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Analysis of Nitrogen Minimum Miscibility Pressure MMP and Its Impact on Instability of Asphaltene Aggregates - An Experimental Study

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Abstract

Minimum miscibility pressure (MMP) is a critical parameter when undergoing miscible gas injection operations for enhanced oil recovery (EOR). Miscibility has become a major term in designing the gas injection process. When the miscible gas contacts the reservoir oil, it causes changes in the basic oil properties, affecting reservoir oil composition and equilibrium conditions. Changes in conditions may also favor flocculation and deposition of organic solids, mainly asphaltene, which were previously in thermodynamic equilibrium. The main purpose of this study is to investigate how the most important parameters, such as oil temperature and oil viscosity, could affect the nitrogen (N_2) MMP and the instability of asphaltene aggregation. Three sets of experiments were conducted: first, the determination of MMP was performed using a slim-tube packed with sand. The impact of crude oil viscosity using 32, 19, and 5.7 cp; and temperature using 32, 45, and 70 °C, were investigated. The results showed that the N_2 MMP decreased when crude oil temperature increased. The temperature is inversely proportional to the N_2 MMP due to the N_2 remaining in a gaseous phase at the same conditions. In terms of viscosity, the MMP for N_2 was found to decrease with the reduction in oil viscosity. Second, the effect of miscibility N_2 injection pressure on asphaltene aggregation using 750 psi (below miscible pressure) and 1500 psi (at miscible pressure) was investigated using a specially designed filtration vessel. Various filter membrane pores sizes were placed inside the vessel to highlight the effect of asphaltene molecules on plugging the unconventional pore structure. The results demonstrated that increasing the pressure increased asphaltene weight percentage. The asphaltene weight percent was higher when using miscible injection pressure compared to immiscible injection pressure. Also, the asphaltene weight percentage increased when the pore size structure decreased. Finally, the visualization of asphaltene deposition over time was conducted, and the results showed that asphaltene particles started to precipitate after 2 hours. After 12 hours, the colloidal asphaltenes were fully precipitated.

Introduction

The decline of oil recoveries has motivated oil companies to develop new technologies to extract the remaining oil from oil reservoirs. Various methods were developed to increase the recovery for both conventional and unconventional reservoirs. The most common non-thermal, enhanced oil recovery method is gas injection. The gas (i.e., CO_2, N_2, and natural gas) is injected into the reservoir oil to create the miscibility. Miscible gas flooding has been widely used to increase oil recovery for many years. Gas injection above the MMP has a significant impact on improving oil recovery from the target reservoir. MMP can be defined as the lowest pressure at which a gas can create miscibility with the reservoir oil at the reservoir temperature. In other words, MMP is the lowest pressure at which miscibility between the injected gas and reservoir oil is achieved when the interfacial tension between oil and gas vanishes after multiple contacts. To develop multi-contact miscibility and displacement in the reservoir, the gas must be injected at a pressure higher than the MMP. However, if the reservoir pressure is below the MMP, oil recovery decreases due to immiscible displacement. There is no interfacial tension between the fluids at the MMP. Gas miscibility can be developed at or above the MMP through a vaporizing process, a condensing process, or a combination of the two processes (Elsharkawy et al., 1992). The injected gas diffuses into the reservoir oil, and the oil swells with a decrease in its viscosity. The oil recovery increases once the capillary forces are eliminated from the miscibility phenomenon. Pressure, temperature, oil and gas properties, and gas type have an intrinsic impact on miscibility. Reaching miscibility is often limited by the reservoir temperature and pressure. Vahidi et al. (2007) conducted a simulation study and concluded that the most important factor to reach miscibility between N_2 and crude oil is the amount of light and intermediate components in the oil. They demonstrated that MMP decreases when decreasing the methane content and increasing the intermediate components.

