

INNOVATIVE TECHNOLOGIES OF OIL AND GAS

STUDY ON THE PLUGGING MECHANISM OF CARBON DIOXIDE OIL DRIVE IN SHALE

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The CO₂ enhanced oil recovery (EOR) technology is widely utilized in the development of shale oil reservoirs. However, reservoir plugging issues often occur during the CO₂ injection process, leading to reduced onsite development efficiency. Currently, the mechanism behind reservoir plugging is still unclear. Therefore, this research selects Jimsar crude oil from Xinjiang to conduct CO₂ displacement experiments on shale cores and analysis of crude oil components. A comparative analysis is conducted with previous studies to explore the plugging mechanism. The research results demonstrate that core plugging occurs when the displacement pressure exceeds 25 MPa, resulting in a significant decrease in core permeability. Under a displacement pressure of 40 MPa, the additional resistance accounts for as much as 72% of the total resistance, with high asphalt content and the extractive effect of CO₂ on crude oil components being important factors leading to core plugging. Therefore, in the field development process, the CO₂ enhanced oil recovery technology should be applied in a rational and tailored manner according to the properties of the field's crude oil.

Keywords: shale reservoir, carbon dioxide flooding, reservoir blockage mechanism, carbon dioxide extraction, asphaltene deposition.

1. Introduction

The shale oil and gas revolution has promoted the growth of global oil and gas production and reserves, prolonged the life cycle of the world's oil industry, and deeply affected the energy strategy pattern of various countries. China's recoverable shale oil reserves are 397.4×10^8 t, accounting for 7% of the world's total reserves, which has a great potential for development [1, 2]. The poor physical properties of reservoirs in shale reservoirs, strong non-homogeneity, thin reservoir throats, and many ineffective pores lead to low primary recovery [3, 4], and effective improvement of shale reservoirs recovery is of great significance to the stable and increased production of crude oil in China [5]. CO₂ enhanced recovery technology (throughput) has become an important method of increasing production [6-8], which realizes the production of crude oil while the CO₂ geological burial [9, 10], which is a key technology in CCUS, so it is of great significance to carry out the research on the mechanism of CO₂ oil drive for shale reservoirs. Gao Shusheng [11, 12] and others believe that CO₂ in supercritical state can extract the light components

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of crude oil or vaporize them, reduce the interfacial tension of crude oil, so as to realize the high efficiency of CO₂ and crude oil mixed-phase driving, the higher the driving pressure, the more obvious the phenomenon of mixed-phase between the two, and the better the effect of oil driving. Some studies believe that in the process of CO₂ oil driving, when the dissolved amount of CO₂ in crude oil reaches a certain concentration, the asphaltene in crude oil will appear deposition phenomenon [13, 14], resulting in formation pore blockage, permeability and porosity decrease, the stronger the reservoir inhomogeneity, the more obvious the blockage phenomenon, and the increase of the driving pressure will lead to the aggravation of the blockage situation [15]. Zhou Tuo, Liu Xuewei et al. [16], used a combination of fine tube experiments, component analysis experiments, microscopic simulation experiments, a variety of experimental methods, proved that in the crude oil contains a large number of colloidal, asphaltene and aromatic hydrocarbons, with the fluid pressure increases, aromatic hydrocarbons rapid extraction, resulting in rapid precipitation of colloidal asphaltene, resulting in a serious blockage of the core. In the process of CO₂ injection to improve recovery, the problem of reservoir clogging has become a key influence factor for the application of this technology in engineering. The crude oil of a reservoir in Xinjiang is characterized by high content of gelatinous and saturated hydrocarbon, and the clogging phenomenon may occur in the field application of CO₂ enhanced recovery technology, and the clogging mechanism is unknown. For this reason, this paper designs a long core CO₂ driveback experiment, a crude oil whole component detection experiment and a family component analysis experiment to investigate the clogging mechanism of the reservoir in the process of CO₂ enhanced recovery.

