Experimental Study on Supercritical CO₂ Huff and Puff in Tight Conglomerate Reservoirs

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ABSTRACT: A tight conglomerate reservoir is a kind of unconventional reservoir with strong heterogeneity, and CO₂ injection is an economical and environmentally friendly method to enhance tight oil recovery. Supercritical CO₂ is a very promising fluid medium for unconventional reservoir development due to its gas–liquid dual properties. In this study, the production effects of supercritical CO₂ and non-supercritical CO₂ in tight conglomerate reservoirs were quantitatively analyzed by huff and puff simulation experiments conducted under reservoir conditions (formation pressure 37 MPa, temperature 89 °C). Also, the influencing factors of CO₂ huff and puff production, including injection volume, soaking time, and throughput cycles, were investigated. The results showed that supercritical CO₂ improves the recovery by 4.02% compared with non-supercritical CO₂. It could be seen that supercritical CO₂ plays a positive role in improving tight conglomerate reservoirs. The optimal injection volume, soaking time, and throughput cycles were determined to be 0.50 PV, 2 h, and 3 cycles, respectively. This paper provides an important basis for the study of supercritical CO₂ production in tight conglomerate reservoirs.

1. INTRODUCTION

A tight conglomerate oil reservoir is an indispensable unconventional resource.1 Tight oil reservoirs are widespread, mainly in the United States,2,3 Canada,4 China, and other countries. In China, the amount of tight oil resources accounts for 2/5 of recoverable oil resources, mainly distributed in Ordos, Sichuan, Songliao, Tarim, the Bohai bay, Tuha, and the Junggar Basin.5

Conventional water flooding development is not applicable to tight reservoirs due to high injection pressure, poor compatibility, etc.6 As reported by most researchers, it is difficult to carry out efficient production from the oil field with low permeability and low porosity in water flooding development.7 However, gas flooding might have a good effect on the exploitation of tight oil reservoirs, in which carbon dioxide (CO₂) plays a more important role in the development of tight oil reservoirs. Also, it has been applied in many tight oil reservoirs in the world because its huff and puff development has the advantages of less investment and quick results.5–15

CO₂ as a greenhouse gas has a great impact on the environment. The best treatment method is storage and utilization. Many peers have shown that CO₂ has a favorable effect on the development of tight oil reservoirs. Injecting CO₂ into the reservoir can not only enhance oil and gas recovery but also realize the purpose of burying CO₂ underground for a long time. This accordingly reduces greenhouse gas emissions and rationally uses resources.14–16

As reported, the critical temperature and pressure of supercritical CO₂ (scCO₂) are 31.2 °C and 7.38 MPa, respectively. When both the pressure and temperature of CO₂ exceed the critical point, CO₂ reaches a supercritical state, as shown in the red area in Figure 1. At this point, CO₂ has the dual properties of a gas and liquid, which not only has the same diffusion coefficient and low viscosity as a gas but also has the same density and solubility as a liquid. scCO₂ at this time has a significant effect on enhancing the recovery of tight oil reservoirs.17

In the past several years, a number of researchers have investigated the application of scCO₂ to oilfield development.18–20 Shang et al.21 systematically studied the influence of reservoir conditions such as pressure and reservoir physical properties on the diffusion coefficient and concentration distribution of scCO₂ and established a prediction method for the concentration field and diffusion front of scCO₂. Wei et al.22 indicated that the high injection pressure of scCO₂ into the tight bitumen layer is helpful for enhanced oil recovery but will accelerate the asphaltene precipitation in the formation.
Samara et al. used scCO₂ to study the oil-displacement mechanism of enhanced oil recovery in tight rock reservoirs and analyzed the effects of interfacial tension, wettability, diffusion, and adsorption on scCO₂ development. These works demonstrate the importance of scCO₂ for tight reservoir development.

