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Study on producing effect of CO_2 injection into Gulong shale oil

Deng Sen[1,2*], Zhang Jiang[1,2], Liu Yong[1,2], Li Binhui [1,2], Lan YuBo[1,2], Guo Tianjiao[1,2], Cui huanqi[1,2], Xu Quan[1,2], Yang wenlu[1,2], Cao Sheng[1,2]

1 Exploration and Development Research Institute of Daqing Oilfield Co Ltd, Daqing 163712, China

2 Heilong jiang Provincial Key Laboratory of Reservoir Physics & Fluid Mechanics in Porous Medium, Daqing 163712, China
(*Corresponding Author: 342025761@qq.com)

ABSTRACT

In order to clarify the effectiveness of CO_2 injection at high temperature and high pressure in Gulong shale oil, the conversion coefficient between shale T_2 value and pore throat radius is first given based on the results of shale mercury injection and nitrogen adsorption experiments. Based on the T_2 spectrum characteristics of saturated shale, shale pores are divided into small pores, medium to large pores, and lamellation fractures. At the same time, a calculation method for shale oil recovery is provided. Then, the effects of huff and puff cycle, soak time, and fractures on huff and puff oil displacement effect are examined, And analyze the degree of change in core pore structure after huff and puff, and finally compare the oil displacement effects of shale oil CO_2 huff and puff and CO_2 displacement, and provide the optimal oil displacement method. The experimental results show that the shale oil in the middle to large pores and lamellation fractures has the highest recovery after huff and puff, while the shale oil in the small pores has the lowest recovery. Increasing the soaking time only increases the shale oil recovery by0.81%, and fracturing can increase the shale oil recovery in the small pores by 11.33%, effectively utilizing the shale oil in the small pores, and improving the recovery of shale oil in small pores is the key to improving the recovery of Gulong shale oil; Compared to CO_2displacement, CO_2huff and puff can increase shale oil recovery by 30.98%, and shale oil in dry rock samples can be utilized, with better huff and puff effects than displacement; The combination of displacement and huff-n-puff can increase the recovery rate by more than 12.88% compared to only using huff-n-puff, and can significantly increase the recovery of shale oil in small pores; The pore structure of the core undergoes significant changes after huff and puff, and the difference in shale gravel content is an important reason for the significant differences in pore structure changes before and after shale huff and puff. The experimental results can provide important basic parameters for the practice of the Gulong shale oil field.

Keywords: Shaleoil,pore structure,CO_2 displacement,CO_2 huff-n-puff,nuclear magnetic resonance

NONMENCLATURE

Abbreviations	
IUPAC	International Union of Pure and Applied Chemistry
Symbols	
C	conversion coefficient
r	pore-throat radius
M_{OR}	residual oil mass in the dry rock sample
M_o	the mass of saturated oil in the rock core
S_o	the sum of T_2 spectrum signal amplitudes of the saturated oil core
S_{OR}	the sum of T_2 spectral signal amplitudes of dry rock samples
$M_{produce}$	the cumulative oil production of shale cores
M_{total}	the total oil content of shale cores

1. INTRODUCTION

In the initial stage of exploitation of most unconventional reservoirs, the formation pressure is depleted rapidly, the oil and gas production is declining rapidly, and the production is stagnating at a low level. The oil mining output during primary oil production is estimated to be 5% to 10% of the original oil [1]. Due to the rapid diffusion of CO_2 in crude oil, the role of energy increase and viscosity reduction is obvious, it can enter pores larger than CO_2 molecular diameter (0.33nm), and

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can use shale oil in shale nanoscale pores [2], Therefore, injecting CO_2 has become the most promising measure for improving shale oil recovery.

Foreign scholars conducted indoor gas injection physical simulation experiments using cores from shale reservoirs such as Baken to investigate the oil displacement effect of CO_2 on shale oil. The results showed that CO_2 is easily dissolved in shale oil and has a low miscibility pressure with shale oil. huff and puff can increase shale oil recovery by 33% to 85% [3]. Domestic scholars mainly focus on shale oil reservoirs such as Jimusar in the the Junggar Basin, Chang 7 reservoir in the Ordos Basin, and Jiyang Depression in the Bohai Bay Basin, and use nuclear magnetic resonance, CT scanning, electron microscope scanning and other experimental techniques to study the effects of fractures, soak time, gas injection pressure and other factors on the CO_2 huff and puff oil displacement effect of shale oil [4], but the simulation is all huff and puff under the condition of immiscible state, with low huff and puff temperature and pressure, It is difficult to represent the characteristics of shale oil production in actual reservoirs, and a few scholars have conducted research on the effectiveness of online CO_2 injection displacement using nuclear magnetic resonance for shale oil [5]. However, there is still no consensus on which effect is better for CO_2 huff and puff or CO_2 displacement. During the huff and puff process, the interaction between CO_2 and shale can cause changes in the pore structure of the shale. Under the influence of multiple factors, both a decrease and an increase in reservoir porosity can occur after CO_2 action [6].