2. Research idea and experimental design

2.1. Experimental objectives and design ideas

In this study, for the crude oil of an oil field in Xinjiang, we designed a long core CO₂ drive experiment and component analysis experiment. Combined with the measured pressure at the bottom of the well in the field, we carried out long core CO₂ drive experiments under five sets of constant drive pressure conditions, namely, 10 MPa, 20 MPa, 25 MPa, 30 MPa, and 40 MPa, and the CO₂ drive experiments were able to obtain the dynamic process of the change of the experimental data (such as the import and export pressure, liquid production, gas production, etc.) and crude oil samples produced at different stages, under stable pressure conditions, fluid seepage velocity and core seepage capacity changes are synchronized, the seepage velocity under constant pressure conditions can quantitatively respond to the seepage capacity of the core, through the analysis and processing of the five groups of experimental data, we can get the change of the seepage capacity of the crude oil in the process of CO₂ drive under different conditions of the pressure of different drive, so as to The change process of crude oil seepage capacity during CO₂ replacement under different replacement pressure conditions can be obtained by analyzing and processing the five sets of experimental data, so as to judge the conditions and degree of core blockage. Through the crude oil samples obtained in the process of CO₂ replacement experiments to carry out crude oil family component detection experiments and crude oil whole component detection, we can get the changes in the content of colloidal, asphaltene, saturated hydrocarbons, aromatic hydrocarbons in crude oil and the change rule of the content of the whole component (light, medium and heavy crude oil), to explore the clogging mechanism in the process of CO₂ replacement, and to provide theoretical basis for the rational application of CO₂ oil recovery technology in the field.

2.2. Experimental design for flow evaluation of CO₂ drive plugging

The experimental system consists of thermostat box, pressure sensor, core holder, ring pressure pump, high pressure displacement pump, pressure return valve, liquid, gas metering device (**Figure 1**): thermostat box can be the core holder, high pressure displacement pump heating to the formation temperature (constant temperature to 70°C); pressure sensor can measure, display the experimental system real-time imports of displacement pressure; core holder, ring pressure pump can be the core fixed, wrapped to ensure that the displacement process is axial displacement; high pressure displacement pump consists of crude oil intermediate container and gas intermediate container can control the experimental system displacement fluid medium and import pressure; pressure return valve can control the experimental system export pressure to maintain the Replacement process is axial replacement; high-pressure replacement pump consists of crude oil intermediate container and gas intermediate container, which can control the experimental system of replacement of fluid media and import pressure; back pressure valve can control the export pressure of the experimental system to maintain the import and export pressure difference in a stable state; liquid,

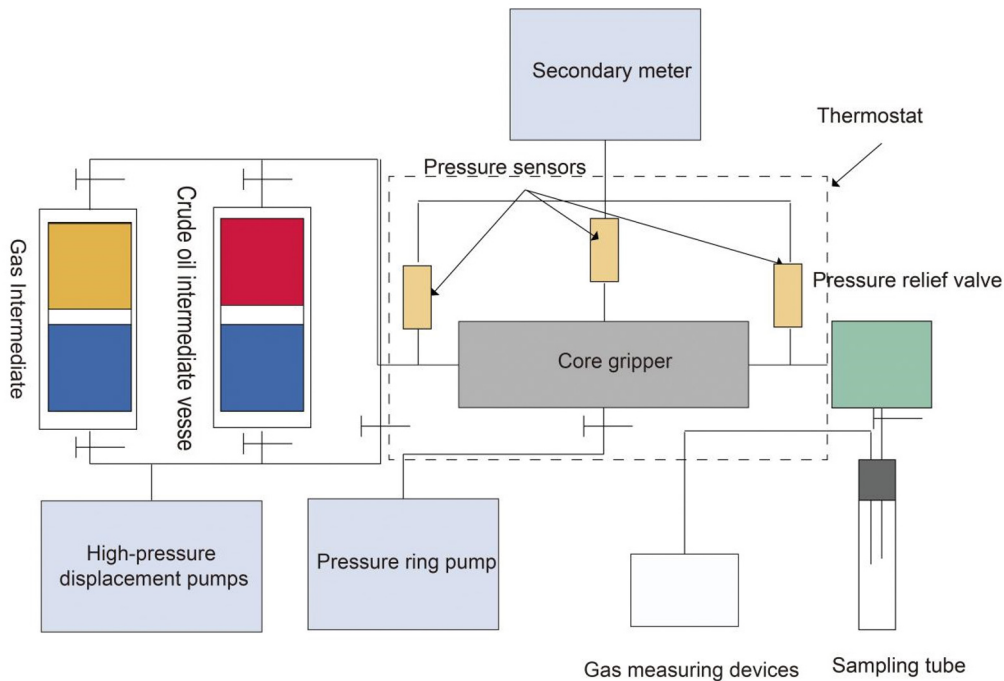


Fig. 1. Schematic diagram of the experimental flow of the CO₂ drive-off experimental setup

gas metering device can obtain real-time production of liquids, gas and calculate the rate of its output, which is connected to the sampling tube and mining outlet is placed in a closed container, can read the real-time liquid production When the experimental system starts to produce gas, the gas enters into the gas metering device through the sampling test tube, and the volume of water discharged by the gas represents the volume of gas production. The whole experimental process is as follows:

