

Study on microscopic development characteristics and influencing factors of nanopores during CO₂ huff-n-puff in Lucaogou Formation shale oil reservoir

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ABSTRACT

The characteristics of low porosity, low permeability, and small pore radius of the shale oil reservoir in the Lucaogou Formation make it difficult to develop and result in low production. CO₂ huff and n-puff can effectively improve the recovery rate of shale oil in the Lucaogou Formation. In this study, high-pressure mercury injection technology was used to characterize the pore distribution of the core samples. Combined with nuclear magnetic resonance technology, the recovery of different pores during CO₂ huff and n-puff were studied, and the effects of injection pressure and soaking time on core recovery were analyzed. Parallel CO₂ huff and puff experiment were conducted to investigate the influence of reservoir heterogeneity on the recovery of CO₂ huff and n-puff. The results showed that the pores in the Lucaogou Formation shale oil reservoir were mainly nano-pores, and small pores with a pore radius between 2nm and 50nm accounted for more than 70% of the total pores. The content of large pores in the core samples increased with the increase of core permeability. CO₂ huff and n-puff can effectively extract shale oil from the Lucaogou Formation. When the injection pressure is higher than the minimum miscibility pressure, increasing the injection pressure can increase the oil recovery efficiency of large pores in the core samples. The higher the content of large pores, the greater the oil recovery rate of the core samples. When the soaking time is increased from 5h to 10h, the dissolution and diffusion of CO₂ in small pores are enhanced, and the recovery rate of small pores increases by about 10%. However, the recovery rate of small pores no longer increases when the soaking time exceeds 10h. The presence of fractures can effectively enhance the recovery of small pores around the fractures. However, the presence of fractures increases the heterogeneity of the reservoir, and the

greater the difference in core permeability, the lower the recovery rate of small pores. The recovery rate of small pores in the matrix core samples is only 12.17%.

Keywords: CO₂, Huff and n-puff, Shale oil reservoir, Reservoir heterogeneity, Nanopores

1. INTRODUCTION

Shale oil reservoirs are a type of unconventional reservoir that possess characteristics such as low porosity, low permeability, and complex reservoir structure. These factors make fluid flow in the reservoirs challenging, ultimately resulting in difficulty in effectively producing resources. At present, the main development method of shale oil reservoirs is to form a fracture network in the shale oil reservoir through large-scale volume fracturing to improve the fluid flow capacity in the reservoir. Despite this, due to the small storage space of shale oil reservoirs and the large number of nanopore throats, the flow resistance of shale oil is extremely great, and the production of shale oil decreases rapidly during the depletion development process.

The CO₂ molecule has a small radius and can enter the smaller pores in the reservoir to supplement the formation energy. At the same time, it has the functions of dissolving and expanding to reduce the crude oil viscosity, extraction, and miscibility, significantly reducing the flow resistance of crude oil in the reservoir. Therefore, it is widely used in the development of unconventional reservoirs. The research of Zuloaga et al. (Zuloaga et al., 2017) on the recovery effects of CO₂ huff-n-puff and continuous CO₂ flooding under different permeabilities showed that matrix permeability is the main factor affecting the development effect. When the matrix permeability is less than 0.03mD, CO₂ huff-n-puff can achieve better recovery. The CO₂ huff-n-puff process is mainly divided into three stages: CO₂ injection stage,

soaking stage, and production stage. Injection pressure, well soaking time and production pressure are the main parameters of each stage and have a great impact on CO₂ huff and puff recovery. Numerous studies have demonstrated that injecting CO₂ under near-miscible and miscible conditions can yield better recovery results (Gamadi et al., 2014; Liu et al., 2022; Song and Yang, 2017; TANG et al., 2021), and the injection pressure should be greater than the minimum miscible pressure of 200 psi measured in slim tube experiments (Li et al., 2017). Increasing the soaking time can effectively enhance shale oil recovery, but prolonged soaking time has a minimal contribution to shale oil recovery (Li et al., 2016; Shi et al., 2023; Zhu et al., 2020).

