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Research Article

Experimental Study of Diffusion and Formation Mineral Change in Supercritical CO_2 Huff and Puff Process of Shale Reservoir

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In order to understand the diffusion during CO_2 huff and puff in the development of shale oil and its influence on the formation, expansion and viscosity reduction experiments of shale oil-CO_2 system, CO_2 extraction experiments, and CO_2 huff and puff physical simulation experiments were conducted. The diffusion characteristics of CO_2 during huff and puff and their effects on formation minerals were studied by chromatographic analysis and X-ray diffraction analysis of artificially fractured natural cores. Research indicates that CO_2 huff and puff technology is an effective method to enhance the recovery of shale reservoirs after fracturing. By injecting CO_2, the light components of shale oil can be effectively extracted; when the amount of injected CO_2 is 50%, the saturation pressure of shale oil increases to 27.72 MPa, and the expansion coefficient increases by 27.9%, the viscosity reduction rate of shale oil can reach 58.97%, and the density reduction rate is 10.02%; under the soaking well pressure of 50 MPa, when 0.5PVCO_2 was injected and the well stuffed for 8 hours, the CO_2 was fully dissolved in the shale oil, and the continuous increase of the injection slug had a little effect on the CO_2 diffusion. During the CO_2 huff and puff process, CO_2 would dissolve in the formation water and fracturing fluid and reacts with dolomite in the reservoir rock, consuming a large amount of dolomite in the reservoir, and the dolomite mineral content of core sample decreased from 30.1% to 2.6%.

1. Introduction

Shale oil is abundant in reserves worldwide, and the United States has become a net oil exporter from a net oil importer by relying on the successful exploitation of shale oil [1–4]. In contrast, the exploration and development of shale oil reservoirs and related research in China are still at a preliminary stage, and the overall understanding and development are relatively insufficient [5–8]. With the increasing demand for energy in economic development, China's external dependence on oil and gas resources has far exceeded the energy security alert of 50% in recent years [9–12]. Unconventional oil and gas resources have become a major strategic succession for China's oil exploration and development with their considerable resources [13–17].

The Lucaogou Formation shale oil reservoir in Jimsar, Xinjiang, is an ultralow porosity and ultralow permeability reservoir [18–21]. With a pressure of 41.25 MPa and a temperature of 91.05°C, the reservoir is weakly water-sensitive. Shale oil is currently developed by the method of "horizontal well+volume fracturing." Horizontal wells that have been put into production face the problems of large differences in production effect, rapid oil production decline, low recovery rate, and unrecoverable large amount of resources. It is urgent to determine a reasonable and effective enhanced oil recovery method for the Lucaogou Formation shale reservoir in Jimsar and to provide a theoretical basis for field experiments.

The CO_2 huff and puff technology is a mining technology that can effectively improve shale and low permeability reservoirs [22–26]. The development of shale reservoirs in China is still in its infancy [27–31]. However, relatively little research has been conducted on the component diffusion characteristics of CO_2 in shale oil and the impact on the formation. The recent CO_2 huff and puff field test of the Jimsar Lucaogou Formation shale reservoir has achieved remarkable results, but there is still a diffusion effect on CO_2 components [32–35] and the problem of unclear understanding of the oil-increasing mechanism [36–39]. Due to the high formation pressure and huff and puff construction pressure in the Jimsar shale reservoir, the current research on its huff and puff effect is mostly numerical simulation or molecular simulation research [40–42]. The research on the modification effect of shale rocks is relatively scarce [43–46]. Different from previous literature, the new insights of this study using the natural shale cores of the Jimsar Lucaogou Formation were used to carry out ultrahigh pressure physical simulation huff and puff experiments, CO_2 shale oil interaction experiments under high temperature and high pressure, and CO_2 shale oil extraction experiments under high temperature and high pressure, combined with chromatographic analysis, and X-ray diffraction whole rock analysis experiments are used to gain a deeper understanding of the CO_2 huff and puff mechanism, which provides guidance for further optimization of the CO_2 huff and puff parameters.

