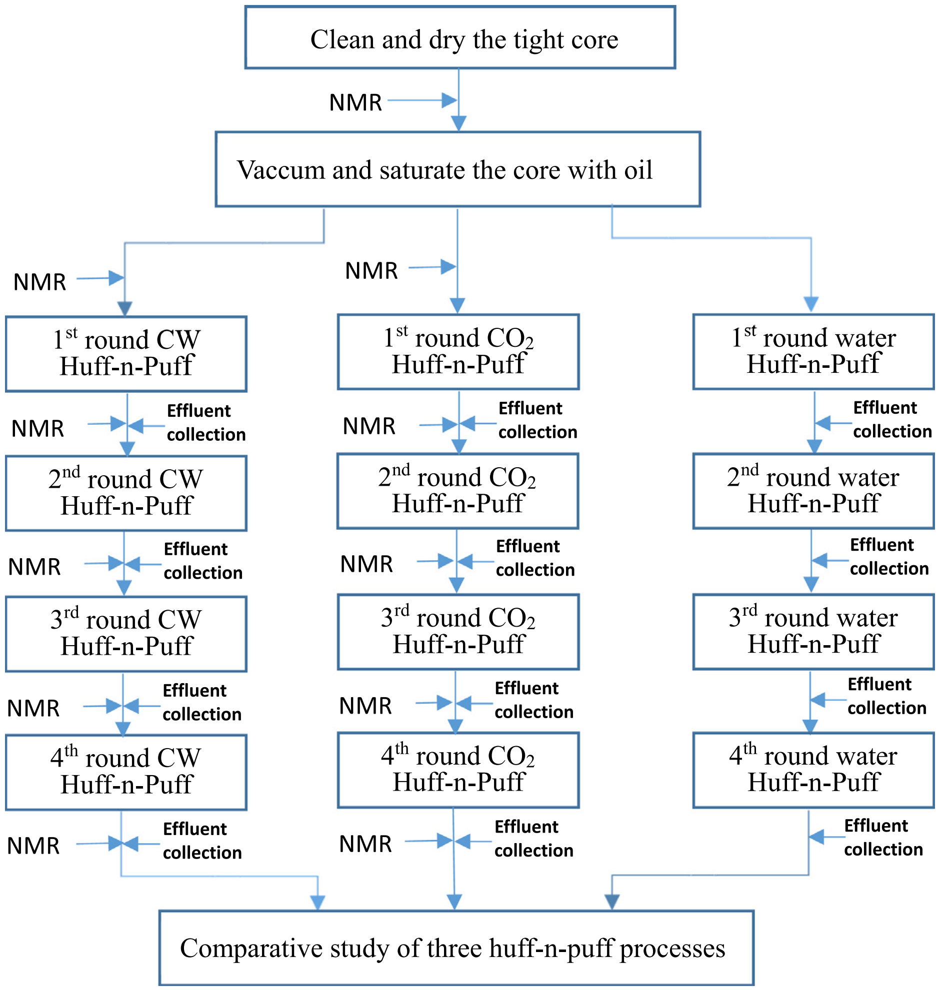


Fig. 5 Flow chart of the experimental works on CW, CO_2 and water huff-n-puff processes.

results, NMR measurements are also carried out for CO_2 huff-n-puff process to understand the EOR mechanisms of the CW and CO_2 huff-n-puff process. As to the water huff-n-puff processes, as it is revealed later, the oil recovery rate is negligible small, therefore NMR test is not performed.

3. Results and discussions

3.1. Oil recovery characteristics

3.1.1. CW Huff-n-puff process

Based on the above mentioned procedure, CW huff-n-puff processes are carried out and the oil recovery characteristics were obtained based on effluent oil collection results.

