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EXPERIMENTAL STUDY ESTUDIO ON IMMISCIBLE AND MISCIBLE DYNAMIC CHARACTERISTICS OF CO₂ AND CRUDE OIL IN VISUAL SLIM TUBE

ESTUDIO EXPERIMENTAL SOBRE LAS CARACTERÍSTICAS DINÁMICAS INMISCIBLES Y MISCIBLES DEL CO₂ Y EL PETRÓLEO CRUDO EN UN TUBO VISUAL DELGADO

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ABSTRACT

CO₂ flooding for oil recovery is a dynamic process that requires further investigation of oil-gas interface change characteristics, interfacial mass transfer processes, and oil-gas composition variation during both immiscible and miscible displacement. Understanding these factors is crucial for better comprehending their impact on CO₂-enhanced oil recovery (EOR). This research used a jointly developed CO₂ miscible visual flooding experimental apparatus to study the horizontal dynamic characteristics of CO₂ and crude oil under different pressures and flow rates in visual slim tube. At 10 MPa, the stratification results of CO₂ and crude oil indicate that the experiment is immiscible flooding. The contact angle (7.9°) between the two phases of CO₂ and crude oil at the flow rate of 15 cm/min is larger than that (5.2°) at 1.5 cm/min, and the grey scale of CO₂ increases at 100 cm/min. The quantity, individual content, and shape of the light and medium hydrocarbon components condensed on the inner wall of the tube vary with different flow rates. At 15 MPa, the appearance of the CO₂ and crude oil transition interval proves that the experiment is miscible flooding. At different flow rates, the inclination angle and distribution of black stripes vary. The whole transition interval is divided into 6 intervals, and the transition interval lengthens with increasing fluid velocity. The experiments visually demonstrate the occurrence of the miscible phase, and identify experimental pressure and fluid flow rate as key factors influencing the miscibility of CO_2 and crude oil.

RESUMEN

La inundación de CO₂ para la recuperación de petróleo es un proceso dinámico que requiere más investigación sobre las características de cambio de la interfaz petróleo-gas, los procesos de transferencia de masa interfacial y la variación de la composición petróleogas durante el desplazamiento tanto inmiscible como miscible. Comprender estos factores es crucial para comprender mejor su impacto en la recuperación de petróleo mejorada con CO₂ (EOR). Esta investigación adopta un aparato experimental de inundación visual miscible con CO₂ desarrollado conjuntamente para estudiar las características dinámicas horizontales del CO, y el petróleo crudo bajo diferentes presiones y caudales en un tubo visual delgado. A 10 MPa, los resultados de la estratificación del CO₂ y del petróleo crudo indican que el experimento es una inundación inmiscible. El ángulo de contacto $(7,9^{\circ})$ entre las dos fases de CO_2 y el petróleo crudo a un caudal de 15 cm/min es mayor que el $(5,2^{\circ})$ a 1,5 cm/ min, y la escala de grises del CO₂ aumenta a 100 cm/min. La cantidad, el contenido individual y la forma de los componentes de hidrocarburos ligeros y medios condensados en la pared interior del tubo varían con diferentes caudales. A 15 MPa, la aparición del intervalo de transición de CO₂ y petróleo crudo demuestra que el experimento es una inundación miscible. A diferentes caudales, el ángulo de inclinación y la distribución de las franjas negras son diferentes. Todo el intervalo de transición se divide en 6 intervalos y el intervalo de transición se alarga al aumentar la velocidad del fluido. Los experimentos demuestran visualmente la aparición de la fase miscible e identifican la presión experimental y el caudal de fluido como factores clave que influyen en la miscibilidad del CO_2 y el petróleo crudo.

AFFILIATION

KEYWORDS / PALABRAS CLAVE

 $\rm CO_2$ and Crude Oil | Immiscible Flooding | Miscible Flooding | Dynamic Characteristics | Visual Slim Tube.

