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Abstract

The utilization of gas enhanced oil recovery to extract oil from unconventional reservoirs has become a widely discussed topic, as it has proven to be effective in significantly boosting oil recovery rates. Among various enhanced oil recovery methods, Gas Enhanced Oil Recovery (GEOR) is a frequently implemented approach. However, a significant challenge encountered during the process of injecting Carbon Dioxide (CO₂) or Nitrogen (N₂) to displace oil is the occurrence of asphaltene precipitation and deposition, which can impede production. This work is an experimental study to examine the effects of cyclic (huff-n-puff) CO₂ or N₂ processes on oil recovery performance and asphaltene deposition using Eagle Ford shale cores. The minimum miscibility pressure (MMP) was first determined for CO₂ and N₂, and then different injection pressures (miscible and immiscible) were chosen to carry out CO₂ and N₂ huff-n-puff tests. Miscible and immiscible pressures were selected to implement the huff-n-puff test for CO₂ and N₂. Pore size distribution was analyzed to highlight the impact of asphaltene particles on pore plugging.

Introduction

The demand for oil from shale, also known as shale oil or tight oil, has increased significantly over the past decade. Shale oil is extracted through enhanced oil recovery methods or hydraulic fracturing, a process that involves injecting water, sand, and chemicals into shale rock formations to release oil and gas (Elturki and Imqam, 2020a; 2020b). GEOR techniques may be more beneficial than hydraulic fracturing procedures, which can only retrieve a small fraction of trapped oil (Elwegaa et al., 2019; Ahmed et al., 2019; Elturki et al., 2021). MMP is an important parameter in enhanced oil recovery (EOR) techniques such as gas injection (Du et al., 2019). As oil production from shale has become increasingly important, understanding the effects of MMP on the recovery of oil from shale reservoirs has become a key area of research (Lashgari et al., 2019). One of the challenges in oil production from shale is the deposition of asphaltenes, which can lead to decreased production and even well failure (Li et al., 2020; Elturki and

Imqam, 2022b, 2022c, 2022d). Research has shown that understanding the relationship between MMP and asphaltene deposition can help to mitigate these issues and improve recovery (Soroush et al., 2014; Lo et al., 2022; Elturki et al., 2021a). By carefully selecting injection gas compositions and pressures, it is possible to optimize the MMP and reduce the risk of asphaltene deposition, leading to more efficient and effective oil recovery from shale reservoirs.

Asphaltene deposition is a common issue encountered during gas injection operations in shale unconventional reservoirs (Elturki and Imqam, 2021b). Gas injection, particularly CO₂ injection, is an EOR method that involves injecting gas into the reservoir to increase oil recovery. However, the injection gas can cause the destabilization and precipitation of asphaltenes, which are complex organic compounds found in crude oil (Elturki and Imqam, 2021c). Asphaltenes can accumulate and deposit on the pore throats of shale formations, reducing permeability and impeding gas flow (Ahmed et al., 2022). This can lead to a decrease in reservoir performance and, in some cases, a complete cessation of oil and gas production. Asphaltene deposition occurs when the reservoir conditions, such as pressure and temperature, cause the asphaltenes to become unstable and agglomerate, forming solid deposits (Mohammed et al., 2021). The severity of asphaltene deposition depends on several factors, such as the reservoir's geology, the injection gas composition and pressure, and the crude oil's properties (Adebiyi, 2021). To mitigate asphaltene deposition, operators can employ several strategies, such as adjusting the injection gas composition and pressure to minimize asphaltenes' destabilization, using asphaltene inhibitors to prevent precipitation and deposition, and implementing effective surveillance and monitoring programs to identify early signs of deposition (Vargas and Tavakkoli, 2018). Other strategies include periodic solvent soaking and mechanical removal of deposits (Al-Qasim et al., 2018).