Nuclear magnetic resonance can measure the hydrogen signal of fluid in the core under non-destructive conditions, thereby determining the distribution of fluid in the core. With the application of nuclear magnetic technology in the field of petroleum

2. EXPERIMENT

The Lucaogou Formation cores was used in this experiment. The main components of the core are quartz and dolomite, with a small amount of potassium feldspar, plagioclase, and calcite. The core has a low clay mineral content of approximately 10%. The experiment utilized a simulated oil composed of kerosene and crude oil, with an oil viscosity of 3.5mPa·s and an oil density of 0.857g/cm³.

2.1 High-pressure mercury intrusion experiment

High-pressure mercury intrusion is the most common pore throat structure testing method. In this experiment, the American corelab CMS300 and the American AutoPore IV 9505 mercury intrusion instrument were used to conduct high-pressure mercury intrusion tests on three cores. The selected cores porosity ranges from 2.178% to 7.579%, and the cores permeability ranges from 0.003 to 0.038 mD. The core

Table 1 Core parameters

Core No.	Core length / cm	Core diameter / cm	Porosity / %	Permeability / mD
Core 1	2.50	2.454	7.597	0.006
Core 2	2.50	2.458	5.431	0.008
Core 3	2.50	2.463	2.178	0.038

engineering, scholars have combined nuclear magnetic resonance technology to conduct detailed studies on the fluid distribution in the core pores during the CO₂ huff-n-puff process. Gao et al. (Gao et al., 2021) studied the changes in the recovery of different pores with injection pressure and soaking time. As the injection pressure and well soaking time increase, the recovery of large pores increases. Short soaking time is beneficial to shale oil production in small pores. According to Huang et al. (HUANG et al., 2022), the recovery rate of small pores increases linearly with an increase in injection pressure. Furthermore, increasing the soaking time can effectively enhance the recovery rate of small pores, with a suggested soaking time of 10 hours.

Reservoir heterogeneity is an important factor affecting CO₂ huff-n-puff (Chen et al., 2013). However, few studies have conducted experiments to investigate this factor. In this study, nuclear magnetic resonance and parallel huff-n-puff experiments were combined to characterize the microscopic flow characteristics in core pores to study the impact of reservoir heterogeneity on the CO₂ huff-n-puff. At the same time, the effects of injection pressure and well soaking time on CO₂ huff-n-puff were analyzed.

parameters are shown in Table 1. The maximum mercury injection pressure in the experiment is 200MPa.

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2.2 CO₂ huff-n-puff experiment

In this study, 9 cores were used to conduct CO₂ huff-n-puff experiments. The experimental flowchart is shown in Fig. 1. The detailed experimental steps are as follows:

(1) After the cores were washed and dried, the porosity and permeability were tested, and the dry weight of the cores were measured;

(2) The cores were evacuated for 24 hours using a vacuum pump, and then the high-pressure saturation device was used to pressurize and saturate the cores;

(3) After the saturation is completed, the wet weight of the cores were measured, and a nuclear magnetic resonance test was conducted to obtain the nuclear magnetic T₂ spectrum of the cores in the saturated oil state;

(4) Then placed the cores into the core holder for the CO₂ huff-n-puff experiment;

(5) Use the ISCO pump to inject CO₂ at constant pressure. When the pressure rises to the set pressure, closed the valve and soaked;

(6) After a period of soaking, gradually reduce the pressure to atmospheric pressure. When no fluid is produced, end the huff and puff experiment, and NMR testing was performed.

injection experiment are shown in Fig. 2. It can be found from the figure that the pores of the Lucaogou Formation shale oil reservoir are mainly nanopores. The core pore radius ranges from 1nm to 1000nm, and the average pore radius is less than 100nm. As the permeability of the core increases, the distribution range of pore radius in the core becomes larger and the number of large pores increases. The core parameters measured by high-pressure mercury injection are shown in Table 2.