2. Materials and Methods

2.1. Materials. In this study, artificially fractured natural cores were used to conduct CO_2 huff and puff diffusion experiment. The cores were cut from Jimsar Lucaogou Formation shale reservoir. The core was first cut in half along the midline and filled with quartz sand to simulate postfracturing fractures. Use the heat-shrinkable tubing for fixing to ensure the overall strength of the core. The preparation process is shown in Figures 1 and 2. The properties (permeability and porosity) of the cores are listed in Table 1.

The salinity of formation water in the Jimsar Lucaogou Formation shale oil reservoir is 69 860 mg/L; in the degassed shale oil in the Lucaogou Formation shale reservoir in Jimsar, the shale oil viscosity at 50°C is 61.6 mPa·s, the shale oil density under surface conditions is 0.893 g/cm^3, and the single degassed oil ratio is 21 m^3/m^3.

Experimental Equipment: Agilent 7820A gas chromatography-mass spectrometer, Huck MARS III rotational rheometer, X-ray photoelectron spectrometer, oven, core flooding device, etc.

2.2. Methods

2.2.1. Configuration of Live Oil Samples. The first-time degassing data of the test well (Table 2) and the gas-oil ratio (21m^3/m^3) produced in the lower sweet spot of shale oil in the Jimsar Lucaogou Formation were used for compounding natural gas and configuring live oil samples.

2.2.2. CO_2 Shale Oil Extraction Experiment under High Temperature and High Pressure. (1) Estimate the sample viscosity; select the appropriate cone and plate diameter according to the sample viscosity; (2) Evaluate whether the selected fixture, speed, and clearance value are appropriate through the data measured by the instrument, and then modify the fixture selection to optimize the working range; (3) Turn on the air compressor, release the air bearing lock, and turn on the Harker rheometer host; (4) Measure live oil sample viscosity at formation temperature(91.1°C) using the Huck rheometer; (5) Under the current reservoir temperature (91.1°C) and reservoir pressure (42.25 MPa), introduce the prepared live oil sample into the high temperature and high pressure PVT tube; (6) CO_2 is injected into the PVT tube to make it fully mixed with shale oil; (7) Maintain the temperature, shake the sample for 60 minutes, and let it stand for 60 minutes; (8) Depressurize the PVT tube and measure shale oil viscosity again at formation temperature (91.1°C) using the Huck rheometer; (9) Collect the produced oil samples and record the pressure and residual oil volume in the PVT pipe; And (10) the components of the configured shale oil and the remaining oil were analyzed by chromatography-mass spectrometry.

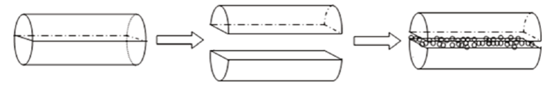

Figure 1: Physical treatment of natural cores.

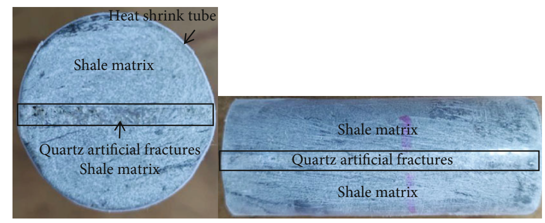

Figure 2: Artificially fractured natural core.

2.2.3. CO_2 Shale-Oil Interaction Experiment under High Temperature and High Pressure. (1) Under the current reservoir temperature (91.1°C) and reservoir pressure (42.25 MPa), inject CO_2 into the PVT pipe, and fully stir the CO_2 and shale oil to make it completely dissolved in the shale oil and reach saturation; And (2) as shown in Figure 3, continuously increase the gas-oil ratio, and measure the changes of high-pressure physical properties (viscosity, density, saturation pressure, expansion coefficient, and gas-oil ratio) of CO_2 under different gas injection rates.