- 1) Drying the core and measuring core parameters such as porosity and permeability;
- 2) Vacuum the core to saturate the formation water;
- 3) Place the core into the core clamping device, load the ring pressure to 10MPa, empty the experimental process with water, and then carry out the single-phase water-driven displacement process to empty the air in the process, so that the process is filled with formation water;
- 4) Fill the middle container of crude oil with the configured crude oil, and use the crude oil to remove the water in the pipeline at the front end of the core, and then use the configured crude oil to replace the water in the core, and at the same time, measure the amount of water that has been driven out. Replacement is continuous until no water is produced;
- 5) Increase the annular pressure to the annular pressure set by the experimental pressure of the drive-off, and set the pressure of the pressure return device to a value above the experimental drive-off pressure;
- 6) Close the other valves, open the CO₂ intermediate container valve, open the outlet valve, set the displacement pump to constant-pressure mode, and start pressurizing the CO₂. When the inlet and outlet pressures are maintained to this top pressure, set the pressure return device pressure to the experimental set back pressure value, and then start the experiment and timing;

2.3. Experimental design of CO₂ drive plugging evaluation component

In order to deepen the understanding of the blockage mechanism, it is necessary to carry out component detection on the crude oil samples produced in the process of replacement, by analyzing the changing law of crude oil components in different replacement stages, analyzing the interaction between CO₂ and crude oil in the process of CO₂ replacement, and clarifying the reasons for the formation of the blockage in the process of replacement, this paper carries out the detection of the family composition of the crude oil samples and the detection of the heavy hydrocarbon components, respectively.

Table 1. Basic parameters of the core

Core number	Lengths (cm)	Calibre (cm)	Permeability (mD)	Saturated volume (mL)	Porosity volumetric (mL)	Porosity (%)
1	18.8	2.5	0.56	9.63	9.63	10.9
2	18.8	2.5	0.67	9.63	9.63	10.9
3	19	2.5	0.62	9.63	9.63	10.4
4	18.5	2.5	0.64	9.19	9.19	10.1
5	16.4	2.5	0.51	9.36	9.36	10.6

Crude oil family composition testing using chromatographic column instrument, using SY/T 5119-2008 reference standard [17]. Through the crude oil family composition detection, the content of asphaltene, saturated hydrocarbons, aromatic hydrocarbons and colloidal oil samples can be measured at different stages of displacement, and the changing rule of crude oil family composition content can be further analyzed to provide a basis for analyzing the core blockage.

Crude oil heavy hydrocarbon component detection using Agilent 7890A gas chromatograph, using SY/T 5779-2008 reference standard [18]. Through the detection of heavy hydrocarbon components of crude oil, we can measure the content of light, medium and heavy components of crude oil, further analyze the change rule, and clarify the characteristics of the differentiation process of crude oil components.

2.4. Core samples and fluids used in the experiments

Literature 16 uses the thin tube experiment to investigate the mechanism of reservoir plugging, the long thin tube is filled by quartz sand with a particle size of 96-109 μm , the nature of the filling medium determines that the pore type of the filled long thin tube is relatively single, homogeneous, and the porosity is as high as 36%, and the experimental conclusions obtained by using it as a research object are not applicable to shale reservoirs with complex pore structure and strong non-homogeneous properties. For this reason, this paper chooses the long core for CO_2 replacement experiment, the long core can increase the transportation distance of crude oil in the core, reduce the possibility of the clogging material being driven out of the core directly under the high replacement pressure difference, and better reflect the clogging situation inside the core. The crude oil used in CO_2 replacement experiment is the compounded crude oil with kerosene compounded to 12cP after the on-site degassed crude oil is dewatered at a high temperature. The core is from an oil field in Xinjiang, the porosity of the core is 10-11%, the permeability is 0.5-0.7 mD, the core diameter is 25 mm, the length is about 200 mm, the specific core parameters are shown in **Table 1**.