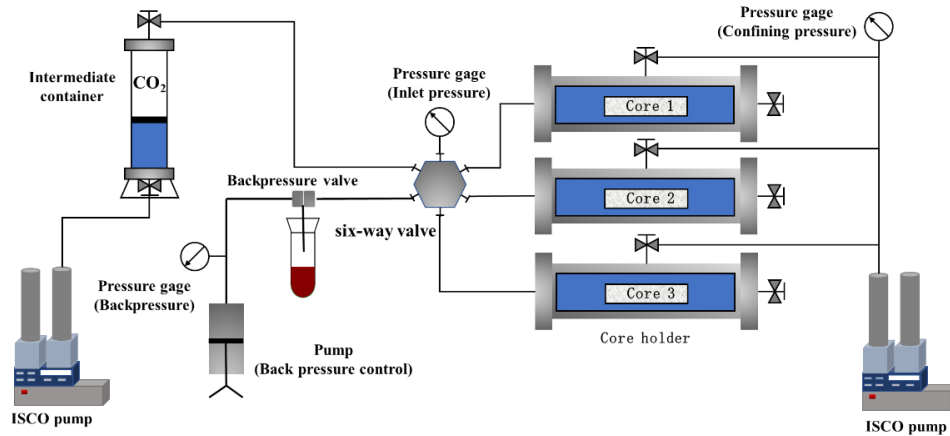


Fig. 1 Experimental flowchart of CO₂ huff-n-puff parallel experiment

3. RESULT AND DISCUSSION

3.1 Core pore distribution

The capillary force curve and pore distribution curve of the core measured through the high-pressure mercury

3.2 Effect of injection pressure on CO₂ huff-n-puff

The thin crude oil used in the experiment has a minimum miscibility pressure of 9MPa with CO₂ at reservoir temperature. This pressure is significantly

Table 2 Core physical property parameters measured by high-pressure mercury injection experiment

Core No.	permeability / mD	Porosity / %	Maximum pore radius / μm	Mean pore radius / μm	Sorting coefficient	Skewness	Homogeneity coefficient	Maximum mercury saturation / %	Mercury removal efficiency / %	Displacement pressure / MPa
Core 1	0.006	7.66	0.268	0.058	1.93	-0.192	0.218	95.55	11.38	2.74
Core 2	0.008	6.82	0.053	0.016	1.15	-0.048	0.307	97.61	10.02	13.78
Core 3	0.038	10.88	0.537	0.097	2.27	-0.488	0.181	94.25	24.42	1.37

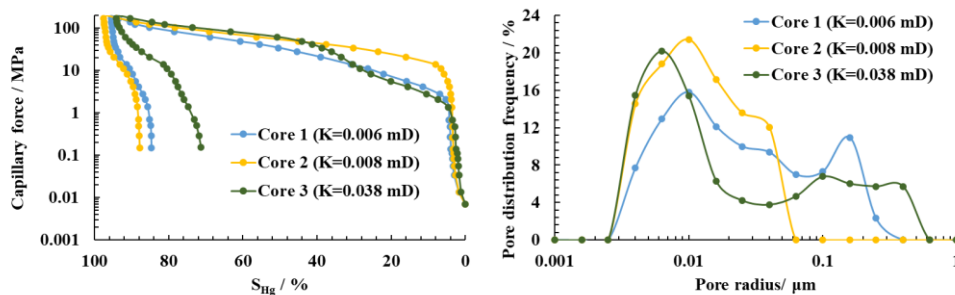


Fig. 2 Capillary force curve and core pore distribution

lower than the formation pressure of shale oil reservoirs, which is 27.4MPa. Therefore, achieving a miscible state is possible under the formation pressure. In order to clarify the impact of injection pressure on the CO₂ huff-n-puff, this study set different injection pressures to study the CO₂ huff-n-puff process. The core parameters and recovery rate are shown in Table 3. The NMR T₂

permeability of Core 6 and Core 7 is greater than 0.01mD, and the pores with larger radius in the core increase. Although the core output is mainly small pores, the proportion of large pore output in the total produced oil volume increases. When the injection pressure increases from 20MPa to 25MPa, the recovery degree of small pores does not increase significantly. As the

Table 3 CO₂ huff-n-puff recovery of Cores under different injection pressures

Core No.	Porosity / %	Permeability / mD	Injection pressure / MPa	Soaking time / h	Cyclic number	Recovery / %
Core 4	3.74	0.004	20	10	3	30.54
Core 5	12.02	0.007	25	10	3	31.48
Core 6	3.05	0.019	20	10	3	29.14
Core 7	6.80	0.04	25	10	3	37.12