There have been few studies in recent years that have focused on investigating the impact of asphaltene precipitation under huff-and-puff gas injection (Shen and Sheng, 2017; Mohammad et al., 2017; Elturki et al., 2022a; 2023). Shen and Sheng (2017) investigated the effect of CO₂ huff-and-puff injection on the permeability and pore clogging resulting from asphaltene blockage in Eagle Ford shale. Findings indicated that after six CO₂ cycles, pore diameters in the range of 100–800 nm and pores smaller than 100 nm were reduced. Mohammad et al. (2017) estimated the production of asphaltene in low-permeability reservoirs after CO₂ injection using computational methods. They intended to optimize CO₂ injection by adding brine to huff-and-puff CO₂ injection in order to reduce asphaltene problems. Shen et al. (2019) performed a simulated investigation in order to obtain comprehensive understanding of the primary aspects that might influence asphaltene deposition and precipitation in hydraulically fractured shale formations using the CO₂ huff-and-puff injection technique. They observed that asphaltene deposition might vary between the rock matrix and the cracked network; hence, the decrease in permeability would also vary. Although the studies mentioned focus on various factors that affect shale oil production using the gas huff-and-puff method, there is a deficiency in comprehensive research on how to assess problems related to asphaltene precipitation and its effect on oil production performance in shale reservoirs using CO₂ and N₂ huff-n-puff technique. The purpose of this investigation is to give an experimental comparison of the effectiveness of CO₂ and N₂ huff-and-puff miscible and immiscible conditions on oil recovery, as well as the role of asphaltenes in oil reduction.

Materials and Methods

The slim tube technique was utilized to determine the MMP of CO₂ and N₂. The main components of the experiment are shown in Figure 1. All the experiments were conducted at 70°C to mimic the reservoir temperature. The tube had dimensions of length 13.10 m, inside diameter 0.21 cm, and outside diameter 0.41 cm. The following steps were followed to conduct the tests:

1. The first step was cleaning the slim tube, followed by saturating it with crude oil and injecting gas. The first accumulator stored crude oil for saturating the slim tube, the second accumulator

contained n-heptane solvent for washing the slim tube, and the third accumulator contained gas for injection into the slim tube during the tests.

- The tests started with completely filling the slim tube with distilled water, followed by constant injections of crude oil at a rate of 0.25 ml/min until the tube was saturated with oil. Gas was injected at a pressure previously determined (the predetermined pressures were ranged from 200 psi to 2000 psi) using the syringe pump's constant pressure mode until the gas broke through or 1.2 pore volume of gas was injected, at which point the test was stopped.
- The MMP was calculated by comparing and plotting the pressure of gas injection (X-axis) to the total amount of recovered oil (Y-axis). The intersection will be the MMP, as shown in Figure 2.
- After each experiment, the slim tube setup was cleaned thoroughly with xylene to remove any oil residue that may have impacted the next experiment or/and pressure.
- The above steps were repeated for each gas (i.e., CO₂ and N₂).

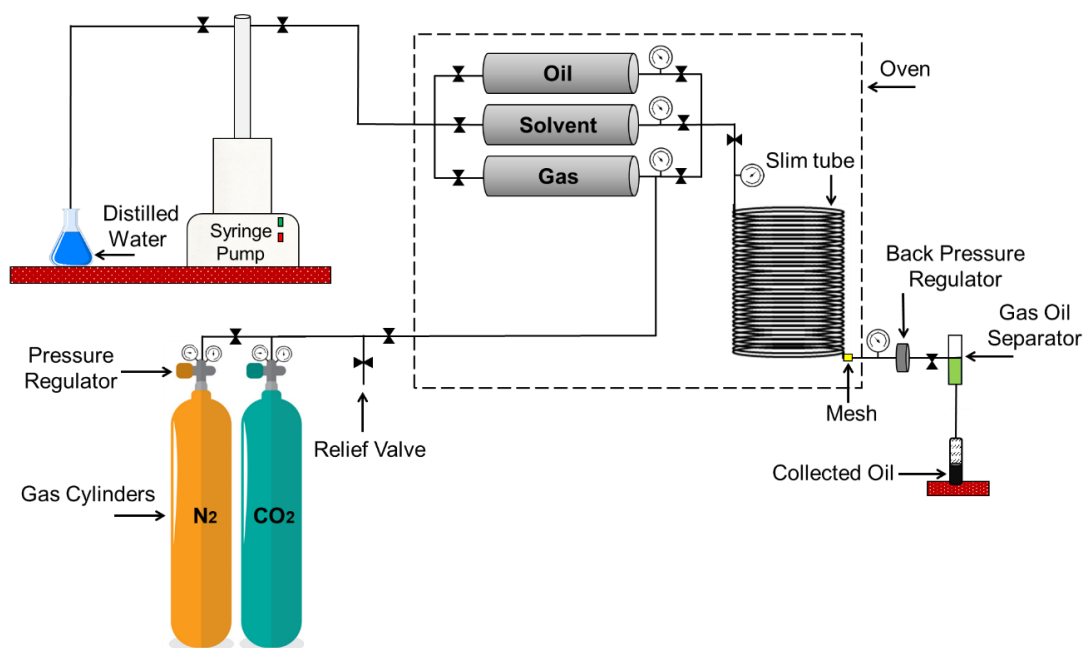


Figure 1. Slim tube experiment setup.