3. Experimental results and discussion

3.1. Plugging process and evaluation of CO_2 drive plugging of a crude oil in Xinjiang

3.1.1. Analysis of replacement results

Under the condition of constant-pressure driving force, the extraction velocity of crude oil can reflect the seepage capacity of the core to a certain extent, and in order to exclude the influence of unstable inlet and outlet pressures on the experimental results in the experimental process, the seepage capacity of the core is quantified by the driving velocity V_1 under the unit pressure difference:

$$V_1 = \frac{V}{\Delta P},$$

where V is the measured crude oil extraction rate; ΔP is the pressure difference between the inlet and outlet of the experimental system.

Figure 2 shows the oil recovery velocity curves per unit differential pressure under five groups of different replacement pressures, in which the oil recovery velocity per unit differential pressure represents the seepage capacity of the core. As seen in Fig. 2, under the condition of lower replacement pressure, the seepage capacity of the core shows a rising trend with the increase of replacement pressure, and the seepage capacity of the core decreases obviously when the replacement pressure reaches 25 MPa.

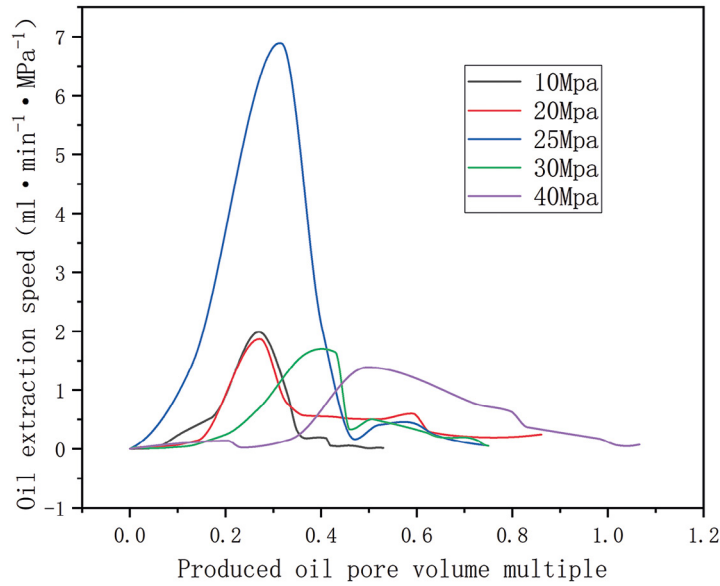


Fig. 2. Production rate curve under unit pressure difference

Figure 3 shows the gas recovery rate curves under five different replacement pressures. As seen in Fig. 3, under the low replacement pressure conditions of 10 MPa, 20 MPa and 25 MPa, the gas recovery rate curve after gas breakthrough is relatively smooth, steadily rising, and finally tends to stabilize; under the high replacement pressure conditions of 30 MPa and 40 MPa, the gas recovery rate curve after gas breakthrough is jagged, with large fluctuations in the recovery rate, which is unable to maintain a relatively stable state. maintained in a relatively stable state.

Comprehensive analysis of the two sets of experimental data, compared with the low replacement pressure, when the replacement pressure is greater than 25 MPa, the two sets of curves with the trend of replacement pressure changes appear inflection point, which is mainly manifested in the decline of the seepage capacity of the core, the fluctuation of the gas recovery rate, and the core blockage phenomenon. It is initially believed that the high replacement pressure makes certain components of crude oil deposited in the pore, and under the impact of CO₂, the deposited components are continuously transported and accumulated in