2.2.4. CO_2 Huff and Puff Simulation Experiment under High Temperature and High Pressure. (1) Clean the core, dry it, vacuumize it, and saturate the simulated formation water. Test the porosity and permeability of the core; (2) Under the current reservoir temperature (91.1°C) and reservoir pressure (42.25 MPa), a high-pressure displacement pump is used to inject oil into the core at a rate of 0.05 mL/min. When the core is saturated, record the saturated oil content of the core. Then, put the core into shale oil to age for 24 hours; (3) Inject CO_2 into the core at a rate of 0.05 mL/

Table 1: CO_2 huff and puff diffusion experiment core parameters.

Core number	Experiments applied	Lithology	Length [cm]	Diameter [cm]	Porosity [%]	Permeability (gas test) [$10^{-3}\ \mu m^2$]	Permeability after artificial fracture (water test) [$10^{-3}\ \mu m^2$]
Core #1	Injection slug on CO_2 diffusion	Argillaceous siltstone	6.75	2.50	10.15	0.46	93.15
Core #2			6.67	2.50	9.84	0.38	93.14
Core #3			6.39	2.50	10.35	0.63	96.65
Core #4			6.82	2.50	9.58	0.47	91.16

Table 2: Simulated natural gas composition.

	Methane	Ethane	Propane	Isobutane	n-Butane	Isopentane	n-Pentane	Hexane
Component proportion%	63.43	11.43	14.69	2.81	3.42	0.59	0.51	0.08

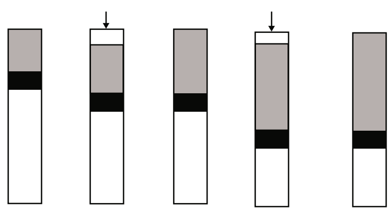

Figure 3: The process of gas injection expansion experiment.

min, when the CO_2 input reaches the target, turn off the pump and record the inlet pressure at different times; And (4) after 8 hours, open the inlet valve of the core and measure the pressure and oil production, when the pressure depletion drops to 8.00 MPa, the experiment ends. The experimental process is shown in Figure 4.

3. Results and Discussion

3.1. Effect of CO_2 Extraction of Light Components in Shale Oil. The mass fraction of the injected CO_2 was 40.0%. It can be seen from the experimental results (Figure 5) that the shale oil components extracted from shale oil are mainly light components (C_2-C_7) and medium components (C_8-C_{12}), and there is almost no C_{13+} group. The fractions were extracted; the light fractions in the remaining oil almost disappeared, and the proportion of C_{13+} heavy fractions increased accordingly. This shows that CO_2 is feasible in extracting light components in shale oil and increasing its recovery.

3.2. Effect of CO_2 on Physical Properties of Shale Oil. Figure 6(a) shows the variation of shale oil volume coefficient and saturation pressure with CO_2 injection. The saturation pressure and volume coefficient of shale oil increase with the increase of CO_2 injection. In Figure 6(b), the changes of viscosity and density of shale oil with the injection amount of CO_2 are reflected. With the increase of CO_2 injection, the viscosity of shale oil decreased significantly, and the density of shale oil decreased.

The mixing experiment of CO_2 and shale oil shows that when the mass fraction of CO_2 is less than 30%, the saturation pressure and expansion coefficient of CO_2 increase slowly, and CO_2 is more likely to dissolve in shale oil, while when the mass fraction of CO_2 is greater than 40%, the saturation pressure of CO_2 is increasing faster than before, and CO_2 is difficult to dissolve in shale oil, as shown in Figure 6. When the amount of CO_2 added reaches 50%, the saturation pressure is 27.72 MPa, the expansion coefficient is 1.279, and the formation energy is effectively supplemented.

When CO_2 is injected into shale oil, its viscosity density decreases with the continuous injection of CO_2. When the amount fraction of CO_2 substances is lower than 10%, the viscosity of shale oil decreases significantly, and with the continuous injection of CO_2, the viscosity of shale oil continues to decrease, but the decrease rate slows down; the density of shale oil increases with the continuous injection of CO_2 has been showing a slow downward trend. At the end of injection, the viscosity of shale oil decreased by 58.97%, and the density of shale oil decreased by 10.02%. The results show that CO_2 has a good extraction effect on light components in shale oil and has a good viscosity reduction effect, which can significantly improve its fluidity.