Gulong shale oil is mainly composed of nanoscale pores and is a pure shale type shale oil. The storage space is mainly composed of clay mineral intergranular pores and lamellation fractures [7]. In order to explore the effectiveness of CO_2 huff and puff production in Gulong shale oil, based on the low magnetic field nuclear magnetic resonance experimental method and the characteristics of nuclear magnetic resonance T_2 spectrum, the division boundaries of small pores, medium to large pores, and lamellation fractures in Gulong shale are given. The effects of soaking time, huff and puff cycle, and fractures on the effectiveness of shale oil production are analyzed, and the impact of CO_2 huff and puff on shale pore structure is examined. The better method of shale oil production is compared between CO_2 displacement and CO_2 huff and puff, Optimize the optimal gas injection method to provide theoretical support for the development of Gulong shale oil and gas injection.

2. EXPERIMENTAL DESIGN

2.1 Experimental Materials

Experimental core: the shale core sampling horizon is the first member of Qinghe Formation, the lithology is mainly grayish black laminated shale, The core used is mainly used for conducting experiments such as CO_2 huff and puff, CO_2 displacement, CO_2 huff-n-puff after CO_2 displacement. Among them, core 121-1 and core 121-2 are parallel rock samples. The basic parameters of the core are shown in Table 1. The experimental oil used is Gulong surface degassed shale oil, with a minimum miscibility pressure of 20.73MPa and an original formation pressure of 36MPa.

Table 1 Core Foundation Parameters Table

Sample number	length (cm)	diameter (cm)	dry weight （g）
3	7.57	2.50	95.29
14	4.77	2.50	61.49
16	3.81	2.50	47.14
121-1	3.26	2.50	42.69
121-2	2.50	2.50	32.32
27	3.42	2.52	41.36

2.2 Experimental device and Experimental steps

The nuclear magnetic resonance experiment uses the low magnetic field nuclear magnetic resonance rock sample analyzer of MacroMR12-150H-I produced by Suzhou Newman, with a magnetic field intensity of 0.3 ± 0.05T; The maximum displacement pressure of the core holder used in the CO_2 displacement experiment is 70MPa, and the maximum temperature is 150 ℃; The huff and puff experiment adopts a high-temperature and high-pressure CO_2 resistance experimental device, with a maximum huff and puff pressure of 70MPa, and a maximum huff and puff temperature of 150 ℃.

Steps of CO_2 displacement experiment: Set the temperature of the incubator to 100 ℃ and the back pressure to 22MPa, slightly higher than the miscibility pressure of CO_2 and shale oil. Inject at a rate of 0.1ml/min until the core no longer produces oil. Stop the experiment, remove the core, measure its weight and nuclear magnetic resonance T_2 spectrum.

Steps of CO_2 huff and puff experiment: (1) Place the shale core in a high-temperature and high-pressure CO_2 reactor, vacuum the reactor, and inject CO_2 into the reactor. Set the huff and puff temperature to 100 ℃, and set a certain huff and puff pressure and soak time; (2) After the completion of huff and puff, slowly reduce the pressure inside the container, remove the core from the reactor, measure the weight of the core and the nuclear magnetic resonance T_2 spectrum; (3) Before each new huff and puff cycle begins, the container needs to be vacuumed to ensure that there is no residual air inside the container.

3. RESULTS AND DISCUSSION

3.1 The analysis of shale pore structure

3.1.1 The T_2 value and pore throat radius conversion

The T_2 spectrum of shale pore fluid can reflect the pore structure of shale and is proportional to the pore size. The larger the pore size, the longer the relaxation time, and vice versa. Nuclear magnetic resonance can be combined with mercury intrusion experiments and nitrogen adsorption experiments to convert the T_2 relaxation time distribution into the pore throat radius distribution [8]. The conversion formula is:

$$r = CT_2 \qquad (1)$$

The correlation coefficient method is used to fit the pore size distribution frequency curve obtained from the nuclear magnetic resonance T_2 value with the pore size distribution frequency curve obtained from the nitrogen adsorption experiment and high-pressure mercury injection experiment. The fitted conversion coefficient is 30.05nm/ms, as shown in Fig.1.