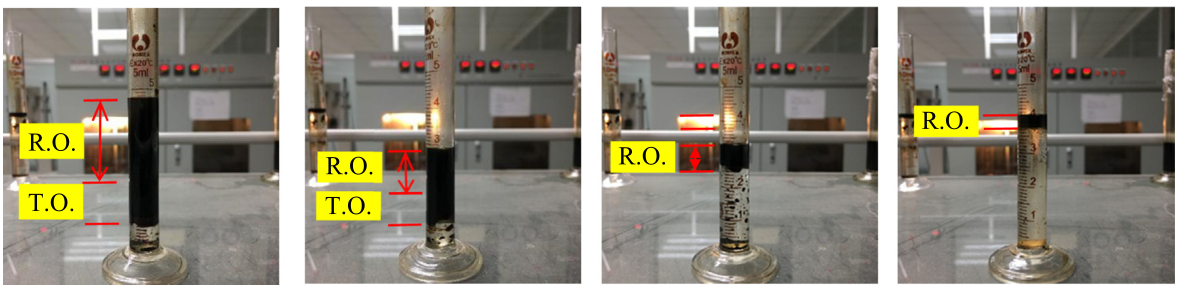
Fig. 6 (a), (b) and (c) show the images of the oil saturated tight core, the core after 1st CW huff-n-puff and the core after 2nd CW Huff-n-puff processes respectively. Just after retrieved from the core holder after pressure depeletion procedure, as shown in Fig. 6 (b), CO_2 bubbles releasing from the inlet of the core could be clearly observed, indicating the CO_2 dissolved in CW may substantially contribute to tight oil recovery. In addition, the horizontally laid core image in Fig. 6 (b) shows clearly that after the 1st round of huff-n-puff process, the CO_2–enriched solution could extract distinctive amount of the liquid content from inlet part of the core, which leaves dry portion in the front part of the core. After the 2nd round operation, as is revealed in Fig. 6(c), the dry part in the core inlet region becomes larger, indicating further oil could be recovered from the tight core with the CW huff-n-puff process.

Fig. 7 (a)-(d) shows the quantitative amount of the collected effluent oil after each round of the CW huff-n-puff processes. The tube oil volume has to be deducted before obtaining the exact amount of recovered oil from the tight core. Tube oil is the oil left in the tubing system in the process of the experiment. As we used the same tubing system for oil saturation process, it is inevitable that some oil stay in the system, even we have made the effort to clean the whole tubing system before the huff-n-puff process. The process of determining the tubing oil is described in the following section of 3.1.2 for the supercritical CO_2 huff-n-puff processes. As indicated in Fig. 7(a) and (b), the Tube Oil (T.O.) volume after 1st and 2nd round huff-n-puff process is 1.5 mL and 1.0 mL respectively, and they are reasonably deducted in the collected volume of oil to get the final Recovered Oil (R.O.) results.

Table 3 lists the quantitative results of the recovered oil volume together with the oil recover rate based on the total saturated oil volume of 13.06 mL in the tight core No.2. It is found

(a) **(b)** **(c)**

Fig. 6 Core images after the (a) oil saturation; (b) 1st and (b) 2nd round CW huff-n-puff processes.



(a) (b) (c) (d)

Fig. 7 Effluent oil collection results after (a) 1st (b) 2nd (c) 3rd and (d) 4th CW huff-n-puff processes (R.O. stands for Recovered Oil, T.O. stand for Tube Oil).

Table 3 Oil recovery rate of after each cycle of CW huff-n-puff process in tight core No. 2.

	Saturated oil in the core	Recovered oil after 1st round	Recovered oil after 2nd round	Recovered oil after 3rd round	Recovered oil after 4th round
Volume (mL)	13.06	2.5	1.25	0.75	0.25
Recovery rate		19.1%	9.6%	5.7%	1.8%

the 1st round could produce as high as 19.1% of oil from the core, and the following 3 rounds could recovery 9.6%, 5.7% and 1.8% respectively, which gives total recovery rate of 36.2%, indicating the CW huff-n-puff process could be potentially employed on tight oil recovering practices. In addition, it is deduced that three rounds of operation is enough to obtain good performance, which is also instructive on guidance of the successful field application of the CW huff-n-puff technique.

3.1.2. CO_2 huff-n-puff process

As the comparative study, supercritical CO_2 huff-n-puff process has also been performed. Fig. 8(a) and (b) displays clearly the tight sample images after the 1st and 2nd round of CO_2 huff-n-puff operations. It is clearly observed the inlet part of the sample gets drier after each cycle of operation, indicating increasing round of CO_2 injection could recover oil mainly from the inlet part of the tight core. With comparison with the cores after CW operation shown in Fig. 6(b) and (c), it is also found the dry part of the core is obviously larger after CO_2 huff-n-puff process, indicating CO_2 could recover more oil content from the tight core after each round of huff-n-puff process.