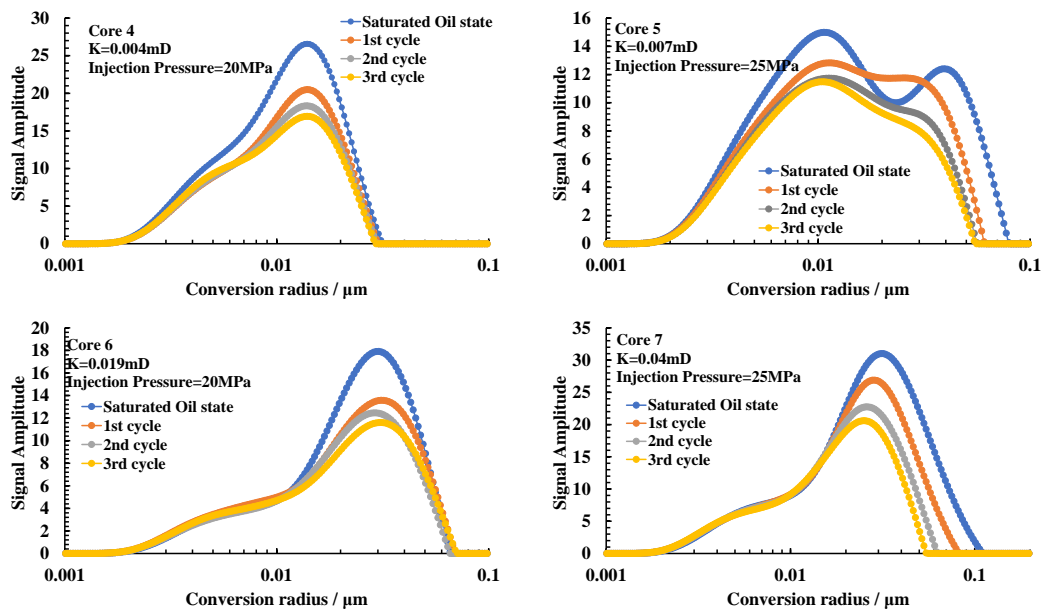


Fig. 3 NMR T₂ spectrum before and after CO₂ huff-n-puff under different injection pressures

spectrum before and after CO₂ huff-n-puff under different injection pressures are shown in Fig. 3.

It can be found that the recovery characteristics of cores with different permeability vary as the injection pressure increases. For cores with a permeability ranging from 0.001 to 0.01mD, the change in core recovery is not significant when the injection pressure is raised from 20MPa to 25MPa. However, for cores with a permeability greater than 0.01mD, the core recovery rate increases by approximately 8%. Comparing the pore distribution of each core, it can be seen that the permeability of Core 4 and Core 5 is less than 0.01mD, the proportion of small pores in the core is greater than 95%, and the oil produced in small pores accounts for more than 90% of the total produced oil. The

permeability of the core increases, the content of large pores in the core increases, and the proportion of oil produced from large pores increases. Therefore, it can be inferred that when the injection pressure is greater than the miscible pressure, increasing the injection pressure can effectively enhance the oil washing efficiency of large pores and enhance the degree of recovery from large pores. However, the sweep range of CO₂ in small pores cannot be effectively increased, and the recovery rate of small pores cannot be effectively improved.

3.3 Effect of soaking time on CO₂ huff-n-puff

Results should be clear and concise. The dissolution and diffusion of CO₂ in crude oil is the main factor for CO₂ huff-n-puff to improve shale oil recovery. Soaking after

injecting CO₂ can increase the contact time of CO₂ with crude oil, enhancing the dissolution and diffusion of CO₂. In this study, three cores were selected to conduct CO₂ huff-n-puff experiments under different soaking times, and the recovery of different pores in each core were analyzed. Each core parameter and core recovery are shown in Table 4. The T₂ spectra of the three cores before and after CO₂ huff-n-puff are shown in Fig. 4.

Increasing the soaking time from 5 to 10 hours results in core recovery rate increase of approximately 10%. However, when the soaking time is increased to 15 hours, the core recovery rate remains relatively unchanged. The analysis of the pore distribution in the cores reveals that small pores are the dominant pores in all three cores, accounting for more than 90%. Additionally, the crude oil produced mainly originates from small pores. After soaking for 5 hours, the proportion of crude oil produced from small pores is 14.34%. This percentage increased to 30.49% after soaking for 10 hours. Extending the well soaking time to 15 hours, the proportion of oil produced from small pores was 26.23%, which did not continue to increase.