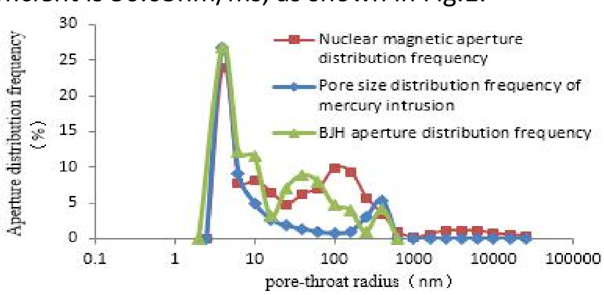

Fig.1 The T_2 value and pore throat radius conversion

3.1.2 The T_2 spectral characteristics of saturated shale

The Gulong shale is different from conventional reservoirs,the T_2 spectrum of Gulong saturated shale is mainly in a three peak state (Fig.2). The number, size, and position of T_2 spectrum peaks in saturated shale can reflect the characteristics of shale pore structure. CT scanning experiments show that at least three types of pores are developed in the shale, including small pores, medium to large pores, and lamellation fractures (Fig.3), In Fig.3, red is pore, gray is clay, quartz, feldspar and other minerals, green is iron bearing carbonate rock, and yellow is pyrite.

The nuclear magnetic resonance T_2 spectrum shows that the position of the T_2 spectrum trough of the shale is located near T_2=1ms (30.05nm) and T_2=33ms (991.65nm). The residual oil in the dry rock sample is mainly distributed in pores less than 1ms (30.05nm). Long term dry rock samples generally only have shale oil in small pores, and shale oil in medium to large pores or lamellation fractures has generally been lost. Therefore, based on the pore classification method of IUPAC and the nuclear magnetic resonance T_2 spectrum morphology characteristics of Gulong shale oil, pores less than 1ms (30.05nm) are classified as small pores, pores ranging from 1 to 33ms are classified as medium to large pores, and pores larger than 33ms (991.65nm) are classified as lamellation fractures.

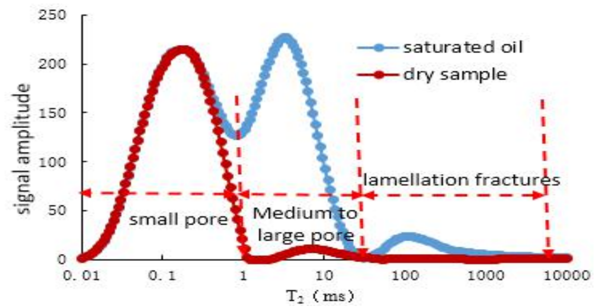

Fig.2 The T_2 spectrum of saturated oil in core 27

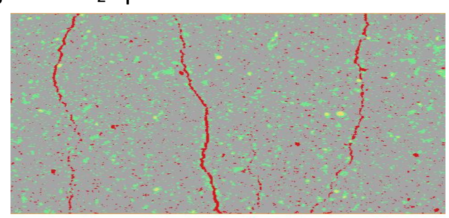

Fig.3 The CT scanning images of core 27

3.1.3 Calculation of total oil content in shale

Shale dry rock samples generally contain a large amount of residual oil, which cannot be ignored when calculating the total oil content of shale. When conducting nuclear magnetic resonance testing, it is necessary to conduct nuclear magnetic resonance T_2 spectrum testing on the dry rock samples. By analyzing the differences in nuclear magnetic resonance T_2 spectrum signals between the core before and after saturation of oil and the quality of saturated oil, the residual oil content in the dry rock sample can be calculated, and then the total oil content can be calculated. The formula for calculating the residual oil content of the dry rock sample is:

$$M_{OR} = \frac{S_{OR}}{S_o - S_{OR}} \times M_o \qquad (2)$$

The formula for calculating the recovery of shale oil is:

$$E_{or} = \frac{M_{produce}}{M_{or} + M_o} = \frac{M_{produce}}{M_{total}} \qquad (3)$$

The total oil content of shale core after saturation with oil is shown in Table 2. It can be seen that there is a large amount of residual oil in the dry rock sample, and the oil saturation reaches over 44%. Therefore, the quality of shale oil in the dry rock sample cannot be ignored when calculating the total oil content of saturated shale core.