CO₂ y Petróleo Crudo | Inundaciones inmiscibles | Inundaciones miscibles | Características Dinámicas | Tubo visual Delgado ¹ The Karamay Branch of National Key Laboratory of Petroleum Resources and Engineering, China University of Petroleum (Beijing) at Karamay, Karamay, China.
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 CO_2 flooding, a relatively mature technology, is also considered one of the most promising methods for Enhanced Oil Recovery (EOR) (Brattekås & Seright, 2023; Prakash et al., 2024; Xiao et al., 2023). Currently there are hundreds of experimental projects and commercial projects of CO_2 flooding in the world (Davoodi et al., 2024; Li et al., 2022). For CO_2 flooding in China, the scale effect has not yet formed. The main reasons are:. First, the content of paraffin, gum, and asphaltene in the crude oil of continental oil fields is high, and the minimum miscibility pressure is high. Second, heterogeneity is significant in continental sedimentation. Third, CO₂ costs are too high due to scarce gas sources (Gür, 2022; Lin et al., 2024; Shen et al., 2022). In the past few decades, CO₂ EOR, as a continuously developing oil production technology, has conducted numerous laboratory tests, numerical simulations, and field practices to enhance oil recovery. These efforts include refining reservoir characterization, enhancing fluid flow control capabilities, and ensuring the consistency of the CO₂ flooding front. CO₂ EOR technology has been proven to increase oil recovery by 8% to 16% (Kumar et al., 2022; Liu, J et al., 2023; Tan et al., 2022).

In recent years, to conduct recycling of CO_2 , the international community has proposed Carbon Capture, Utilization and Storage (CCUS) technology based on Carbon Capture and Storage (CCS) to make the process more economical and practical (Gallo et al., 2020; Gao et al., 2023; Hong, 2022; Huang et al., 2022). The proposal of the carbon peak and carbon neutrality poses a new challenge to the development of the carbon market in China. CO_2 flooding and storage technology not only improves oil recovery by CO_2 flooding but also realizes the geological storage of CO_2 . It is a technology with both economic and social benefits as well as the most effective way to

reduce greenhouse gas emissions under the current economic and technological conditions (Manigandan et al., 2023; Raihan et al., 2022). For example, Liu, Y et al. (2022) proposed dimethyl ether and propanol as efficient agents in assisting conventional CO_2 flooding for oil recovery while increasing CO_2 storage in reservoirs (Liu, Y et al., 2022).

In the EOR technology of light and medium reservoirs, favorable reservoir conditions are one of the key factors for the success of CO_2 flooding (Fu et al., 2023; Wang et al., 2022). If the actual reservoir pressure is lower than the minimum miscible pressure of CO_2 and crude oil, CO_2 flooding can only be immiscible displacement. Conversely, CO_2 flooding can be miscible displacement (Mirazimi et al., 2022). CO_2 miscible flooding is an effective and economical method to improve oil recovery, while CO_2 immiscible flooding is prone to premature CO_2 breakthrough in light oil reservoirs, resulting in lower oil recovery (Chen, Z. et al., 2022; Jiang et al., 2022). Furthermore, the gravity overlap between CO_2 and crude oil due to density difference will also affect the oil recovery (Yang et al., 2024).

On the contrary, the majority of existing research on CO_2 -enhanced oil recovery relies on core plug flooding experiments, which yield limited data. The microscopic visualization slim tube model, however, can provide an intuitive characterization of the microscopic seepage mechanism of oil and gas (Yi et al., 2023). In this paper, the CO_2 miscible visual flooding experimental device is used to study the horizontal contact characteristics and dynamic changes during the process of CO_2 flooding crude oil in the visual slim tube under different experimental pressures and fluid flow rates and analyze the main factors affecting the CO_2 and crude oil the miscibility.