Increasing the soaking time can enhance the sweep range of CO₂, leading to improved diffusion and dissolution of CO₂ in small pores, which can effectively increase the recovery of small pores. However, it is important to note that excessively long soaking times may not lead to further improvement in the recovery of small pores. Therefore, it is advisable to optimize the soaking time to achieve the best possible results.

3.4 Effect of reservoir heterogeneity on CO₂ huff-n-puff

The heterogeneity of the reservoir is an important factor that affects the CO₂ huff-n-puff. Three cores with different permeabilities were selected for parallel flow rate experiments. Prior to the experiment, physical fracturing was carried out on the Core 11, which contained a large number of cracks, resulting in a high permeability of 75.47mD. The Core 12 contained a small number of natural cracks, with a permeability of 6.39mD, much higher than the matrix core. The YLH12 core was a matrix core with a permeability of 0.012mD. The parameters of each core, experimental plan, and experimental results are shown in Table 5. The nuclear

Table 4 CO₂ huff-n-puff recovery rate under different soaking times

Core No.	Porosity / %	Permeability / mD	Injection pressure / MPa	Soaking time / h	Cyclic number	Recovery / %
Core 8	2.17	0.003	20	5	3	14.34
Core 4	3.05	0.004	20	10	3	30.49
Core 10	4.19	0.005	20	15	3	26.23

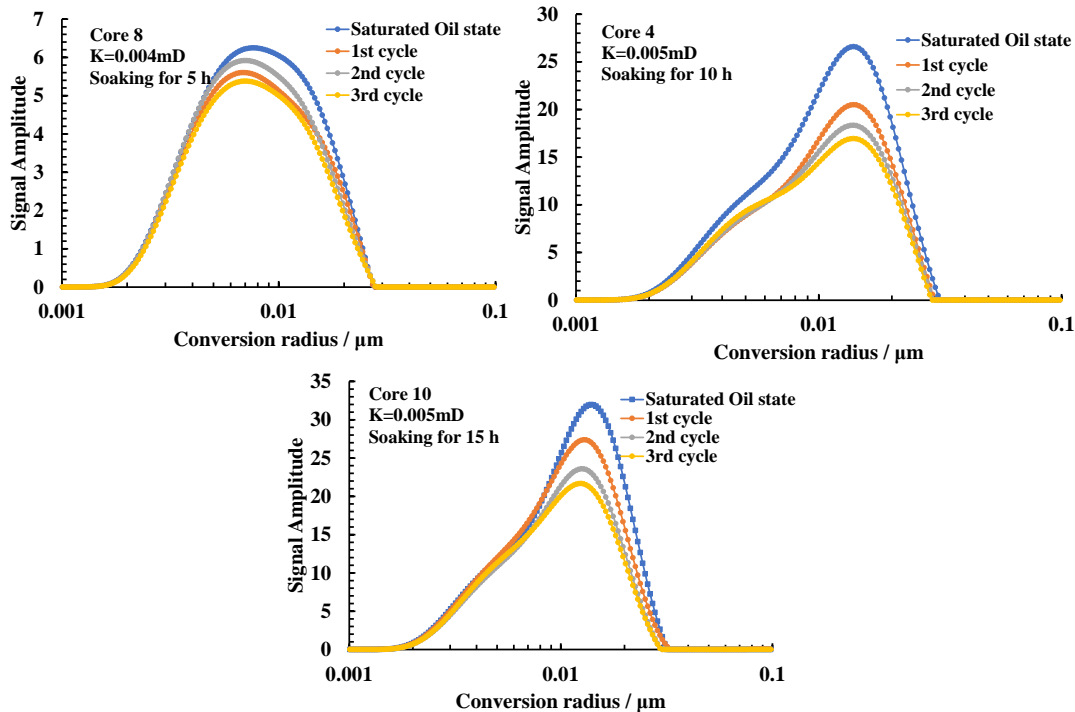


Fig. 4 The T₂ spectra of the three cores before and after CO₂ huff-n-puff under different soaking time

magnetic resonance spectra of the cores before and after flow rate testing are shown in Fig. 5.