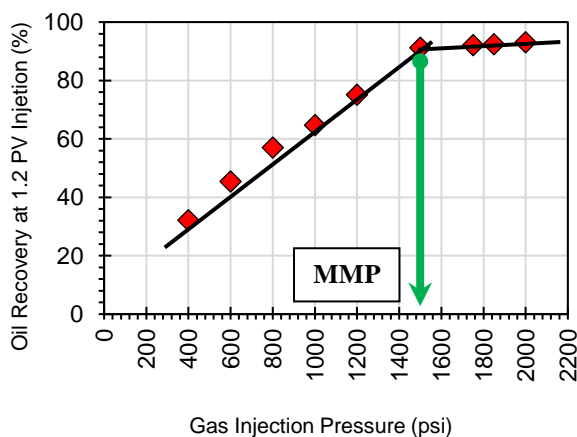


Figure 2. MMP determination example.

The MMPs of CO₂ and N₂ were determined at 70°C to be 1650 psi and 1350 psi, respectively. The lower MMP of N₂ compared to CO₂ can be explained by the fact that as the temperature increased, the viscosity of crude oil decreased, thereby increasing oil stripping and evaporation (Belhaj et al., 2013). Furthermore, when interacting with N₂, the intermediate components in crude oil may have an effect on lowering the MMP of N₂ (Mungan, N, 2000; Necmettin, M., 2003; Belhaj et al., 2013). More details can be found in our previous work (Elturki et al., 2023; 2022). For the huff-n-puff experiments, the setup shown in Figure 3 was used to implement the tests at 70°C. Five cores (1*2 inch) were saturated with crude oil that had the following composition (C₈-C₁₄=65.14%, C₁₅-C₁₉= 6.06%, C₂₀-C₂₄=9.16%, C₂₅-C₂₉=14.48%, C₃₀₊=5.17%). One core was utilized as a standard for pore size distribution and comparative purposes without introducing it to gas pressure. The average helium porosity of cores was 5.7%, and the average permeability was 0.000198 mD. The total organic carbon (TOC) of the cores was 5.5%, determined via Rock-Eval pyrolysis. The XRD results of the cores are shown in Table 1. Two pressures for each gas were selected to conduct the huff-n-puff tests. One of them is below the MMP (i.e., 1000 psi) and the other above the MMP (i.e., 2000 psi). The soaking time was selected to be 6 hours and production time of 60 min. Table 2 summarizes the tests design used in this research. Oil recovery factor can be calculated by the following equation:

$$\text{Oil Recovery Factor (RF)} = \frac{wt_1 - wt_2}{wt_1 - wt_{\text{dry}}}$$

Where: wt_1 is the saturated core weight, wt_2 is the core weight after production time, and wt_{dry} is the core weight when its dry