2 EXPERIMENTAL DEVELOPMENT

EQUIPMENT AND MATERIALS

The experimental equipment is a jointly developed CO_2 miscible visual flooding experimental device shown in Figure 1, including

a visual slim tube (two front and rear transparent visual windows), and a sealed container surrounding it. Images and experimental data use a camera pan-tilt and computer software tracking system to complete image shooting and data collection. The backlight adopts a special lighting set for photography, with adjustable brightness and soft light. The experimental fluid is ground-degassing oil with a density of 0.77 g/cm³ and a viscosity of 12.54 mPa.s. The purity of CO₂ used in the experiment is 99.9%. The experimental temperature is 40 °C, and CO₂ is in supercritical state under 10 MPa and/or 15 MPa.

METHODS AND STEPS

(1) Set the temperature of the incubator at $40 \,^{\circ}$ C, clean the visible slim tube and related

tubes with petroleum ether, and dry them with CO_2 . (2) Inject grounddegassing oil from the output end of the visual module to the injection end, and adjust the experimental pressure (10 MPa or 15 MPa). (3) Open the CO_2 valve from the injection end of the visual module, and carry out the oil displacement experiment. (4) Adjust the injection



Figure 1. Schematic diagram of the visual experimental set-up

flow rate (1.5 cm/min, 15 cm/min or 100 cm/min), and maintain constant pressure. (5) Observe the experimental phenomena and record the relevant data. (6) Discharge the fluid in the visual slim tube and repeat steps (1-6).

3. RESULTS

PRESSURE 10 MPa

FLOW RATE 1.5 cm/min

As shown in Figure 2, when the flow rate of CO₂ and crude oil is 1.5 cm/min, it can be observed that there is a certain contact angle (5.2°) between the two phases of CO₂ and crude oil that is small. Throughout the flow process, the contact surface characteristics between CO₂ and crude oil are evident, with relatively moderate changes. As time passes, the color of CO_2 in the upper part of the visible slim tube becomes light brownish red and the content gradually increases. The color of the crude oil in the lower part becomes lighter and the content gradually decreases. At t_4 =2.489 min, CO₂ occupies the upper half of the visible slim tube, and crude oil occupies the lower half of the visible slim tube, with a distribution of the two in half. With the flow of CO_2 , the amount of crude oil at the bottom decreases until it is depleted. After a long time, there is condensation of light to medium hydrocarbon components on the inner wall of the tube, with a large quantity but small individual content, appearing as small dot-like formations.



Figure 2. Characteristics of $\rm CO_2$ and crude oil at the flow rate of $1.5~\rm cm/min$

FLOW RATE 15 cm/min

Figure 3 shows that CO₂ and crude oil have a large contact angle (7.9°) that changes significantly at a flow rate of 15 cm/min. The change characteristics of the contact surface between CO₂ and crude oil are apparent, and the increase rate of CO₂ content in the upper part and the decrease rate of crude oil content in the lower part are greater. At t₄=0.356 min, CO₂ occupies a significant portion of the visible slim tube of about 4/5, while crude oil occupies a fraction of the visible slim tube of about 1/5. After a long time conducting the experiment, light and medium hydrocarbon components are condensed on the inner wall of the tube, and the quantities are minor. The individual content is large, and the individual appears in the shape of large speckles.

FLOW RATE 100 cm/min

As shown in Figure 4, $\rm CO_2$ and crude oil phases have no contact angle at a high flow rate (100 cm/min). At $t_1\!=\!0.006$ min, the crude



Figure 3. Characteristics of CO₂ and crude oil at the flow rate of 15 cm/min

oil appears with a deep reddish-brown color, without CO₂. At t₂=0.013 min, the crude oil has a reddish-brown color with a small amount of CO₂. At t₃=0.027 min, it is dark gray CO₂ containing a significant quantity of light and medium hydrocarbon components. At t₄=0.051 min, yellow-gray CO₂ contains a small amount of light and medium hydrocarbon components. At t₅=0.133 min, it is large circular speckles of light and medium hydrocarbon components condensed out.