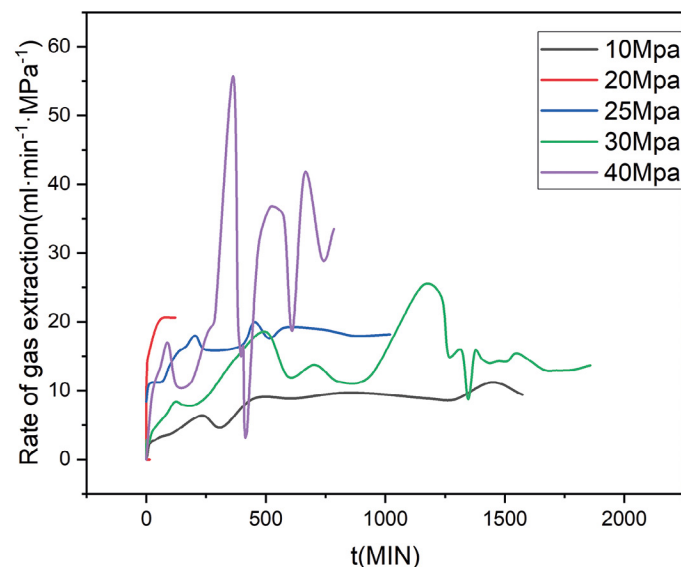


Fig. 3. Gas production speed curve under unit pressure difference

Table 2. Percentage of additional resistance to CO₂ drive plugging

Serial number	Outlet pressure (MPa)	Peak flow rate (mL/min·MPa)	Whether additional resistance is generated	Percentage of additional resistance
1	10	0.039	No	–
2	20	0.038	No	–
3	25	0.1	No	–
4	30	0.033	Yes	67%
5	40	0.028	Yes	72%

the pore, which leads to fluctuation of gas extraction rate, and eventually the accumulated components lead to blockage of the pore, resulting in the decline of seepage capacity of the core.

3.1.2. Evaluation of CO₂ replacement blockage

Through the change process of core seepage capacity during the replacement process, we can judge whether the core is clogged or not and the minimum replacement pressure that leads to clogging. In order to quantify the degree of core clogging, this paper designs the additional resistance ratio parameter (this formula applies to the core after clogging).

$$F = \frac{V' - V'_1}{V'}$$

where V'_1 is the peak value of V_1 under the current replacement pressure, which represents the maximum seepage capacity of the core under the current replacement pressure conditions, and its calculation of the additional resistance ratio can reflect more intuitively the decline in seepage capacity after the core clogging, and V' is the maximum value of V'_1 under the five groups of replacement pressure conditions.

Table 2 shows the additional resistance ratio of CO₂ replacement plugging. From Table 2 it can be seen that no additional resistance will be generated before the core is plugged, and when the core is plugged, the additional resistance ratio will increase with the increase of replacement pressure, and under the condition of 40 MPa replacement pressure, the additional resistance to seepage is as high as 72%, which seriously reduces the seepage capacity of the core. Therefore, in the production process, the replacement pressure should be strictly controlled, and the additional resistance ratio should be controlled within a reasonable range, so as to maximize the benefit of CO₂ oil recovery.

3.2. Analysis of CO₂ drive plugging mechanism

In order to deepen the mechanism of CO₂ drive plugging, several groups of crude oil samples under different drive pressure are analyzed by crude oil family component detection and full component detection. **Figure 4** show the change of crude oil family component content under 25 MPa and 40 MPa drive conditions, respectively, and the numbering represents the oil samples extracted in different experimental phases, which are compiled according to the order of the drive process, and the termination numbering is the oil samples extracted at the time of stopping the drive, 0 represents the component content of the original crude oil sample. As can be seen in Fig. 4, in the process of carbon dioxide replacement of crude oil, the content of saturated hydrocarbons in the extracted oil samples continued to rise, and the content of colloidal, asphaltene and aromatic hydrocarbons continued to decrease, among which the reduction of colloidal and asphaltene was the most obvious, compared with the original samples, under the condition of 25 MPa replacement, the content of colloidal and asphaltene fractions decreased by 30%, and when the pressure of replacement reached 40 MPa, the concentration of colloidal and asphaltene decreased by as much as 37%. When the driving pressure reaches 40 MPa, the concentration of gel and asphaltene decreases as high as 37% and 38%, indicating that the high driving pressure increases the decrease of gel and asphaltene content in the extracted crude oil, resulting in the extreme increase of asphaltene and gel content in the core, and the viscosity of crude oil increases dramatically, which reduces the flow ability of crude oil, and at the same time, the deposition process of asphaltene is accelerated and aggravated, which finally causes the core blockage.

Figure 5 show the changes of the whole hydrocarbon content of crude oil under the conditions of 25 MPa and 40 MPa, respectively, and the numbers represent the pore volume multiples of the extracted oil, which are compiled in accordance with