In contrast with CW huff-n-puff process where the sample core weighing method is not applicable due to the coexistence of brine, CO_2 and oil, the oil recovery characteristics after each CO_2 huff-n-puff process could be exactly obtained from the core weighing method. Table 4 lists the recovered oil weight based on the core weight loss after each operation stage, from which the oil recovery rate of 1st, 2nd, 3rd and 4th round is determined to be 33.4%, 11.9%, 7.8% and 2.3%, respectively.

Also listed in Table 4 is the recovered oil volume calculated the oil weight data of each cycle of the operation, which is employed to calibrate the effluent oil collection results. Due to tube oil existence, the oil collected after each cycle of huff-n-puff process inevitably contains some amount of oil in the tube system. Through comparison of the recovered oil volume listed in table 3 and those in Fig. 9 where the amount of the collected effluent oil are clearly shown, it is concluded at last the tube oil after 1st round is 1.5 mL and the 2nd round tubing oil is 1.0 mL. The tube oil after 3rd and 4th round is deemed to be negligible due to the satisfactory fit of the oil volume obtained from weight loss and the effluent oil collection. The tube oil amount of 1.5 mL applied to all the three huff-n-puff processes at the 1st stage, while tube oil of 1.0 mL

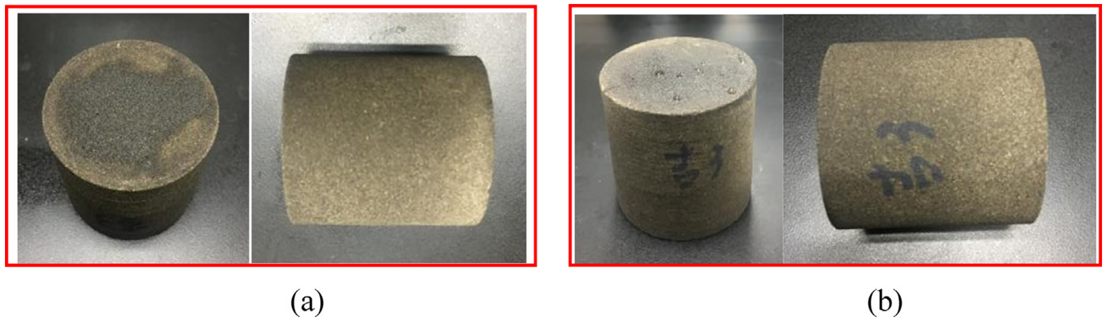


Fig. 8 Core images after the (a) 1st and (b) 2nd round CO_2 huff-n-puff processes.

Table 4 Oil recovery rate of after each cycle of CO huff-n-puff process in tight core No. 1.

	Saturated oil in the core	Recovered oil after 1st round	Recovered oil after 2nd round	Recovered oil after 3rd round	Recovered oil after 4th round
Weight (g)	10.59	3.54	1.26	0.83	0.24
Calibrated Vol. (mL)	13.2	4.4	1.6	1.0	0.3
Recovery rate		33.4%	11.9%	7.8%	2.3%

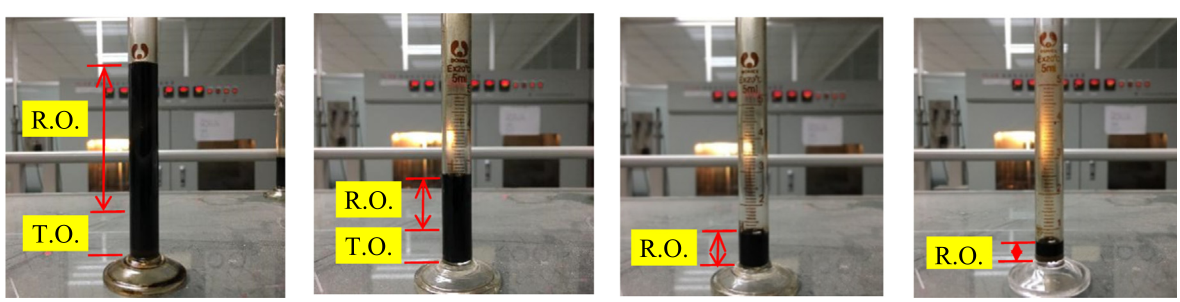


Fig. 9 Effluent oil collection results after (a) 1st (b) 2nd (c) 3rd and (d) 4th CO_2 huff-n-puff processes (T.O. are calibrated for 1st and 2nd round based on oil weight results).

applied to CO_2-related injection medium only due to the extra CO_2 extraction effect.