PRESSURE 15 MPa

FLOW RATE 1.5 cm/min

Under the pressure condition of 15 MPa, the experiment of CO₂ displacement of crude oil is conducted at the flow rate of 1.5 cm/ min. It is noticed that during the flow process, no contact inclination is seen between the two phases of CO_2 and crude oil (no contact angle variation characteristics), but there is a transition interval between CO_2 and crude oil (Figure 5). From the injection end to the output end, the order of transition intervals observed is $(1) CO_2$ interval containing small speckles of light and medium hydrocarbon components, O CO₂ interval containing a small amount of light and medium hydrocarbon components, (3) CO₂ interval containing a large amount of light and medium hydrocarbon components, 4intermediate interval where CO_2 and crude oil are mixed, (5) crude oil interval containing a large amount of CO_2 , (6) crude oil interval containing a small amount of CO_2 . In the transition interval, black stripes can be observed. In the experiment, the direction of fluid flow and black stripes are both from left to right, and the inclination angle (20°) of black stripes is downwards and relatively small. 3.2.2. Flow Rate 15 cm/min





Figure 5. Characteristics of CO_2 and crude oil at the flow rate of 1.5 cm/min



Figure 6. Characteristics of CO_2 and crude oil at the flow rate of 15 cm/min



Figure 7. Characteristics of CO₂ and crude oil at the flow rate of 100 cm/min

Figure 6 shows that the transition interval between CO_2 and crude oil is observed in the flow process at the flow rate of 15 cm/min. Different from the flow rate of 1.5 cm/min experiment, the leftmost side of the transition interval is the CO_2 interval containing large speckles of light and medium hydrocarbon components. The direction of the black stripes appears from left to right, and the dip angle (65°) is downwards and large.

FLOW RATE 100 cm/min

As shown in Figure 7, in the 15 MPa and 100 cm/min displacement experiments, the transition interval between CO_2 and crude oil can also be observed. The difference from the above experiments is that the leftmost side of the transition interval is the CO_2 interval containing the light and medium hydrocarbon components of the large circular speckles. The direction of the black stripes is from left to right and evenly distributed horizontally.

4. RESULTS ANALYSIS

PRESSURE 10 MPa

During the 10 MPa experiments, at flow rates of 1.5 cm/min and 15 cm/min, a distinct contact angle between CO_2 and crude oil is observed, and the angle is different (Table 1). The stratification of CO_2 and crude oil occurs during the flow process, meaning that the experiment is an immiscible experiment. Due to the influence of gravity differences, stratification occurs between CO_2 and crude oil,

which forms a fingering phenomenon in a visible slim tube and forms a certain contact angle. The greater flow rate of the experimental fluid, the larger the contact angle. Specifically, the contact angle changes from 5.2° to 7.9° when the fluid flow rate changes from 1.5 cm/min to 15 cm/min.

In the experiments with the flow rate of 1.5 cm/min and 15 cm/min, due to the extraction of crude oil by CO_2 , the light and medium hydrocarbon components in the crude oil are extracted into the CO_2 , resulting in a light brownish-red color of the CO_2 . In the experiment with the flow rate of 100 cm/min, due to the high fluid flow rate, the color of CO_2 containing a large amount of light and medium hydrocarbon components is dark gray, while when containing a small amount of light and medium hydrocarbon components it is yellow-gray. The color of CO_2 is grayish due to the low transmittance of light when it passes through high-speed fluids.

In the later stages of the experiments with different flow rates, the quantity, individual content, and shape of the light and medium hydrocarbon components condensed on the inner wall of the visible slim tube are different. This is because different fluid flow rates have a certain effect on the extraction of CO_2 . At low flow rates, the contact area between CO_2 and crude oil changes little, and the degree of CO_2 diffusion into crude oil is limited, resulting in poor dissolution and mass transfer between CO_2 and crude oil, and the extraction effect is weak. At high flow rates, the contact area between CO_2 and the degree of CO_2 diffusion into the crude oil changes greatly, and the degree of CO_2 diffusion and mass transfer between the degree of CO_2 diffusion into the crude oil changes greatly and the degree of CO_2 diffusion and mass transfer between CO_2 and crude oil better and the extraction effect stronger.