3.3. Influence of Injection Slug on CO_2 Diffusion Effect. CO_2 huff and puff pressure is 50 MPa, and the experimental temperature is the formation temperature of 91.1°C. The upper sweet spot cores (core#1-core#4) with similar depths were selected for the huff and puff experiment, the core parameters is shown in Table 1. Inject 0.20, 0.40, 0.50, and 0.60 PV of CO_2 at a rate of 0.05 mL/min, hold the well for 8 hours for huff and puff simulation experiment, and record time, metering pump readings, amount of oil produced, and amount of CO_2 gas produced, the experimental results are shown in Table 3. Calculate the oil displacement efficiency of CO_2 huff and puff, etc., the inlet pressure that changes

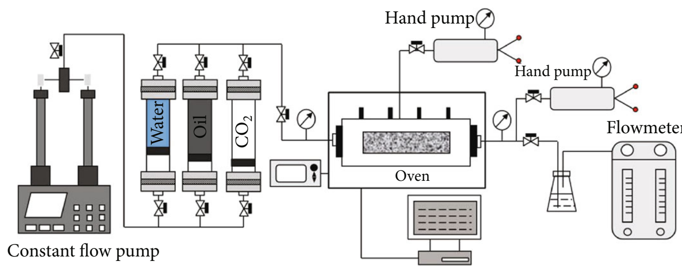

Figure 4: Flow chart of CO_2 huff and puff simulation experiment.

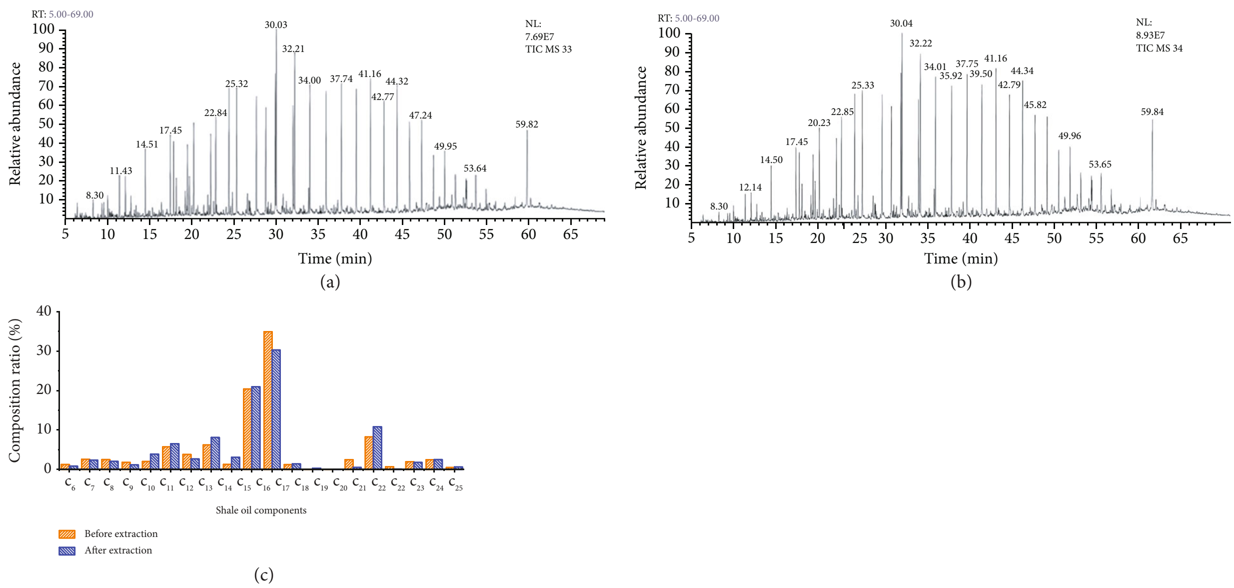

Figure 5: (a) Chromatographic analysis results of shale oil before extraction. (b) Chromatographic analysis results of shale oil after extraction. (c) Changes of carbon components in CO_2 extraction from shale oil.