3.1.3. Water huff-n-puff process

As another comparative study, brine huff-n-puff process is also performed. Fig. 10 displays the collected effluent oil and Table 5 lists the quantity of the oil volume collected after each cycle of the process. The experimental results show much less oil recovered after each cycle of the water huff-n-puff operation as compared to the corresponding CW and CO_2 processes.

3.1.4. Comparison of oil recovery effects

Fig. 11 depicts clearly the total recovery rates for three huff-n-puff processes based on each cycle oil recovery rates. It is found the CO_2 huff-n-puff process has the best capability on tight oil recovery, with the first found recovery rate of 33.4% and the final oil recovery rate of 55.4% from the tight core. Although the CW huff-n-puff process recovers less oil based on cycle and total recovery rate results compared to CO_2 process, its tight oil recovery capacity is still impressing with first round of 19.1% and total recovery rate of 36.4% of the oil in the 0.02mD tight core. The water huff-n-puff process, however, shows the most unfavorable behavior in this study as the operation only takes effect in the first round and the final recovery rate is less than 15%.

3.2. NMR T2 spectrum results

3.2.1. CW huff-n-puff process

To reveal the oil mobilization rules of the CW huff-n-puff process in tight cores, NMR measurements are carried out and the T2 spectrum results on dry core, oil saturated core, the core after 1st, 2nd, 3rd and 4th round operations are summarily depicted in Fig. 12. Since the pore size of reservoir rock is proportional to the relaxation time in NMR T2 spectrum with the longer time corresponding to the larger pore radius, NMR T2 spectra of different cycles could be employed for comparative analysis of the contribution of different size pores on oil recovery.

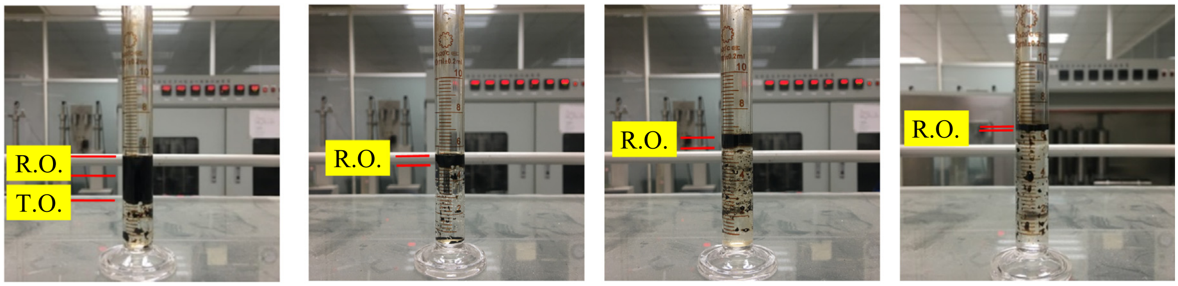


Fig. 10 Effluent oil collection results after (a) 1st (b) 2nd (c) 3rd and (d) 4th round of the water huff-n-puff process.

Table 5 Oil recovery rate of after each cycle of water huff-n-puff process in tight core No. 3.

	Saturated oil in the core	Recovered oil after 1st round	Recovered oil after 2nd round	Recovered oil after 3rd round	Recovered oil after 4th round
Volume (mL)	13.85	1.0	0.4	0.4	0.2
Recovery rate		7.2%	2.9%	2.9%	1.4%