Pressure (MPa)	Flow Rate (cm/min)	Contact Characteristics and Variation Characteristics	Miscibility	Interval
10	1.5	Stratification, Small contact angle, Small circular speckles of light and medium hydrocarbons (Large quantity, Small content)	No	No
	15	Stratification, Large contact angle, Large speckles of light medium hydrocarbons (Small quantity, Large content)	No	No
	100	No stratification, Large CO2 color gray, Large circular speckles of light medium hydrocarbons	No	No
15	1.5	Black stripes, Small inclination angle, Small circular speckles of light and medium hydrocarbons (Large quantity, Small content)	Yes	Yes
	15	Black stripes, Large inclination angle, Large speckles of light and medium hydrocarbons (Small quantity, Large content)	Yes	Yes
	100	Black stripes, Horizontal uniform distribution, Large circular speckles of light and medium hydrocarbons	Yes	Yes

Table 1. Experimental Characteristics of Visual Slim Tube

PRESSURE 15 MPa

fluid from $\rm CO_2$ component to crude oil component is formed, which is divided into 6 intervals.

Under the experimental pressure of 15 MPa, the transitional interval between CO_2 and crude oil can be observed in the displacement process with different flow rates, meaningthat the experiment is CO_2 miscible flooding. The experiment confirms that the miscibility is a dynamic process, with different fluid properties and phase intervals. Each interval is variable and forward, allowing to visualize the transitional interval between CO_2 and crude oil.

In the experiment of visual slim tube, the whole transition interval is divided into 6 intervals, based on the difference of fluid color, shape and phenomenon. In fact, the whole transition interval is uniform and continuous, and it is difficult to divide it finely at present. The slim tube used in this experiment is not a porous medium, so the miscible transition interval of CO₂ and crude oil cannot fully represent the variation characteristics in a porous medium, but it has some indicative significance.

During the experiment of different flow rates, in addition to the different morphology and distribution of condensate droplets on the tube wall, black stripes appear in the transition interval, and their inclination angles and distribution are different. When the flow rates are 1.5 cm/min and 15 cm/min, the dip angle of black stripes is small. The diffusion and mass transfer of CO_2 in crude oil are weak, and the transition interval is short. When the flow rate is 100 cm/min, the black stripes are evenly distributed horizontally. The diffusion and mass transfer of CO_2 in crude oil are strong, and the transition interval is long.

CHARACTERISTIC ANALYSIS

Compared to vertical static experiments, horizontal dynamic experiments show that under immiscible conditions, due to the gravity difference, CO_2 and crude oil are stratified during the flow process, resulting in fingering. In actual reservoir development, CO_2 breakthrough occurs in the production wells, and gas kick is formed prematurely. During the experiments, the fluid flow rates are different. Under the immiscible flooding, the contact angle between CO_2 and crude oil is different and the breakthrough time of CO_2 after gas injection is different. Under the miscible condition, the length of the transition interval between CO_2 and crude oil are different. Under the condition of 15 MPa, CO_2 and crude oil are miscible, and a transition interval of

Furthermore, there is an interphase mass transfer between CO_2 and crude oil, reducing the interfacial tension, thus effectively promoting sweep efficiency. When the injection pressure reaches the maximum value of 15 MPa, the displacement efficiency factor is further increased. The microscopic mechanism involved in CO_2 flooding is illustrated in Figure 8.







Figure 8. Microscopic oil recovery mechanism during $\rm CO_2$ flooding



APPLICATION ANALYSIS

Studying the visualization characteristics of CO₂ miscibility can enhance our understanding of the various factors influencing miscibility during the CO₂ flooding process (Liu, X. et al., 2024; Song et al., 2022). These factors include experimental pressure and fluid flow rate. By controlling and adjusting these factors, the CO₂ flooding process is optimized to enhance oil recovery. Moreover, in the actual production process, the injection rate and injection pressure of CO₂ are adjusted according to the miscible visualization characteristics of CO₂ to achieve the best results.