Slim-tube is the primary experimental technique to determine the MMP. The slim-tube test has been conducted for decades to measure MMP because it simulates the one-dimension displacement of reservoir oil (Ekundayo et al., 2013; Amao et al., 2012). Increasing oil recovery can be more efficient by developing miscibility when the intermediate components of oil vaporize, thus the miscible bank is created. However, changes in the reservoir's condition such as oil composition and pressure could lead to asphaltene instability and thus asphaltene precipitation, which causes severe problems, such as pore plugging, during oil production processes (Elsharkawy et al., 1992; Jamaluddin et al., 2002; Mansoori et al., 2010; Moradi et al., 2012; 2019; Soroush et al., 2014; Khalaf et al., 2019; Elturki et al., 2020a; Fakher et al., 2019a; Fakher et al., 2019b). The effects of slim-tube length and the injection flow rate were reported (Flock et al, 1984; Hudgins et al., 1990; Sebastian et al., 1992). Flock et al. (1984)conducted 24 tests using different slim-tube lengths. They concluded that the longer length of the slim-tube, the higher oil recovery for miscible displacement but not immiscible tests. Moreover, oil recovery will decrease as the flow rate increases. Ekundayo et al. (2013)suggested using moderate flow rates of the injection gas, small diameter, and long coil length to avoid unwanted problems such as fingering, transitions zone length, and transverse compositional variations.

Most of the reported studies focused on using CO_2 miscible gas, especially in unconventional reservoirs. Sanger et al. (1998)conducted slim-tube experiments and numerical simulations of the displacement of a model gas condensate at reservoir conditions by both N_2 and methane. Their results showed that N_2 is an efficient alternative to gas cycling in condensate reservoirs. Belhaj et al. (2013)identified various obstacles that affect the success of the CO_2 process, which are the availability of CO_2, well and surface facilities corrosion, environmental constraints, and high CO_2 cost. Fakher et al. (2019)conducted experiments using nanocomposite filter membranes to investigate asphaltene precipitation during carbon dioxide injection. Their study concluded that oil recovery increased when increasing the pore size; thus, asphaltene weight percent was decreased. Ghorbani et al., (2020)studied the effect of asphaltene content over the CO_2 MMP of oil/gas systems, and they concluded that, based on slim-tube results, increasing the asphaltene content in oil resulted in an increase in MMP. However, N_2 has not been extensively studied for use as a miscible solvent as CO_2. Therefore, there is not enough information on how N_2 acts as miscible in the laboratory or in field conditions. This present research aims to investigate how crude oil temperature and viscosity impact the MMP of N_2 and to also identify the asphaltene amount that could be created as a result of the N_2 gas miscibility.

N_2 as an EOR Agent

N_2 injection is one of the proven technologies to increase recovery. N_2 is usually cheaper than CO_2 gas displacement EOR applications and it's not corrosive. N_2, in terms of chemical properties, is inert and not miscible with the formation fluids that will be treated, so there are no chemical reactions or interference. Also, N_2 can be used to energize most fluids and the pH levels will not be affected. The volume of gas is not easy to determine because N_2 under pressure has different volumes. For some high-pressure oil reservoirs, N_2 could be suitable to achieve miscibility conditions. Miscible N_2 gas injection was used in many field projects such as Jay Field, Florida, and Painter Field, Wyoming (Christian et al., 1981; Clancy et al., 1985). Also, some successful projects were reported in East Binger Field, Oklahoma, and Lake Barre Field, Louisiana (Peterson, 1978). The largest field scale that used CO_2 and N_2 gas injection was in the Devonian reservoir of field Block 31 in west Texas. According to (Guzmann, 2014), the world's largest N_2 injection project since the year 2000 is in Cantarell, Mexico. The production was increased from 1 million barrels per day in 1996 to 2.2 million in 2014 (Daltaban et al., 2008). Based on the aforementioned, gas injection has been implemented to increase the oil recovery utilizing the drive mechanisms of gas. Menouar (2013)summarized gas drive mechanisms in the literature based on the reservoir pressure, the temperature, and the oil composition, as shown in Table 1.