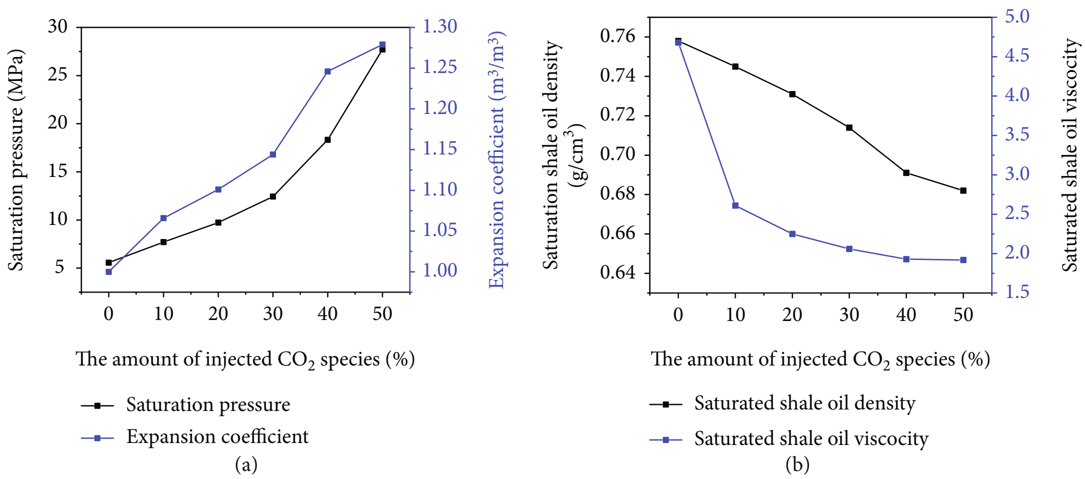

Figure 6: (a) Swelling effect of shale oil during CO_2 injection. (b) Viscosity reduction effect of shale oil during CO_2 injection.

with time is shown in Figure 7, and the core recovery degree and the produced gas-oil ratio are shown in Figure 8.

It can be seen from Figures 7 and 8 that with the continuous injection of CO_2, CO_2 is continuously dissolved into the shale oil, increasing its volume and increasing its inlet pressure. During the soaking stage, the swelling reaction between CO_2 and shale oil is more obvious, which further increases the inlet pressure. When 0.2PV CO_2 is injected,

TABLE 3: Experimental results of CO_2 huff and puff diffusion with different slugs.

Core number	Injection slug [PV]	Produced gas volume [cm^3]	Produced fluid volume [cm^3]	Recovery factor [%]	Gas oil ratio [m^3/m^3]
Core #1	0.2	5.96	0.42	14.68	13.86
Core #2	0.4	50.08	0.76	27.05	65.89
Core #3	0.5	83.91	1.03	37.05	81.47
Core #4	0.6	179.76	1.10	40.15	163.42

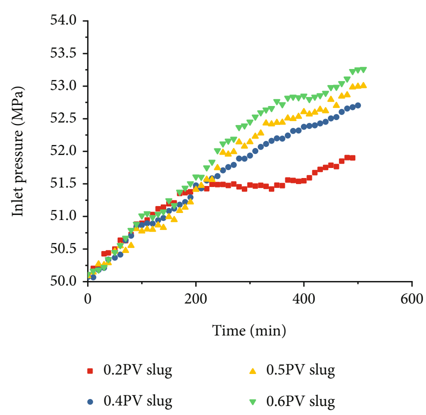

FIGURE 7: Pressure changes under different injection slugs.

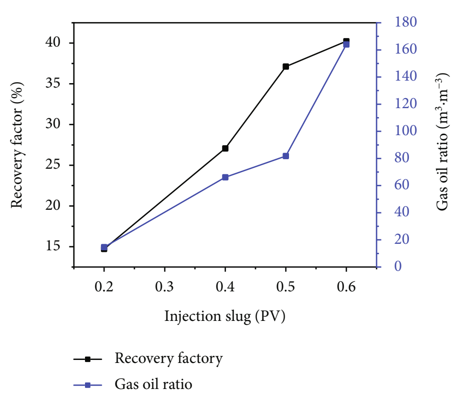

FIGURE 8: Oil production under different injection slugs.