Table 1. Eagle Ford XRD results

Mineral	Calcite	Quartz	Dolomite	Pyrite	Kaolinite
Composition (%)	70	18	2	1	9

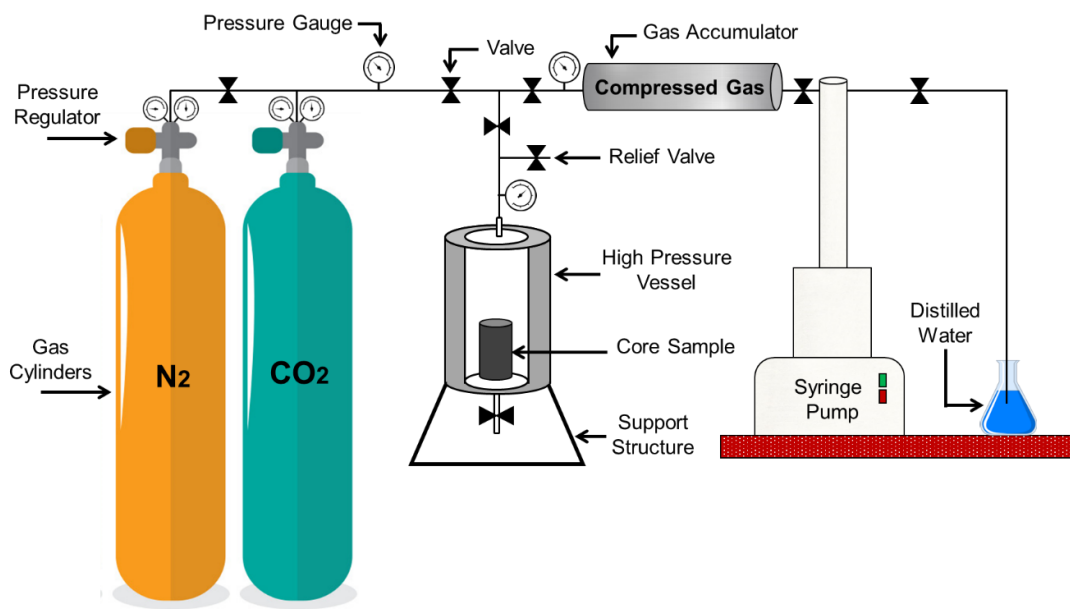


Figure 3. Huff-n-Puff experiments setup.

Table 2. Operating parameters.

Test #	Core #	Pressure, psi	Gas	Soaking time, hr.
1	1	1000	CO ₂	6
2	2	1000	N ₂	6
3	3	2000	CO ₂	6
4	4	2000	N ₂	6
-	5 (reference core)	No pressure applied	-	-

Results and Discussion

The utilization of the huff-n-puff process with CO₂ gas proved to be more efficient in extracting oil from shale cores compared to using N₂ gas. This is because CO₂ has a greater capacity to decrease the interfacial tension, which results in a more effective extraction of oil. There was a higher rate of recovery observed during the first three cycles during immiscible gas injection, after which the rate stabilized or slightly increased. For miscible injection, the recovery increased after the third cycle during CO₂ injection compared with lower oil production during N₂ injection. As an instance, the lowest amount of oil recovery observed through immiscible N₂ injection was roughly 5%, while during CO₂ gas injection, it was about 14%, as shown in Figure 4-A. Conversely, under miscible conditions, there was a higher rate of recovery observed up to 41% after the sixth cycle of CO₂ injection compared to only 16.8% after N₂ injection, as shown in Figure 4-B. These observations provide confirmation that miscible CO₂ injection is more effective than miscible N₂ injection. Achieving miscibility has the potential to improve the performance of both gases in terms of oil recovery. In terms of asphaltene precipitation and pore plugging, the results from the experiments indicated that the oil recovery factor decreased in the later cycles. This finding suggests that the deposition of asphaltene had an immediate effect after the first cycle, but it accumulated over the following cycles. The information conveyed in Figure 5 shows the presence of asphaltene particles located on the core's surface following the final cycle of a 2000 psi CO₂ huff-n-puff process.

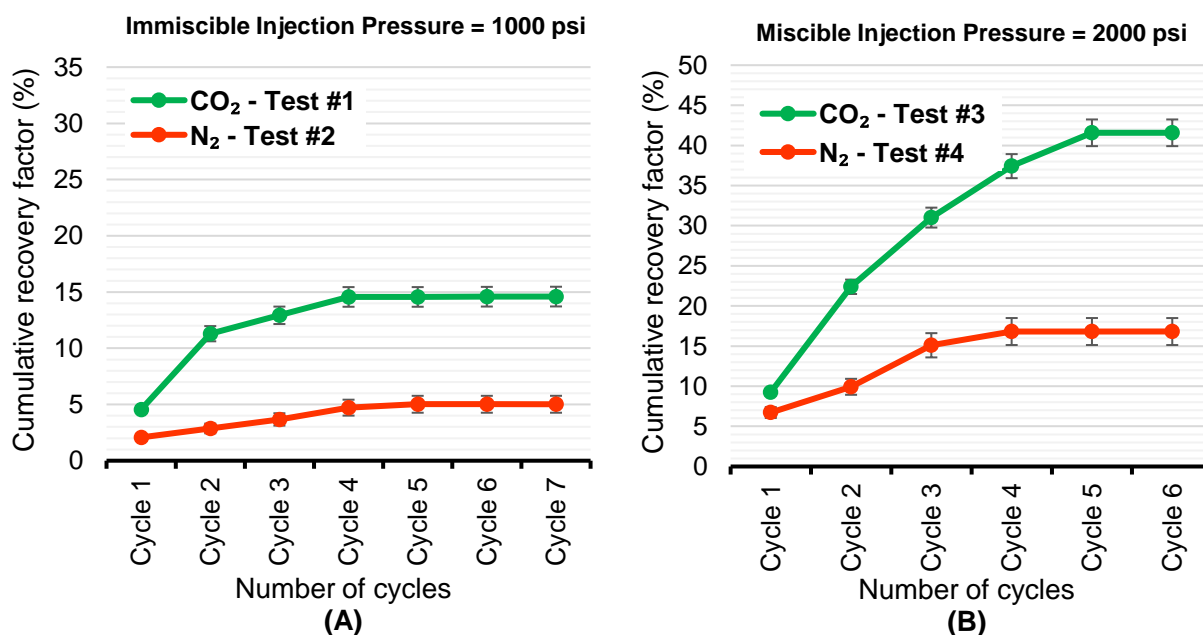


Figure 4. Oil recovery performance during immiscible and miscible gas injections.