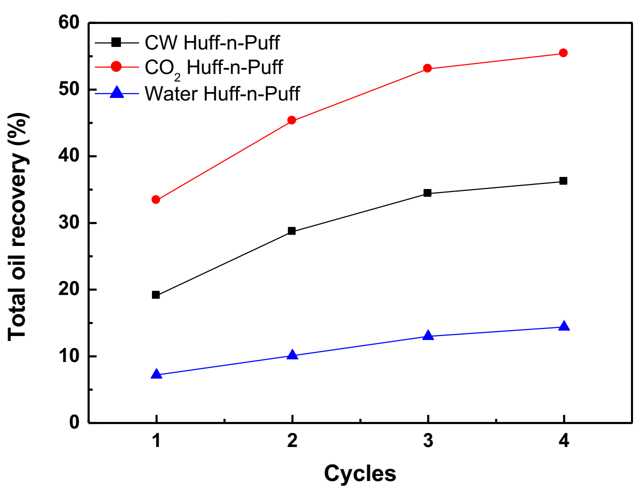


Fig. 11 Comparison of oil recovery effects of the three huff-n-puff processes.

It is clearly observed from Fig. 12 that the T2 spectrum for oil saturated core shows the highest signal strength among all the six curves and mainly exists in the range of 3–300 ms, with two peaks in the range of 3–25 ms and 25–300 ms respectively, indicating there are two distinct pore size ranges in the tight porous medium. According to Ma et al. (Ma and Zhang, 2017), who employed conventional mercury injection and constant velocity mercury injection method on the pore structure analysis of tight samples in the same reservoir region, the size of small pores corresponding to the lower T2 spectrum ranges in 0.05–0.4 μm with the median radius of 0.2 μm, whereas the large pores corresponding to the higher T2 spectrum locate in the size range of 0.4–3.0 μm with the median radius of is 0.8 μm. The strength of both signal peaks becomes significantly lower after the 1st CW huff-n-puff processes. Compared to the decreasing percentage in the lower T2 time range, the signal decrement is more significant in the higher T2 time range, indicating most part of the recovered oil come from large size pores in the 1st huff-n-puff operation. The T2 spectrums measured after the second, third and fourth round

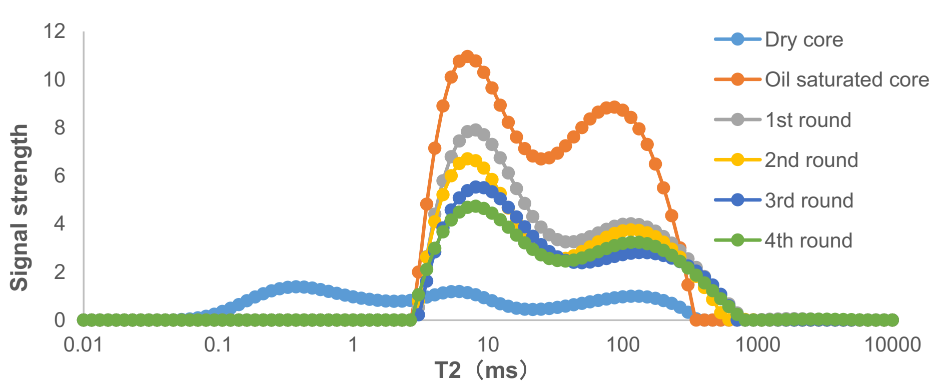


Fig. 12 NMR T2 spectrum results in various stages of the CW huff-n-puff process.

huff-n-puff process, on the other hand, show gradually smaller variations corresponding to the decreasing oil recovery rate with the increasing cycles. It is also found from Fig. 12 that spectral peak in lower T2 time range decreases more obviously along with the 2nd to 4th cycles, indicating the oil in small pores could also be partially mobilized through the CW huff-n-puff process.

3.2.2. CO_2 huff-n-puff process

Fig. 13 depicts comparatively the T2 spectrum of the dry core, the oil saturated core, the core after 1st, 2nd, 3rd and 4th round of CO_2 huff-n-puff process.