almost no gas is produced, the scope of CO_2 is limited, and the recovery degree is low. When 0.20-0.50 PV CO_2 is injected, increasing the CO_2 injection amount can effectively increase the CO_2 action distance and improve the expansion of shale oil increase the inlet pressure and effectively replenish the energy of the reservoir. Its recovery factor increased from 14.72% to 37.13%. The recovery factor did not change significantly when the CO_2 injection volume increased from 0.50 PV to 0.6 PV. At this time, the method of increasing CO_2 injection alone could not significantly improve the recovery factor, and the produced gas volume increased sharply, and the gas-oil ratio rose sharply.

From the results of CO_2-shale oil chromatography (Figure 5) and CO_2 huff and puff results (Figure 7), it can be seen that the diffusion effect of CO_2 is obviously better than that of heavy alkanes for light alkanes. For C_2-C_7 alkane molecules, due to the small molecules and large intermolecular gaps, CO_2 can pass through the gaps between C_2-C_7 molecules and quickly achieve mixing with CO_2 and alkane molecule system, fully stretching, and the system becomes more and more dispersed, and the free motion of C_2-C_7 alkanes is enhanced, so that CO_2 can almost achieve complete diffusion of C_2-C_7 molecules. CO_2 improves the mobility of alkane molecules and makes the oil phase more mobile. The diffusion effect of CO_2 on C_7-C_{12} alkane molecules is still very prominent. C_7-C_{12} alkane molecules are rapidly miscible with CO_2, and C_7-C_{12} alkane molecules are partially extracted. The C_{18}-C_{25} alkane molecules have characteristics that differ from the C_7-C_{12} system, and the molecules themselves are less mobile and are hardly extracted.

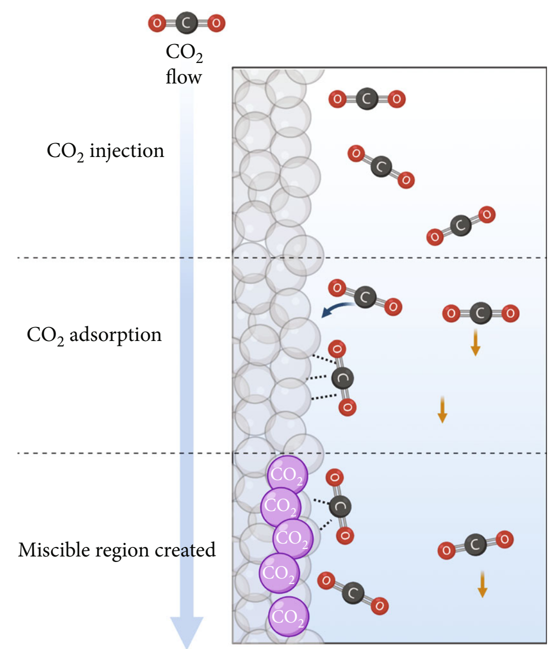

FIGURE 9: CO_2 huff and puff diffusion process.

As shown in Figure 9, with the injection of CO_2, CO_2 continuously transports and diffuses to the oil formation, forming a miscible region on the surface of the oil formation, which has the effect of swelling and viscosity reduction and increasing the mobility of shale oil. Meanwhile, as the