recovery of artificially fractured Core 11 is about four times that of the matrix core. The CO₂ huff-n-puff

Table 5 Recovery of CO₂ huff-n-puff with different permeability

Core No.	Porosity / %	Permeability / mD	Injection pressure / MPa	Soaking time / h	Cyclic number	Recovery / %
Core 11	6.33	75.47	20	10	4	60.19
Core 12	3.84	6.39	20	10	4	30.20
Core 13	3.13	0.012	20	10	4	16.00

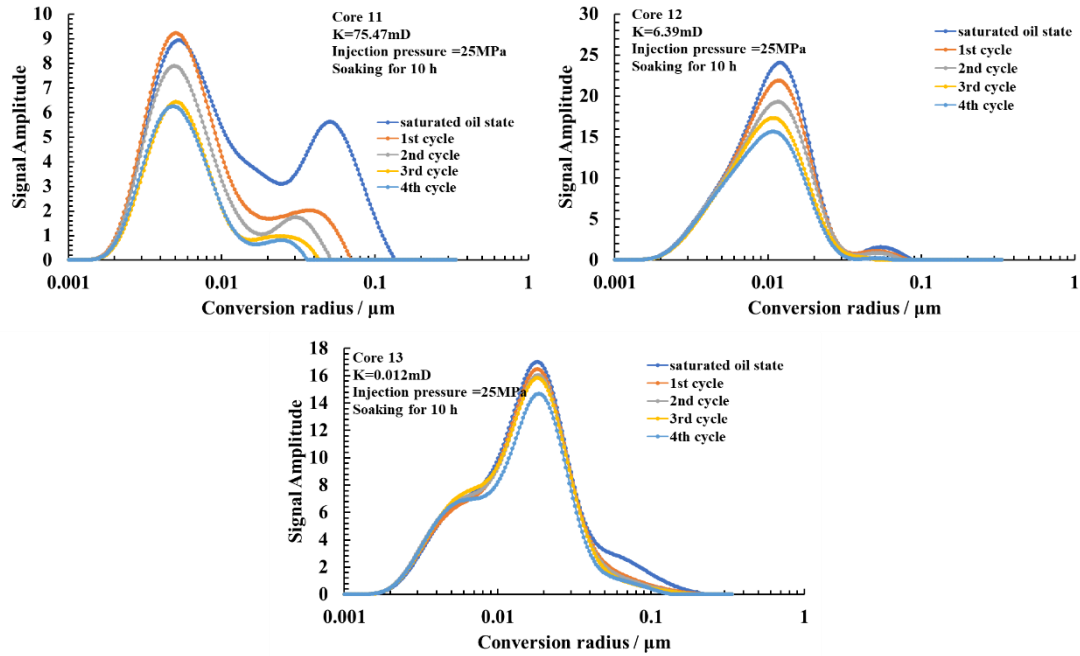


Fig. 5 NMR T₂ spectrum of parallel core before and after CO₂ huff-n-puff

It can be found from the table that the recovery of the Core 12, which contains natural fractures, is approximately twice that of the matrix Core 13, and the

recovery increases with the permeability of the core. This is mainly due to the significant increase in the size of the pores, as the core permeability increases. Consequently,