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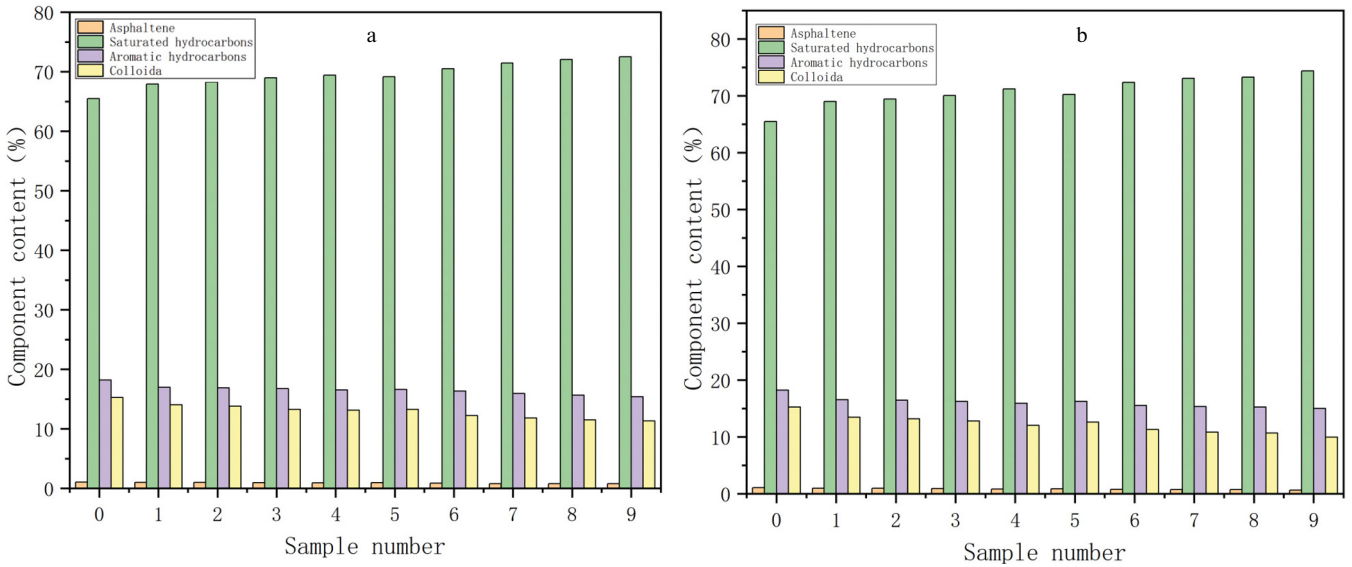


Fig. 4. Changes in group composition content of crude oil under 25 MPa (a) and 40 MPa (b)

the order of the replacement process 0 represents the component content of the original crude oil sample, and the termination number is the sample of the extracted oil at the time of stopping the replacement. As can be seen in Fig. 5 under the extraction effect of CO₂ on crude oil, in the interval of C₁₀–C₁₆ light component, the content of light component of crude oil increased at the beginning of replacement, in the interval of C₁₇–C₂₈ medium component, the content of medium component of crude oil increased slowly with the process of replacement, and in the interval of C₃₆–C₃₉ heavy component, the content of the output was decreasing, which indicates that the extraction process of light component of crude oil by CO₂ is rapid and the extraction process of light component is rapid, and the extraction process of medium component is rapid and the extraction process of light component is rapid. This indicates that at the beginning of the drive, CO₂ extracts the light components of crude oil rapidly, and extracts the medium components more slowly. With the slow progress of the drive process, the crude oil in the core gradually shows the phenomenon of component differentiation during the extraction process, and the heavy components start to pile up in the core. Combined with the analysis of the two groups of component content