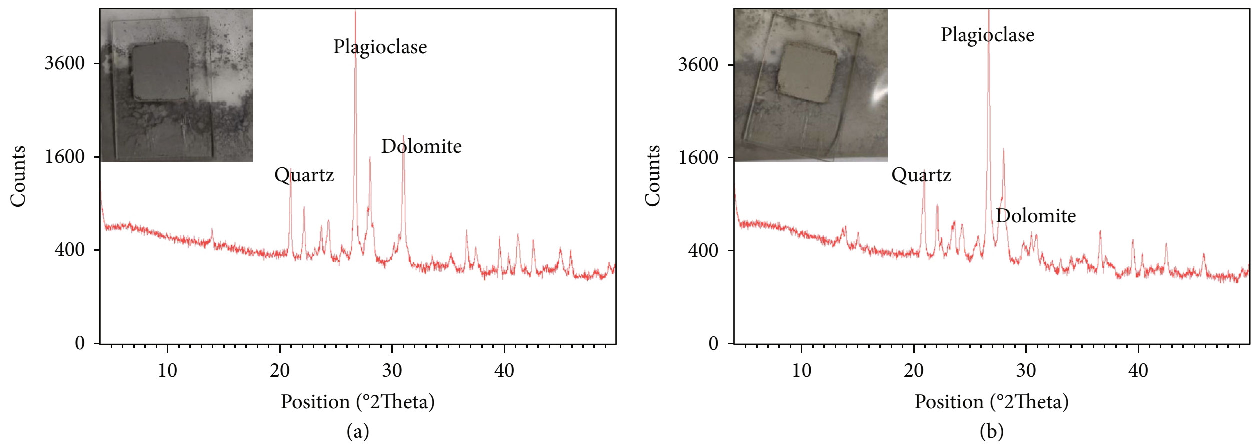

Figure 10: (a) X-ray diffraction results before CO_2 huff and puff. (b) X-ray diffraction results after CO_2 huff and puff.

diffusion degree increases, the CO_2 extraction effect becomes more obvious, and light components are extracted in large quantities, which has the effect of improving the recovery of CO_2 huff and puff.

3.4. Effects of CO_2 Huff and Puff on Reservoir Minerals. Before the experiment, core#1 was ground into 200-mesh particles and tableted. After CO_2 huff and puff, core#1 was ground into 200-mesh particles and tableted, and the quantitative analysis of whole rock minerals was carried out by X-ray diffraction, respectively. As shown in Figure 10, core#1 is mainly composed of quartz, plagioclase, dolomite, and clay minerals, of which plagioclase has the highest content, accounting for 39.5% of the mineral content, followed by quartz and dolomite, accounting for 29.7% and 30.3%, respectively. However, after CO_2 huff and puff, the core dolomite was consumed in large quantities, its wave peak almost disappeared, and the dolomite mineral content decreased from 30.1% to 2.6%.

It can be seen from the experimental results that in the process of high temperature and high pressure CO_2 huff and puff, CO_2 will not only dissolve in oil but also dissolve in formation water or fracturing fluid to form carbonated water and react with dolomite in the reservoir rock. Extensive consumption of dolomitic minerals in reservoirs has almost no effect on other mineral components.

4. Conclusions

The conclusions of this study are listed as follows.

(1) CO_2 huff and puff technology is an effective method to enhance the recovery of shale reservoirs after fracturing. By injecting CO_2, the light components of shale oil can be effectively extracted, and at the same time, the viscosity and density of shale oil can be reduced, and the formation energy can be supplemented

(2) In the experiment of mixing high temperature and high pressure CO_2 with shale oil, when the amount fraction of injected CO_2 is less than 30%, the saturation pressure and expansion rate of shale oil rise relatively slowly, but the viscosity of shale oil decreases sharply, and shale oil density has also been reduced. When the amount of injected CO_2 is greater than 30%, the saturation pressure and expansion rate of shale oil rise sharply with CO_2 injection. However, the viscosity and density of shale oil did not decrease significantly. When the amount of injected CO_2 is 50%, the saturation pressure of shale oil increased to 27.72 MPa, and the expansion coefficient increased by 27.9%, the viscosity reduction rate of shale oil can reach 58.97%, and the density reduction rate is 10.02%

(3) Under the soaking well pressure of 50 MPa, when 0.5 pv CO_2 was injected and the well stuffed for 8 hours, the CO_2 was fully dissolved in the shale oil, and the continuous increase in the injection slug had a little effect on the CO_2 diffusion. During the CO_2 huff and puff process, CO_2 would dissolve in formation water and fracturing fluid and react with dolomite in the reservoir rock, consuming a large amount of dolomite in the reservoir, and the dolomite mineral content of core sample decreased from 30.1% to 2.6%

Data Availability

The data that support the findings of this study are available on request from the corresponding author. The data are not publicly available due to privacy or ethical restrictions.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

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