Studies have explored the adaptability and application prospects of CO₂ in various reservoir types, including light oil reservoirs, low permeability reservoirs, and shale reservoirs (Hou et al., 2023; Mahdaviara et al., 2022; Pal et al., 2022). By observing the miscible characteristics of CO_2 and different quality crude oils in different pore structures, the effect and potential of CO₂ displacement in different reservoirs can be evaluated, and the synergistic utilization of CO₂ displacement and storage can be explored. For example, in light oil reservoirs, higher flow rate and pressure are used to improve the dissolution and mass transfer between CO₂ and crude oil to improve oil recovery. According to pilot test findings from the Yaoyingtai Oilfield in Northeast China, the cumulative injection volume of CO₂ reached 22.6×10⁴ t, yielding effectiveness in 29 corresponding wells. This endeavor resulted in a cumulative oil increase of 1.8×10⁴ t, corresponding to a stage-enhanced recovery rate of 1.1%, with a CO₂ storage efficiency reaching 92.6% (Zhang et al., 2024). Notably, the success rate of CO_2 miscible flooding projects remains notably high, evidenced by 104 successful projects in the U.S., constituting 81.2% of the total.

 $\rm CO_2$ miscibility visualization technology can study various influencing factors in the process of $\rm CO_2$ displacement, such as impurity gas mixing, water shield barrier and pore size effect, as well as their influence mechanisms and laws on miscibility characteristics and displacement effects (Guo et al., 2022; Lei et al., 2022). By observing the concentration change of $\rm CO_2$ in the produced gas under the condition of impure $\rm CO_2$ gas, the separation effect of water shield on the oil and gas system, and the influence of pore size on the degree of miscibility, the key scientific and engineering problems in the promotion and application of $\rm CO_2$ miscible flooding technology can be better solved (Jin et al., 2023; Ma et al., 2022).

In addition, CO₂ visualization research is of great significance to the energy transition. In 2022, 30 global CCUS projects will collectively store 297.6×10⁶t CO₂. Among these, CO₂ geological storage stands out as a crucial component.(Chen et al., 2024) CO₂-enhanced oil and gas recovery technology can store approximately 14.1×10⁸t CO₂, while depleted gas reservoirs offer a capacity of about 15.3×10⁶t. Notably, the CO₂-EOR technology in Shengli Oilfield is poised to increase oil production by 127 million tons while sequestering 204 ×10⁸t CO₂.

CONCLUSIONS

o The CO_2 miscible visual flooding experimental device is used to conduct the horizontal dynamic experiment in the visual slim tube. The experimental phenomena are observed and described by changing the experimental pressure and fluid flow rate. Analysis of the horizontal contact and dynamic change characteristics of CO_2 and crude oil confirms that the main factors affecting the miscible characteristics of CO_2 and crude oil are experimental pressure and fluid flow rate.

o In the 10 MPa experiments, the stratification phenomenon occurred, which is the immiscible displacement experiment of CO_2 and crude oil. The larger the fluid flow rate (1.5 cm/min and 15 cm/min), the larger the contact angle between CO_2 and crude oil becomes (5.2° and 7.9°). At a high fluid flow rate (100 cm/min), the color gray of CO_2 intensifies. In the later stage of the experiment with different flow rates, the quantity, individual content, and shape of the light and medium hydrocarbon components condensed on the inner wall of the visible slim tube are different.

o In the 15 MPa experiments, using different flow rates, the transition interval between CO_2 and crude oil appears in the displacement process, which is the miscible displacement experiment of CO_2 and crude oil. It is confirmed that the existence of the miscible dynamic process makes the transition interval of CO_2 and crude oil visualized. And the whole transition interval is divided into 6 intervals, which have certain indicative significance. In the experiment, the inclination angle and distribution of black stripes are different. The larger the fluid flow rate, the longer the transition interval.

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