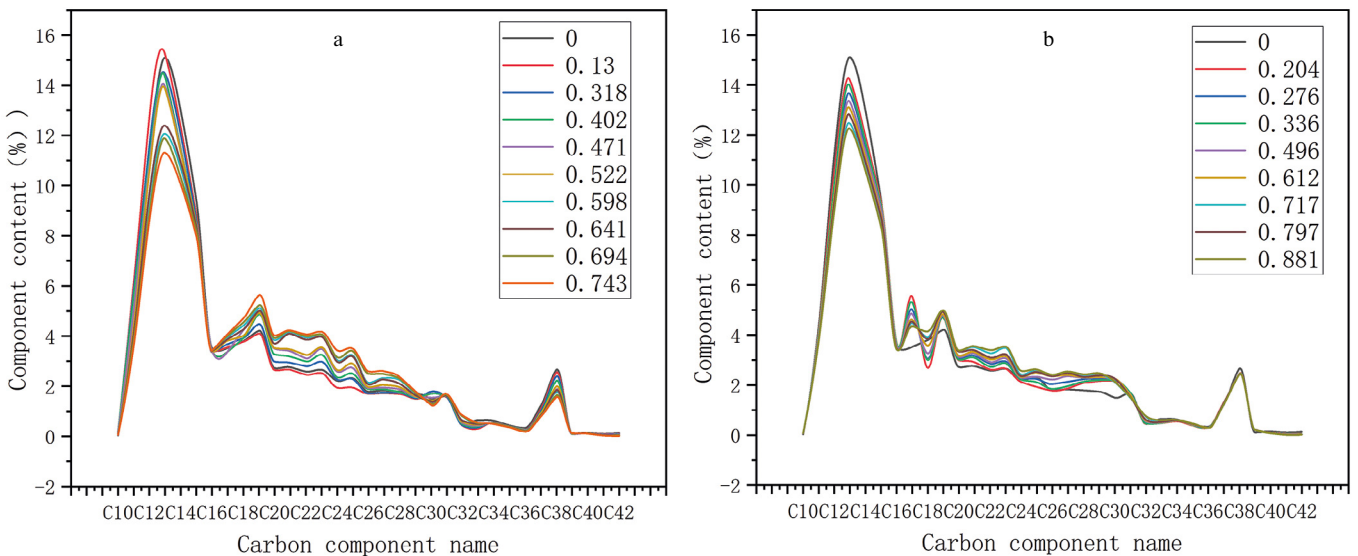


Fig. 5. Changes in all components of crude oil under 25 MPa (a) and 40 MPa (b)

curves, C_{10} – C_{16} light component interval, there is no significant difference between the two groups of curves, indicating that the increase in replacement pressure did not significantly enhance the extraction ability of CO_2 on the light component of the crude oil, C_{17} – C_{28} medium component interval, there is a significant difference between the two curves at C_{17} , mainly manifested in the replacement of the pressure of 25 MPa (before the core is blocked), the C_{17} component content in the replacement of 25 MPa, the C_{17} component content is significantly higher than that in the replacement of 25 MPa, the C_{17} component content is significantly lower than the C_{17} component content in the core. When the replacement pressure reaches 40 MPa, the content of C_{17} component rises sharply in the pre-excavation period, and its rise is as high as 57%, indicating that the high replacement pressure makes the extraction ability of CO_2 on the middle-quality components become wider in a small range, and the phenomenon of component differentiation is aggravated, which ultimately results in the clogging of the core.

3.3 Comparative analysis of CO_2 replacement clogging mechanism

Literature [16] selected Jilin oilfield crude oil to carry out clogging research, high replacement pressure, this paper and literature [16] experimental core are clogging phenomenon, literature 16 in the core of the serious clogging phenomenon, seepage ability down to a lower level, as seen in Fig.2, this paper, the core of the slight clogging phenomenon, but still has a certain degree of seepage ability. In order to explore the reasons, the following comparative analysis is carried out. **Figure 6** show the change curves of the whole component content of crude oil in the literature [16]. Fig. 6 show the change curves of the whole component content of crude oil in the literature [16], in the process of CO_2 oil expulsion, the peak value of the output crude oil component moves from C_9 to C_{14} , and with the increase of the expulsion pressure, the extraction ability of CO_2 on the light component of crude oil becomes stronger, the peak value of the component moves to a wider range, and the fluid component is seriously differentiated. From the results of this paper, with the increase of replacement pressure, the extraction ability of CO_2 on the light component has no obvious change, too high replacement pressure will make the extraction range of CO_2 on the medium component become wider, the peak of the fluid component will not be shifted, and the phenomenon of component differentiation is not serious. Combined with the two groups of experiments using crude oil components content analysis, on the one hand, the literature [16] using crude oil samples with high asphaltene content, when the crude oil content of asphaltene is high, there will be a large number of asphaltene deposition in the pore channel during the process of expulsion, more likely to cause the core clogging. On the other hand, this paper uses crude oil samples with relatively high saturated hydrocarbon content, saturated hydrocarbons and colloidal, asphaltene to form a more stable system, CO_2 on the light components of the crude oil extraction is not strong, the degree of differentiation of the crude oil components is low, the core is not easy to be completely blocked, in the process of CO_2 oil expulsion, the peak value of the output crude oil component moves from C_9 to C_{14} , and with the increase of