It is clearly observed from Fig. 13 that the oil saturated core has two peaks in the range of 2–25 ms and 25–300 ms, which is similar to the sample employed for CW huff-n-puff processes in Fig. 12. It is concluded, therefore, the pore size of the samples employed for various processes is similar to each other and the obtained results could be reasonably employed for comparative studies on oil recovery properties. After the 1st round of CO_2 huff-n-puff procedure, it is observed the peak at 87 ms in higher T2 time region significantly decreases in comparison with peak variation at 6 ms in lower T2 region, indicating the 1st cycle of CO_2 huff-n-puff mainly recovers the oil in larger size pores of the tight core while leaves most oil in smaller pores intact. After 2nd round of operation, the peak values at 6 ms and 87 ms both decreases remarkably, showing the oil in either small or large pores is mobilized in this stage of operation. In addition, it is found the peak diminishes at 87 ms, revealing most oil in larger pores is extracted with the two cycles of huff-n-puff processes. The T2 curves for the 3rd round of processes show more obvious peak decrement at T2 = 6 ms rather than in higher T2 time ranges, indicating the oil recovery at this cycle mainly comes from the small pores of the tight core. Based on the duplicate T2 curves for 3rd and 4th cycles, however, it is deduced no distinct amount of oil could be recovery from the tight core with further increasing cycle of operation, which is in consistent with the oil recovery results listed in Table 3.

3.3. Discussions

As revealed in Section 3.1, the CO_2 huff-n-puff process has higher oil recovery efficiency of 55.4% compared to the 36.2% of the CW huff-n-puff processes, which could be contributed to the miscibility of CO_2/oil system for CO_2 huff-n-puff processes under the laboratory conditions. With the increasing system pressure, the oil–gas two-phase region enlarges and the saturation pressure increases as more CO_2 is dissolved in the formation oil, and the resulting oil swelling and viscosity reduction effect is beneficial to oil production. With further pressure elevation to the miscible conditions, the diffusion process between CO_2 and oil phase could dramatically increase and thus lead to higher oil recovery rate. Du et al. (Du et al., 2019) measured the diffusion coefficient of a miscible CO_2/n-hexadecane system with Dynamic Pendant Drop Volume Analysis (DPDVA) technique and reported the diffusion coefficient could be 10 times higher under miscible conditions in comparison with under immiscible conditions. Li et al. (Li et al., 2017) conducted 15 CO_2 huff-n-puff experiments in shale cores at pressures below and above the MMP and they found the injection pressure had a significant effect on enhancing oil recovery. When the injection pressure is elevated from below MMP to above MMP, the oil recovery rate increases accordingly until 200 psi higher than the MMP. In their further studies, Li et al. (Li et al., 2019) investigated the CO_2 huff-n-puff processes under immiscible (8 MPa), near miscible (13 MPa) and miscible (20 MPa) conditions and reported remarkably different recovery rate of 25.9%, 47.1%, and 55.4%, respectively after six huff-n-puff cycles.

On the other hand, in this study, the main EOR mechanisms of carbonated water huff-n-puff could contribute to the oil expansion and oil viscosity reduction due to the gradual CO_2 transfer from water phase to the oil phase. When CO_2 enters the oil phase from the water phase, the volume of oil increases, which makes the elastic driving energy of oil increase and the oil saturation increase. In addition, the expanded oil can also connect with isolated droplets in some of the dead pores, allowing more oil to flow. However, due to the lower mass concentration difference of CO_2 in the water phase and oil phase, the diffusion rate of CO_2 from water phase to the oil phase is limited compared to the miscible CO_2/oil system, which results to the lower recovery efficiency of CW huff-n-puff process. It has to be mentioned though, besides the advantages of much less CO_2 consumption and less CO_2 leakage risk from the reservoir formations, there are also some unique advantages of employing CW as the injection fluid in comparison with pure CO_2, such as the water lock breaking and the rock wettability improvement effect. For example, it may be difficult for applying CO_2 in the field after water displacement as the oil attached to the pore surface is covered with water

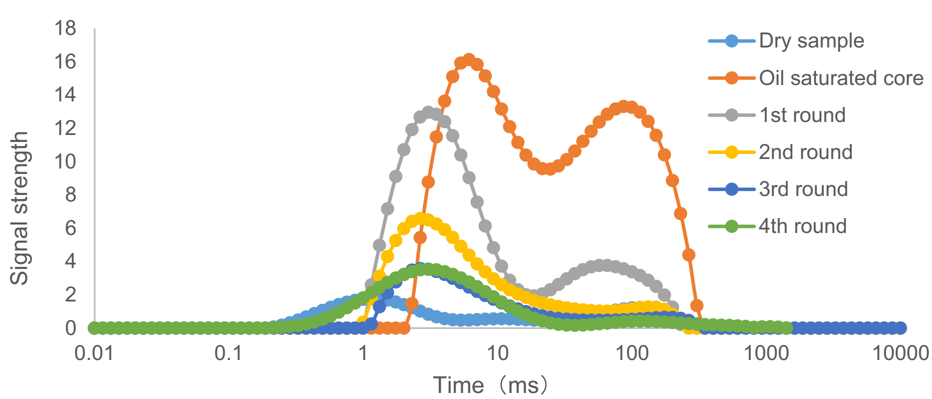


Fig. 13 NMR T2 spectrum of the core after various stages of the CO_2 huff-n-puff process.