Table 2 Total Oil Content of Shale Core

Core number	S_{or}/S_o	S_o	M_o(g)	M_{or} (g)	M_{total}(g)
27	0.52	15830	1.03	1.09	2.12
16	0.50	14344	0.94	0.94	1.87
121-1	0.44	6442	0.48	0.38	0.86
3	0.51	83113	1.42	1.50	2.92

3.2 The factors affecting recovery of CO_2 huff and puff

3.2.1 The impact of huff and puff cycle on recovery

Conduct a huff and puff experiment on core 16, with a huff and puff pressure of 41MPa, slightly higher than the formation pressure, and a soak time of 2 days. The experimental results show that the more cyclic huff and puff, the higher the recovery of shale oil. After 4 th cycles of huff and puff, the recovery of shale oil does not change much, and after 6 th cycles of huff and puff, the recovery of shale oil reaches 62.49%. The variation trend of the recovery of shale oil of medium to large pores and lamellation fractures is the same, manifested as the optimal huff and puff cycle of 3, while the trend of recovery of small pores and total pores is the same, manifested as the optimal huff and puff cycle of 4. After the end of the 6th cycle, the recovery of shale oil in lamellation fractures, medium to large pores, and small pores reaches 85.06%, 92.29%, and 35.79%, respectively. The shale oil in medium to large pores and lamellation fractures has the highest recovery (see Fig.4), It can be seen that improving the recovery of shale oil in small pores is the key to improving shale oil recovery.

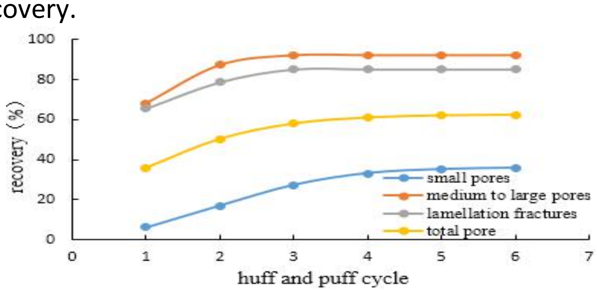

Fig.4 The Change of recovery in different pores

3.2.2 The impact of soaking time on recovery

A huff and puf test was conducted on core 27 with a soaking time of 4 days and a huff and puf pressure of 41MPa,and Six cycles of huff and puf were conducted. Compared with core 16, the soaking time increased from 2 days to 4 days. In the first cycle, the recovery of small pores, medium to large pores, lamellation fractures, and total pores increased by 5.44%, 9.20%, 8.61%, and 5.11%, respectively. The recovery of medium to large pores increased the most significantly, while the recovery of small pores increased the least. In the sixth huff and puff cycle, the recovery of small pores, medium to large pores, lamellation fractures, and total pores increased by 0.69%, 5.55%, 8.88%, and 0.81% respectively(Table 3). It can be seen that as long as the huff and puff frequency is sufficient, increasing the soaking time has little effect on the final recovery of small pores and total pores. However, the shale oil in medium to large pores and lamellation fractures has been effectively utilized. Improving the recovery of shale oil in small pores is still the key to improving shale oil recovery rate, However, extending the soaking time to improve oil recovery has limited effect, and excessively extending the soaking time has lower benefits [9].

Table 3 Comparison of shale oil recovery

Pore type	Core number	recovery(%)	
		1st cycle	6th cycle
small pores	16	6.20	35.79
	27	11.64	36.48
medium to large pores	16	68.17	92.29
	27	77.37	97.84
Lamellation fractures	16	65.64	85.06
	27	74.25	93.94
total pores	16	35.76	62.49
	27	40.87	63.30

3.2.3The impact of fractures on recovery

Core 121-2 has obvious fractures that run through the entire core, while core 121-1 has no obvious cracks. Due to the presence of fractures, there is a significant difference in the signal amplitude of saturated oil T_2 spectra of the two cores with pores greater than 33ms (see Fig.5 and Fig.6).