Table 1—Mechanisms involved in each of the four regions (Menouar, 2013)

Region		Mechanisms
I	Low pressure	▪ Swelling and Viscosity Reduction
II	Intermediary pressure, High Temperature	▪Swelling, Viscosity Reduction, and Crude Vaporization
III	Intermediary pressure, Intermediary Temperature	▪ Dynamic Miscibility Effect, Viscosity Reduction, and Mobility Effect
IV	High Pressure High Temperature	▪ Multiple contact Miscibility, Viscosity Reduction, and Vaporization Effect

The asphaltene precipitation and deposition are major problems during gas injection processes, which result in pore plugging and permeability reduction (Elturki et al., 2020b). Few studies were reported using N_2 as an EOR agent to investigate the asphlatene problems during N_2 gas injection. Jamaluddin et al. (2002)contacted various molar concentrations of N_2 with the reservoir fluid to investigate the instability of asphaltene. Their results showed that increasing the concentration of N_2 aggravated the instability of asphaltene and the increased the bulk precipitated amount. Moradi et al. (2012)studied asphaltene particle precipitation, aggregation, and breakup using natural depletion and miscible N_2 injection processes. Using high-pressure filtration, it was observed that N_2 injection stabilized asphaltenes. The change in pressure and temperature aggregates the asphaltene particles and thereby increase the precipitation in the reservoir, which reduces the recovery (Fakher et al., 2018). Figure 1 shows the precipitation and deposition of asphaltene molecules due to alteration of reservoir thermodynamic equilibrium.

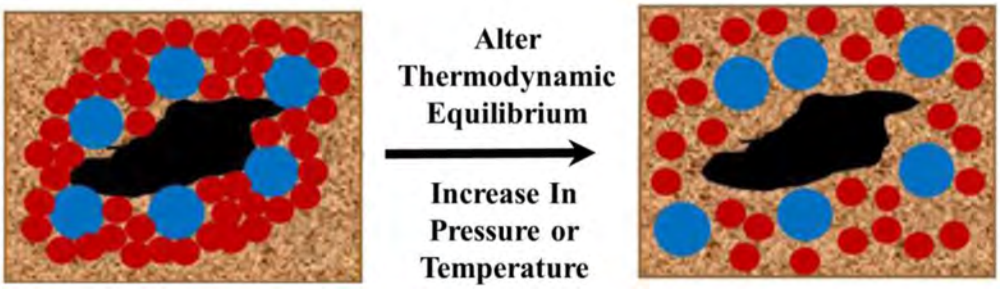

Figure 1—Precipitation and deposition of asphaltene molecule due to alteration of reservoir thermodynamic equilibrium, the black color shows the asphaltene, the red indicates the oil molecules, and the blue shows the resins (Fakher et al., 2018)

Experimental Apparatus and Materials Description

N_2 MMP Experiment Apparatus

In this study, crude oils with viscosities 32, 19, 5.7 cp were used to perform the experiments. The viscosities were measured using the rheometer. Gas Chromatography-Mass Spectrometry was used to determine the composition of the crude oil, as shown in Table 2. Various temperatures of 32, 45, and 70 °C were used to investigate the effect on MMP. Figure 2 shows a schematic diagram of the slim-tube experimental setup for measuring the MMP using N_2. The main components of the slim-tube test include a syringe pump, an oven that was used to investigate the effect of various temperatures on MMP during N_2 injection, three accumulators, a N_2 gas cylinder for N_2 injection, a stainless-steel slim-tube packed with sands, and a pack pressure regulator. The slim-tube tests were divided into several steps, starting with the pre-test preparation in which the slim-tube was filled with dry sand. This step was important to measure the weight of the slim-tube before it was saturated with water; the weight was compared with the same slim-tube when it was first saturated with water to calculate the pore volume. The second step was to fill the slim-tube with the crude oil at a low rate to ensure that the slim-tube is fully saturated with oil. The final step was to conduct the experiment by adjust the temperature, filling the accumulator with gas, and pump the gas into the slim-tube. The obtained MMP will be used in filtration experiments to evaluate the asphaltene precipitation during N_2 injection.