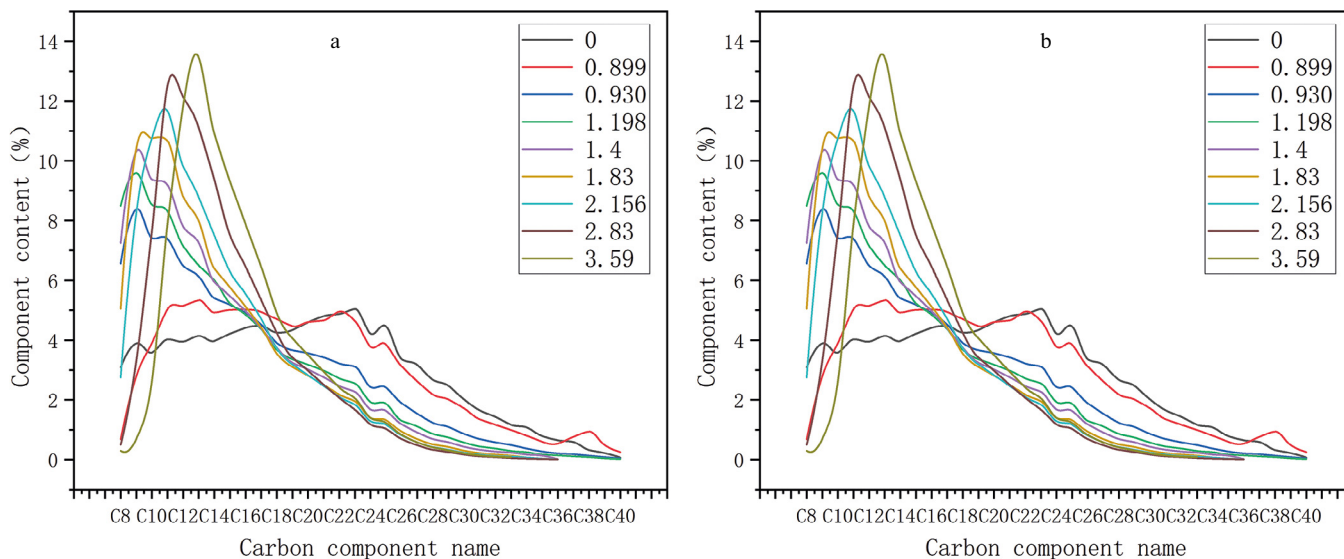


Fig. 6. Changes in Pseudo Component Content under 18 MPa (a) and 25 MPa (b) [16]

the expulsion pressure, the extraction ability of CO₂ on the light component of crude oil becomes stronger, the peak value of the component moves to a wider range, and the fluid component is seriously differentiated. From the results of this paper, with the increase of replacement pressure, the extraction ability of CO₂ on the light component has no obvious change, too high replacement pressure will make the extraction range of CO₂ on the medium component become wider, the peak of the fluid component will not be shifted, and the phenomenon of component differentiation is not serious. Combined with the two groups of experiments using crude oil components content analysis, on the one hand, the literature [16] using crude oil samples with high asphaltene content, when the crude oil content of asphaltene is high, there will be a large number of asphaltene deposition in the pore channel during the process of expulsion, more likely to cause the core clogging. On the other hand, this paper uses crude oil samples with relatively high saturated hydrocarbon content, saturated hydrocarbons and colloidal, asphaltene to form a more stable system, CO₂ on the light components of the crude oil extraction is not strong, the degree of differentiation of the crude oil components is low, the core is not easy to be completely blocked.

4. Conclusion

In this study, we established the experimental flow, experimental method and analysis method for CO₂ drive plugging evaluation of long cores, analyzed the mechanism of CO₂ drive plugging in Xinjiang crude oil, and compared it with the results of plugging evaluation of crude oil in an oil field in Jilin, and obtained the following understanding.

1. In the process of CO₂ replacement, the phenomenon of component differentiation caused by the extraction of crude oil by CO₂, as well as the rise in the content of colloid and asphaltene in crude oil lead to the rise in the viscosity of crude oil and the deposition of asphaltene, so that the core has the risk of blockage in the process of replacement.

2. The crude oil of an oilfield in Xinjiang is heavy crude oil, and the experiment shows that heavy crude oil has higher risk of clogging, in which the content of asphaltene and saturated hydrocarbons is the dominant factor leading to clogging of cores, and high asphaltene and low saturated hydrocarbons are more likely to clog the cores during the replacement process.

3. Before production, the feasibility of CO₂ oil recovery technology in this field should be judged according to the content of crude oil components in the field and the characteristics of component differentiation during the CO₂ displacement experiment.

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