The target reservoir is located in the Jungger Basin, northwest China. The formation pressure and temperature are 37 MPa and 89 °C, respectively. The reservoir is a tight conglomerate reservoir with strong formation heterogeneity and a permeability range of 0.05−94.8 mD. The average permeability is only 2.30 mD, and the average porosity is 9.23%. During early periods of oilfield development, water injection is not effective, so it is extremely needed to conduct gas injection research. Therefore, this paper studies the effect and affecting factors of scCO₂ huff and puff development in tight conglomerate reservoirs.

In this work, the properties of tight crude oil were tested. Then, the interaction between scCO₂ and tight oil was analyzed to clarify the influence of scCO₂ on the properties of crude oil, including bubble point pressure, gas−oil ratio (GOR), swell coefficient, viscosity, and density. Moreover, the minimum miscibility pressure (MMP) of scCO₂ with tight oil was determined, and the extraction effect of scCO₂ on tight oil was studied. Finally, the effects of scCO₂ and non-scCO₂ on crude oil production were studied through the huff-n-puff simulated experiment. Meanwhile, the influences of injection volume, soaking time, and throughput cycles in the simulation process of scCO₂ were analyzed. It was believed that this work can provide a further understanding of scCO₂-injected tight crude oil production.

2. EXPERIMENT MATERIALS AND METHODS

2.1. Materials. 2.1.1. Crude Oil and Brine Water. Crude oil and brine water were sourced from Xinjiang oilfield. The density of crude oil and GOR were 0.824 kg/m³ and 138 m³/m³, respectively. The saturated hydrocarbon distribution of degassed oil was measured using an Agilent 6890 AGC chromatography system with the distribution diagram shown in Figure 2. The ion content distribution of brine water was measured using an IC761 ion chromatograph (given in Table 1). The salinity of brine water was 20,512.59 mg/L.

Figure 1. Phase diagram of CO₂.

Figure 2. Alkane component distribution in degassed crude oil.

2.1.2. Gas. The gas-phase components were obtained by flash separation. The gas-phase components of the separated gas were measured using an Agilent 6890 AGC chromatography system with the molar fraction distribution listed in Table 2. CO₂ gas was taken from the CO₂ cylinder with a purity of 99.99%.

2.1.3. Core. All the cores used in the experiments were formation tight conglomerate cores collected from the oilfield. The cores were processed into standard cores with a diameter of 3.8 cm and a length of 6−8 cm. Figure 3 shows the tight conglomerate core sample. The average overburden pressure gas permeability and porosity of the cores were 0.16 mD and 8.84%, respectively, which were similar to the original formation properties.

2.2. Preparation of Live Oil. To simulate the crude oil in the original reservoir, live oil was prepared according to the original formation properties, and subsequent experiments were conducted with the prepared live oil and scCO₂. These experiments were carried out in a pressure−volume−temperature (PVT) analysis system manufactured by DBR Canada Inc. (hereinafter referred to as DBR-PVT).

First, the formation fluid recombination was used to fully stir the natural gas and degassed crude oil to prepare the target live oil under the original formation conditions (formation pressure: 37 MPa; temperature: 89 °C; GOR: 138 m³/m³). Then, the properties of the prepared live oil were measured in DBR-PVT.

The single degassing method was used in the experiment. The total components of the whole system remain unchanged in the oil−gas separation experiment, and the single-phase sample under the formation conditions was instantly degassed and converted to surface conditions. The change of sample volume and gas liquid volume before and after flash evaporation were measured. Also, the fluid density and viscosity were measured using a densitometer and falling ball viscometer, respectively. As shown in Table 3, the properties of prepared live oil were basically consistent with those of formation crude oil, so subsequent experiments were carried out with prepared live oil.
2.3. Interaction of Live Oil with scCO2. To study the influence of scCO2 on the target formation fluid and clarify the oil recovery mechanism of scCO2 huff and puff, the interaction between scCO2 and the prepared live oil was studied by DBR-PVT under the formation conditions. The effects of mole fractions of scCO2 on live oil were also studied by the single degassing experiment, including saturation pressure, swell coefficient, GOR, density, and viscosity. The saturation pressure was determined by the pore volume (PV) curve, and the swell coefficient was determined by the volume change of the added different mole fractions of CO2. The density and viscosity were measured using the densitometer and falling ball viscometer, respectively. Table 4 presents the properties of live oil with different mole fractions of scCO2.