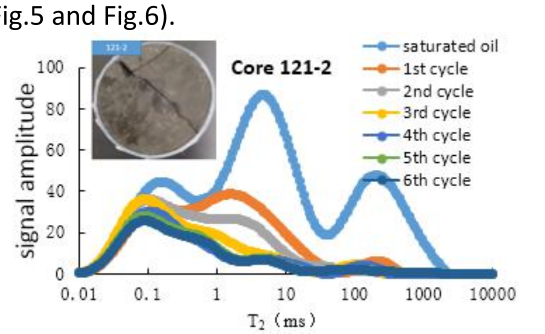

Fig.5 The T_2 Spectral of Core 121-2 in Different Cycles

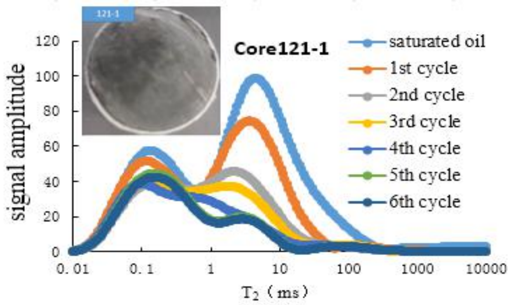

Fig.6 The T_2 Spectral of Core 121-1 in Different Cycles

Fractures can increase the drainage area of the matrix, reduce the flow resistance of crude oil discharged from the matrix, and promote material exchange between the fractures and the matrix. Therefore, the first huff-n-puff cycle of core 121-2 can achieve a relatively high recovery, which increases by 30.07%. After 6th cycle, the recovery of small pores, medium to large pores, fractures, and total pores increased by 11.33%, 5.83%, 9.27%, and 12.18%, respectively(Table4). The small pores contributed the most to the recovery, indicating that fracturing can significantly improve the recovery of shale oil in small pores.

Table 4 Comparison of shale oil recovery

Pore type	Core number	recovery(%)	
		1st cycle	6th cycle
small pores	121-2	16.48	40.89
	121-1	6.22	29.56
medium to large pores	121-2	56.07	91.25
	121-1	28.07	85.43
fractures	121-2	91.57	96.5
	121-1	73.68	87.22
total pores	121-2	54.24	78.14
	121-1	24.17	65.96

3.3 The changes in pore structure during huff and puff

After huff and puff, the core was re saturated with shale oil for T_2 spectrum testing, and compared with the T_2 spectrum of the saturated shale oil core before huff and puff. The experimental results showed that there was a significant change in the T_2 spectrum of the saturated oil core after huff and puff. The small porosity of core 27 decreased significantly, with a reduction degree of 29.72%, while the medium to large pores increased, with an increase rate of only 4.17%. The fracture volume increased most significantly, with an increase rate of 33.79% (see Fig.7); The overall porosity of core 16 decreased, with a decrease of 10.68% in small pores and only 6.73% in medium to large pores. The largest reduction in crack volume was observed, with a decrease of 66.61% (see Fig.8).

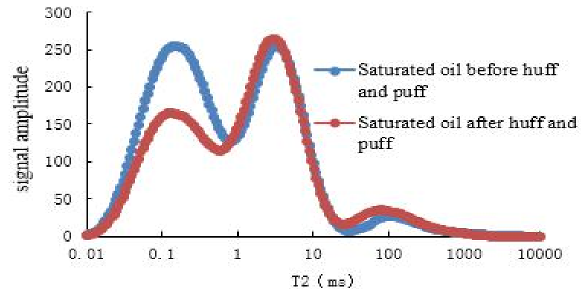

Fig.7 T_2 spectrum before and after core 27 huff-n-puff

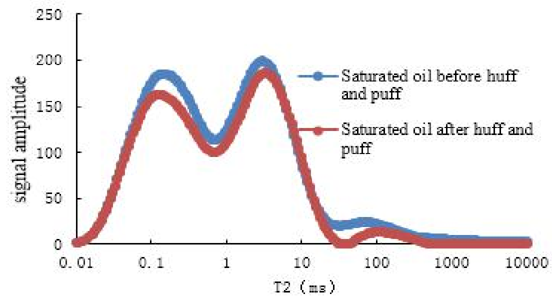

Fig.8 T_2 spectrum before and after core 16 huff-n-puff

The different content of shale sand and gravel is an important reason for the significant difference in pore structure changes before and after shale stimulation. Core 27 does not contain fine sand, and the silty sand content is 8.49% lower than Core 16. The clay content is 12.97% higher than Core 16. This is the main reason for the significant decrease in small pores and the increase in large pores after huff and puff of core 27, as shown in Table5.