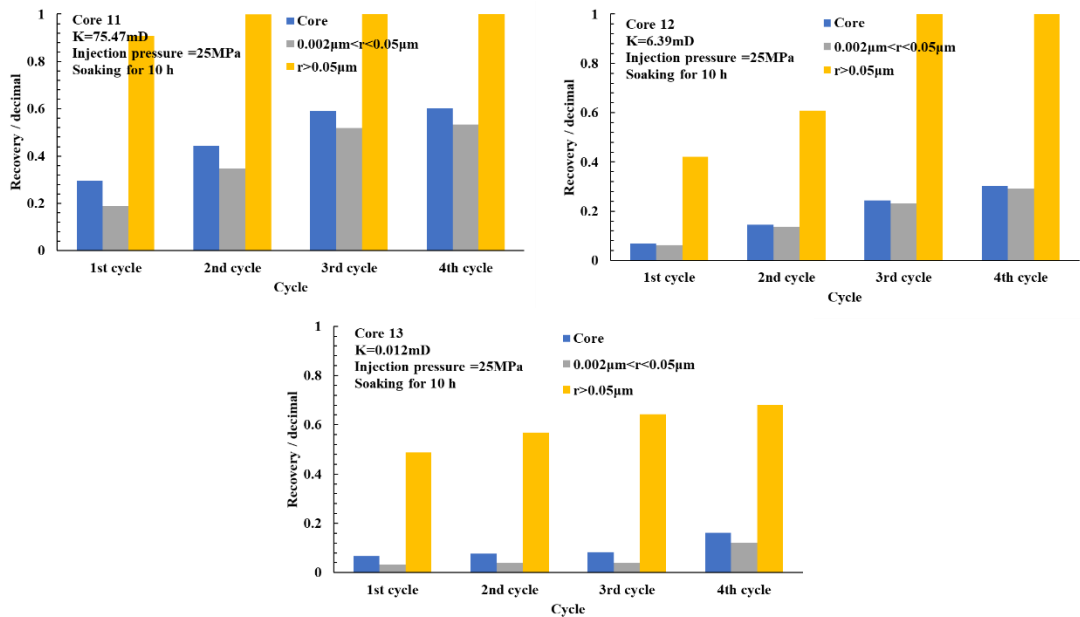


Fig. 6 Different core pore recovery of parallel core

the recovery of large pores also increases. Based on the recovery degree of each pore in the core, it can be deduced that the recovery degree of large pores is significantly higher than that of small pores. However, since the volume of large pores accounts for a small proportion of the core, their impact on the total recovery degree is minor. The contribution to the core recovery mainly comes from small pores with a radius of 2nm to 50nm, which accounts for more than 70% of the total recovery. The presence of fractures in the core greatly increases the contact area between CO₂ and the core matrix, which significantly improves the recovery of small pores. The recovery of small pores in artificially fractured core accounts for approximately 45% of the total pore volume, while the recovery of small pores in naturally fractured core accounts for approximately 29%. The recovery of small pores in matrix core accounts for approximately 11% of the total pore volume.

Although the existence of fractures greatly improves the recovery of fractured cores, compared with the recovery rate of cores with the same permeability level, the recovery rate of cores with lower permeability in the parallel experiment is significantly lower. In the single core CO₂ huff-n-puff experiment, the matrix core recovery rate was greater than 20%, and the small pore recovery accounted for about 20% of the total pore volume, which was much higher than that in parallel core experiment. This shows that although the existence of fractures enhances the mobility of matrix pores around the fractures, the existence of fractures also increases the heterogeneity of the parallel cores, causing CO₂ to channel and flow more easily to parts with higher permeability, thereby inhibiting the matrix core recovery.

4 CONCLUSION

In this study, nuclear magnetic resonance was used to characterize the nanopore production characteristics during the CO₂ huff-n-puff process. The study investigated the effects of reservoir heterogeneity, injection pressure, and soaking time on the pore production characteristics. Based on the results, the following conclusions were drawn:

(1) The pores of the Lucaogou Formation shale oil reservoir are mainly nanopores. The core pore radius ranges from 1nm to 1000nm, and the pores with a radius between 2nm and 50nm account for more than 70%. The core mercury removal efficiency is extremely low and the pore connectivity is poor.

(2) when the injection pressure is greater than the miscible pressure, increasing the injection pressure can

effectively enhance the oil washing efficiency of large pores and enhance the degree of recovery from large pores. However, the sweep range of CO₂ in small pores cannot be effectively increased, and the recovery rate of small pores cannot be effectively improved.

(3) Increasing the soaking time can enhance the sweep range of CO₂, improving the recovery of small pores. However, long soaking times may not lead to further improvement in the recovery of small pores. It is advisable to optimize the soaking time to achieve the best possible results.

(4) Although the existence of fractures enhances the mobility of matrix pores around the fractures, the existence of fractures also increases the heterogeneity of the parallel cores, causing CO₂ to channel and flow more easily to parts with higher permeability, thereby inhibiting the matrix core recovery.

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DECLARATION OF INTEREST STATEMENT

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. All authors read and approved the final manuscript.

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