As the scCO2 mole fraction increases, the saturation pressure, GOR, and swell coefficient increase, while the viscosity and density decrease. The viscosity and density of live oil decreased by 60 and 17%, respectively, with an increase of the mole fractions of scCO2 from 0 to 49%. All these results gave a strong hint that scCO2 injection was conducive to the increase of oil recovery.

2.4. Minimum Miscible Pressure. MMP is the minimum pressure at which the injected gas and crude oil reach miscibility through multiple contacts at the reservoir temperature. MMP of crude oil is usually measured by the method of slim tube experiment.24

First, the slim tube was saturated with live oil at 1 mL/min under the reservoir conditions, and then, CO2 was injected at 0.1 mL/min to displace the oil. When 1.2 PV was injected, gas injection was stopped and oil production was recorded at this time. After the experiment, the petroleum ether was pumped into the thin tube model for cleaning, and the bound oil in the model was removed. Finally, the remaining petroleum ether was blown out of the pipeline with high-pressure air. The appeal process was repeated to determine the respective recovery factor at different pressure points and is plotted in Figure 4 to determine the minimum miscible pressure point.

According to industry standards,25 miscibility was considered to be achieved when the oil recovery exceeded 90% after injection of 1.2 PV gas. On the contrary, if not, it was immiscible. The miscible (more than 90%) and the immiscible (less than 90%) were fitted into two straight lines. The pressure at the intersection of the two lines was 33.6 MPa, which was identified as MMP between crude oil and CO2.

2.5. CO2 Extraction Experiment. The extraction experiment was conducted with a JEFRI visual mercury-free formation fluid PVT analyzer produced by DBR, Canada. The glass cylinder was the core component of the PVT visual analyzer, with a maximum volume of 150 mL. The temperature testing range of the equipment was −30 to 200 °C with an accuracy of 0.1 °C. The flow chart of the extraction experiment is shown in Figure 5.

First, the prepared live oil was transferred into DBR-PVT under the reservoir conditions (37 MPa, 89 °C). A certain
volume of scCO$_2$ was then added to the PVT cell and stirred for 2 h to mix it fully with the live oil. The valve was opened and the sample was released slowly until the pressure in the container dropped to 37 MPa. The volume of fluid in the PVT cell was recorded through the observer mirror. The extracted fluid was separated into the oil phase and gas phase by a
separator, and the components of the gas phase and oil phase were analyzed using a gas chromatograph. The above steps were repeated to complete the follow-up extraction experiment.

2.6. Process of scCO2 Huff and Puff Experiments. The main principle of the CO2 huff and puff experiment is to inject a certain amount of CO2 into the formation and to conduct a period of soaking. During the soaking stage, CO2 dissolves into the crude oil, increasing its volume and reducing its viscosity. Finally, the well was opened for production, thus increasing the production.

CO2 huff-n-puff development has a significant oil-increasing effect on low-permeability tight reservoirs, complex small oil fields, and small fault block oil fields. CO2 huff and puff development is suitable for small and complex reservoirs due primarily to its advantages such as a short simulation time, low cost, and quick benefits.

The specific steps of the huff and puff experiment are as follows: First, the tight conglomerate core is vacuumed and saturated with formation water to establish bound water saturation. Then, the prepared live oil is injected into the tight conglomerate cores to establish the initial saturated oil state to simulate the reservoir state. At this time, a certain volume of CO2 slug is pumped into the core, and then, the switch is turned off for soaking so that the live oil and CO2 fully interact. After a period of time, the inlet switch is turned on for oil recovery. Figure 6 is the flow diagram of the huff and puff experiment.