film, but the injected carbonated water can be mutually soluble with the water film, thus make it easier for CO_2 to enter the oil phase. In addition, the CO_2 dissolved in water will form carbonic acid and the resulting ionic reactions with the contact reservoir surface can gradually improve the reservoir wettability to promote the flow of the oil (Riazi et al., 2011; Seyyedi and Sohrabi, 2016; Lashkarbolooki et al., 2018). Taking all the advantages of CW injection processes together with the considerable amount of 36.2% oil recovery rate obtained in this paper, we declare the CW huff-n-puff process could be a promising technique on tight oil recovery and carbon geological storage practices.

Based on the T2 spectrum results for both CW and CO_2 huff-n-puff processes, it is found the oil in the large size pores of the matrix is recovered more efficiently in the early rounds. It is deduced the injected CO_2 or CO_2 in CW water would firstly penetrate into the large pores of the core, and then expand to supplement the formation energy as well as to push out the oil in the large size pores. In the later stage of the huff-n-puff process, on the other hand, as the oil produced in the early cycles is replaced by invaded CO_2 or CW fluid in the large size pores, the oil relative permeability could be adversely affected, that is, the injected phase occupying the large size pores could retard the diffusive transport of CO_2 further deep into micropores, which is reflected by the much less oil production profiles during the later stages of the CW and CO_2 huff-n-puff processes (Mahzari et al., 2021).

4. Conclusions

In this paper, CW huff-n-puff process is scrutinized based on the oil recovery characteristics and the NMR T2 spectrum measurement. In addition, comparative studies on CO_2 and water huff-n-puff processes are also performed. The conclusions are listed as follows,

1) The total oil recover rate for CW huff-n-puff process could reach to 36.2% with 19.1%, 9,6%, 5.7% and 1.8% for the 1st, 2nd, 3rd and 4th cycle, respectively. NMR T2 spectrum results indicate the large size pores contribute most part of the recovered oil in the 1st cycle, while the oil in small size pores could be mobilized in the 2nd, 3rd and 4th cycle operations. The main EOR mechanisms of the CW huff-n-puff process could contribute to the oil expansion and oil viscosity reduction due to the gradual CO_2 transfer from water phase to the oil phase.
2) Compared with CW huff-n-puff process, CO_2 huff-n-puff process obtained higher oil recovery rate of 55.4%, which consists of 33.4%, 11.9%, 7.8% and 2.3% for the 1st, 2nd, 3rd and 4th cycle respectively. NMR test results indicate the most part of the oil in the large size pores could be recovered in the first two round operations, while further injection cycles may extract small amount of the oil from the small size pores of the tight core. The higher recovery rate could be contributed to the miscibility of the CO_2/oil system, which results in significantly increased diffusive transportation of CO_2 between CO_2 and the oil phase.
3) Compared with CW huff-n-puff process, water huff-n-puff process shows much lower oil recovery results of 14.4%, with 7.2%, 2.9%, 2.9% and 1.4% for the sequential four cycles.
4) As an effective alternative of CO_2, CW huff-n-puff process could recover considerable amount of oil from the tight cores with its unique advantages. It is concluded, therefore, the CW huff-n-puff technique could contribute to the tight oil recovery and carbon geological storage practices.

CRediT authorship contribution statement

Dongxing Du: Conceptualization, Methodology. **Yinjie Shen:** Investigation, Writing - original draft. **Weifeng Lv:** Formal analysis. **Chaofan Li:** Investigation. **Ninghong Jia:** Conceptualization. **Xiakai Song:** Investigation. **Xinrong Wang:** Investigation. **Yingge Li:** Writing - review & editing.

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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