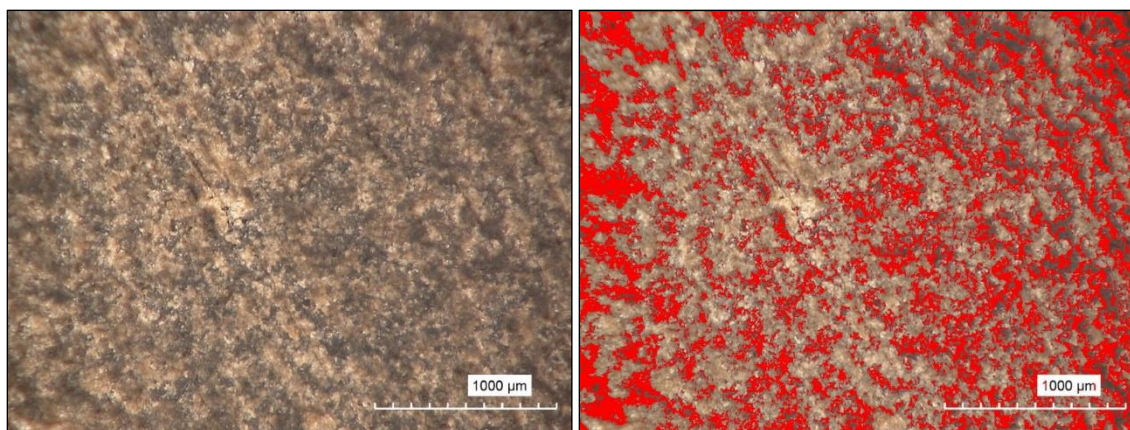


Figure 5. Asphaltene particles on the surface of the core after CO₂ huff-n-puff test. (Left: original photo, Right: highlighted asphaltenes in red)

To compare the pore size distribution after the huff-n-puff tests, a PoreMaster mercury porosimeter was used to determine the change in pore size of the core before and after the tests. Core#5 was saturated with crude oil without applying gas on it. This core was used as a reference and to compare the results of the other cores (after test #3 and #4). All cores were smashed into small pieces to fit the device, as shown in Figure 6. The cores' microstructure pores and throats were assessed by subjecting them to a measurement process where a significant pressure of 60,000 psi was employed. As shown in Table 3, the maximum pore size was found in Core#5 which was about 293.8 µm. After test#3, the pore size was decreased significantly to 208.0 µm compared to 250.0 µm after test#4. CO₂ huff-n-puff process resulted in more pore plugging due to asphaltene particles. These observations confirm that the oil was recovered at a higher rate at the first cycles.



Figure 6. Crashed cores for pore size distribution analysis.

Table 3. Pore size analysis results.

Core #	Gas Applied	Maximum (µm)	Minimum (µm)	Average (µm)
5	-	293.8	0.0046	12.38
3	CO ₂	208.0	0.0037	9.81
4	N ₂	250.0	0.0036	9.45

Conclusions

A comparative experimental investigation was undertaken to investigate the influence of CO₂ and N₂ miscible and immiscible huff-n-puff injection on the oil recovery performance as well as the asphaltene instability in shale cores. The MMP was determined for both gases at 70°C using the slim tube method. After then, two pressures, one above and one below the MMP, were selected in order to evaluate the influence of miscibility on oil recovery. When compared to the miscible N₂ injection, the miscible CO₂ pressure resulted in a higher percentage of oil recovery. Immiscible conditions led to lower oil recovery for both gases. The results indicate that higher levels of recovery were observed in the starting cycles for all applied pressures. In addition, asphaltene particulates began to precipitate in the early cycles and accumulated in the following ones. Also, our results indicated that oil reduction and asphaltene deposition accumulated primarily in the later cycles as there was a decrease in the volume of oil recovered during those cycles. The test for pore size distribution examined the change in pore size of shale cores following cycle experiments for both gases. More research needs to be conducted to expand these laboratory results to actual shale resources and to identify other parameters that may affect the efficacy of such operations in tight-shale reservoirs.

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