3. RESULTS AND DISCUSSION

3.1. Extraction Experiment. The mole content of C1 in the original formation crude oil was 39.04%, while the mole content of C2−C6 was relatively low. In the extracted gas-phase components, as shown in Figure 7, C1 was the main component, while the C2−C6 content was relatively small. Similarly, the oil-phase components extracted, as shown in Figure 8, were mainly concentrated in the light components of C6−C17, while the heavy components of C18+ were less extracted.

It could be seen that CO2 had a certain extraction effect on all components of formation crude oil, especially on light hydrocarbons. Moreover, with the increase of carbon number, the extraction effect of CO2 on the components weakened accordingly. At the same time, while increasing CO2 extraction times, the light component in crude oil was gradually extracted, as evidenced by Figures 7 and 8.

3.2. Comparison between scCO2 and Non-scCO2. Under the formation conditions, 0.5 PV scCO2 and 0.5 PV non-scCO2 slugs were pumped into the tight cores at an injection rate of 0.05 mL/min. Then, the switch was turned off, and the tight core was soaked for 2 h. Finally, the inlet switch was turned on for oil recovery, and the oil recovery factor was recorded.

Figure 9 shows the relation of scCO2 and non-scCO2 injection and soaking pressure with time. It could be seen that the pressure increased with time in the process of injection and soaking. It was of note that after 2 h of soaking, the pressure for scCO2 was 2.02 MPa higher than that for non-scCO2, which has a very important effect on the recovery of the reservoir. The experimental results also showed that the recovery rate of scCO2 was 21.88%, which was 4.02% higher than that of non-scCO2 injection. This fact indeed indicated that scCO2 was more favorable for enhancing the recovery rate of the tight reservoir.

Figure 7. Gas-phase composition distribution of extraction.

Figure 8. Oil-phase composition distribution of extraction.

Figure 9. Pressure comparison with injecting scCO2 and non-scCO2.
ScCO₂ had excellent properties that are different from the conventional liquid and gas. It had very good solubility in non-polar organic matter and good diffusion performance. Therefore, scCO₂ could improve the recovery efficiency of tight reservoirs more than non-scCO₂.²⁷ In the process of huff-n-puff experiments, scCO₂ could better interact with crude oil than non-scCO₂. It could better dissolve and expand in crude oil, thereby reducing its viscosity. In this way, it would generate a higher pressure in the soaking and thus had a higher oil recovery factor.

3.3. Effect of Injection Volume. In these experiments, we investigated the effect of scCO₂ injection volume on the huff and puff production efficiency in tight formations. Under the reservoir conditions, different volumes of scCO₂ (0.25, 0.50, and 0.75 PV) were pumped into the tight conglomerate cores for simulated experiments, and the soaking time was 2 h.

Figure 10 exhibits the pressure curves of scCO₂ simulated experiments with different injected volumes. During the scCO₂ injection phase, the pressure increased as scCO₂ was injected into the core. Also, the more the scCO₂ injection, the greater the pressure increase. In the soaking stage, the crude oil dissolved and expanded in the presence of scCO₂, so the pressure increased gradually.

By comparing the curves of different injection volumes, the recovery was the highest when injecting 0.75 PV scCO₂, which was 23.81%, only 1.93% higher than that when injecting 0.50 PV. When 0.50 PV scCO₂ was injected, the pressure increased to the maximum, 1.97 MPa, at the 2 h soaking stage. At that time, scCO₂ interacted with crude oil sufficiently, and the injection volume was more economical and effective, which was the best scCO₂ injection volume in the process of huff-n-puff simulated experiments.

3.4. Effect of Soaking Time. To investigate the effect of soaking times on the huff and puff experimental results, three different soaking times (1, 2, and 4 h) were selected for these experiments. In each experiment, 0.50 PV scCO₂ was injected into the tight core for huff-n-puff simulated experiments under the reservoir conditions. Finally, the inlet switch was turned on for production, and the experimental results were compared.