Table5 Statistics of Shale Sand and Gravel Content

Core number	fine sand content (%)	Silty sand content (%)	clay content (%)	Average particle size (Φ)
16	4.48	68.53	26.99	6.735
27	0	60.04	39.96	7.492

3.4 Analysis of displacement experiment results

During the CO_2 displacement experiment, The injection pressure finally stabilizes at 23MPa. The weight of the core after displacement is 95.75g, and a total of 0.96g of shale oil is driven out, with a recovery of 32.88%. The T_2 cut-off value is 1.703ms (51.16nm), and the displacement mainly uses shale oil in pores above 51.16nm(see Fig.9).

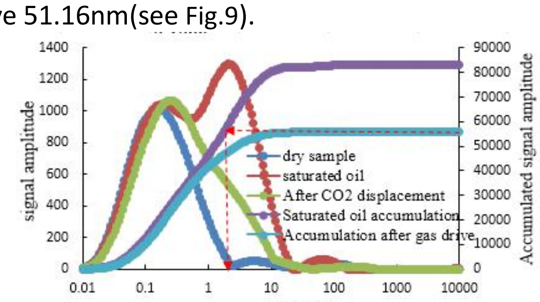

Fig.9 TheT_2 spectrum before and after CO_2 displacement

After displacement, the core 3 was subjected to CO_2 huff and puff, with a huff and puff pressure of 23MPa. After 5 rounds of huff and puff, the recovery reached 63.86%, and CO_2 huff and puff can increase the recovery by 30.98%. The recovery of small, medium to large pores, and lamellation fractures were increased by 28.26%, 28.56%, and 26.72%, respectively. The T_2 cut-off value reached 0.302ms(9.08nm), and the CO_2 huff and puff oil displacement effect is better than CO_2 displacement. Starting from the 6th huff and puff cycle, increasing the huff and puff pressure to 41MPa can increase the recovery by 12.32%, with a T_2 cutoff value of 0.161ms(4.91nm). After huff and puff, the recovery of shale oil in small pores, medium to large pores, and lamellation fractures can reach 47.71%, 29.77%, and 26.72%, respectively(seeFig.10).

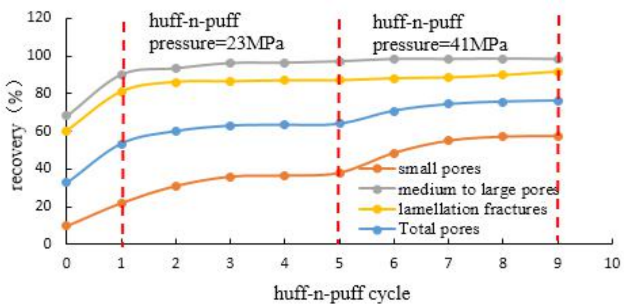

Fig.10 The recovery of core 3 at different cycles

The combination of displacement and huff-n-puff can increase the recovery by more than 12.88% compared to only huff and puff, and the recovery of shale oil in small pores can be increased by more than 20.97%(see Table 6). The combination of displacement and huff-n-puff can significantly improve the recovery rate of shale oil in small pores compared to only displacement.

Table 6 Comparison of recovery of different methods

Core number	recovery (%)				Oil displacement method
	small pores	medium to large pores	lamellation fractures	total pores	
27	36.48	97.84	93.94	63.30	Huff-n-puff
16	35.79	92.29	85.06	62.49	Huff-n-puff
3	57.45	98.12	91.53	76.18	displacement and huff-n-puff

4. CONCLUSIONS

The T_2 spectrum of Gulong saturated shale shows a three peak state, with three types of pores developed: small pores (less than 30.05nm), medium to large pores (30.05-991.65 nm), and lamellation fractures (greater than 991.65 nm). The dry rock samples contain a large amount of residual oil.

The shale oil in the medium to large pores and lamellation fractures has the greatest recovery after huff-n-puff. Increasing the soaking time only increases the shale oil recovery by 0.81%, while fracturing can increase the shale oil recovery rate in the small pores by 11.33%.

The combination of displacement and huff-n-puff can increase the recovery of shale oil by 30.98%, and the recovery of shale oil of small pores can be increased by 28.26%. the CO_2 huff-n-puff effect is better than CO_2 displacement. The combination of displacement and huff-n-puff can increase the recovery by more than 12.88% compared to only huff and puff, and the recovery of shale oil in small pores can be increased by more than 20.97%.

The pore structure of shale core undergoes significant changes after huff and puff, The lower the content of fine sand and silt, the higher the clay content, and the larger the average particle size, which is more likely to lead to an increase in fracture volume after huff and puff, and the more significant the reduction in small pores.

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