As shown in Figure 11, in the scCO₂ injection stage, the pressure increased with the continuous injection of scCO₂. In the soaking stage, with the increase of the soaking time in the simulated experiment, the interaction between scCO₂ and crude oil became more sufficient, the pressure gradually increased, and the production degree increased. When the soaking time was extended from 1 to 2 h, the recovery rate increased by 5.88%. When the soaking time was extended to 4 h, there was little change in the production degree of crude oil. As shown in Figure 11, when the soaking time was longer than 2 h, the degree of pressure change of the system gradually decreased, indicating that the scCO₂ effect predominantly contributed to the initial stage of the soaking process. However, when the soaking time was increased to a certain extent, the prolonged soaking time was not ideal for the recovery, and the most economical and effective soaking time was 2 h.

3.5. Effects of Throughput Cycles. In these huff and puff experiments, we studied the effect of multiple throughput cycles on enhanced oil recovery. First, 0.5 PV of scCO₂ was injected into the saturated oil core, and then, the soaking lasted for 2 h. Finally, the inlet switch was turned on for oil recovery. After the oil production was over, scCO₂ was injected into the tight core again, and the above steps were repeated successively to complete multiple throughput cycles. Several cycles later, the experimental results were analyzed. All experiments were completed under the formation conditions.

Figure 12 shows the change of pressure with time in the whole process of the simulated experiment. In the stage of scCO₂ injection, with the injection of scCO₂, the system energy and the pressure gradually increased. In the soaking stage, the crude oil dissolved and expanded in the presence of scCO₂, which made the pressure increase gradually. In the production stage, with the opening of the inlet switch, the pressure dropped rapidly and the crude oil was produced. However, with the increase of the throughput cycles, the increase of pressure in the scCO₂ injection stage and the soaking stage gradually decreases, and the production degree also decreases continuously. As shown in Figure 13, the first three cycles of oil recovery rates were 21.88, 12.50, and 3.13%, respectively, and almost no oil was produced at the fourth round. This was consistent with the results of Wei et al.,²¹ in which the significant increase in oil production only occurred in the first two cycles, and the increase after that was not
obvious. The total production from the first two cycles accounted for 91.66% of the total recovery.

After four cycles of simulation, the total recovery rates were 37.51 and 15.63% higher than that of a single throughput cycle. Thus, after several throughput cycles of scCO2, it could effectively improve the ultra-low-permeability reservoir recovery factor, but the recovery did not improve after more than three throughput cycles. Therefore, it was advisable to suggest three cycles of the scCO2 huff-n-puff simulated experiment.

4. CONCLUSIONS

In this study, the effect of scCO2 on enhancing oil recovery in the tight conglomerate reservoir was studied, and the following conclusions were drawn:

1. The MMP between scCO2 and crude oil was 33.6 MPa. Therefore, scCO2 and crude oil could realize a miscible phase under a reservoir pressure with 37 MPa.
2. ScCO2 had a certain extraction effect on crude oil, especially on light components that were mainly concentrated in C6−C17 hydrocarbons. In the process of CO2 extraction, the content of light components decreased obviously.
3. ScCO2 huff-n-puff simulated experiments could effectively improve the recovery efficiency of tight reservoirs. Compared with non-scCO2, it was of note that after 2 h of soaking, the pressure for scCO2 was 2.02 MPa higher than that for non-scCO2; the oil displacement efficiency increased by 4.02%. Meanwhile, the optimal injection volume, soaking time, and throughput cycles were determined to be 0.50 PV, 2 h, and 3 cycles, respectively.

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Notes
The authors declare no competing financial interest.

ACKNOWLEDGMENTS

The authors gratefully acknowledge the support of the National Natural Science Foundation of China (grant no. U19B2010). The authors also thank the reviewers for their valuable comments.

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