Table 2—Crude oil composition

Component	Weight percentage (%)
C_8-C_{14}	65.14
C_{15}-C_{19}	6.06
C_{20}-C_{24}	9.16
C_{25}-C_{29}	14.48
C_{30+}	5.17
Total	**100.00**

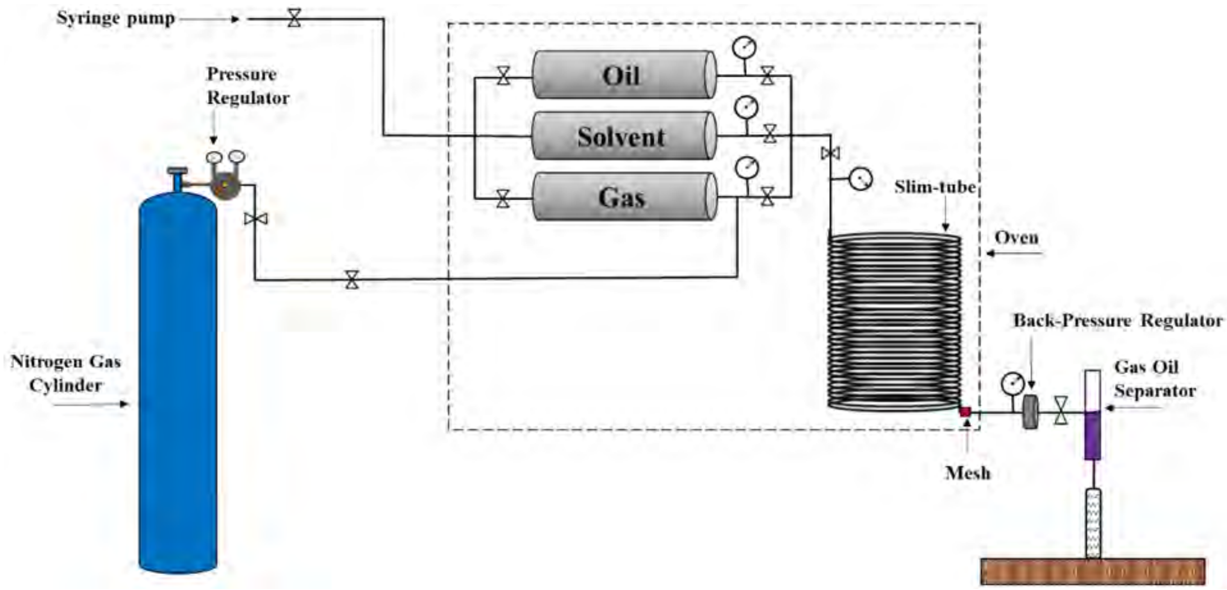

Figure 2—Schematic diagram of the slim-tube apparatus.

N_2 MMP Experimental Procedure

The following steps were followed to determine the MMP:

1. First, the slim-tube was packed with sand and then it was weighted. Then, the slim-tube was saturated with water using a syringe pump at a low rate of 0.10 ml/min to ensure that the slim-tube was fully saturated, and the slim-tube was weighed again. The pore volume was calculated by figuring the difference of the two weights
2. The oven was adjusted to the specified temperatures to study the effect of various temperatures.
3. The crude oil, then, was made ready in the oil accumulator and was injected by rate of 0.5 PV into the slim-tube.
4. A back-pressure regulator was placed at the outlet of the slim-tube and adjusted to the required back pressure to let the crude oil pass through the slim-tub e. The backpressure was created using a syringe pump.
5. The accumulator was filled with N_2, then was injected into the slim-tube at a low flow rate of 0.25 ml/min.
6. Each experiment was stopped when 1.2 PV of N_2 was injected or the gas broke through from the outlet of the slim-tube.
7. The oil recovery at the outlet was collected then plotted with the pressures to determine the MMP. The MMP was estimated when the cumulative oil recovery was equal to or exceeded 90% of OOIP
8. Xylene solvent was then pumped into the slim-tube with a slow flow rate to ensure that all oil was removed.
9. Before filling the new sample of oil for the next experiment, water was used to remove the trapped Xylene in the slim-tube, so it would not affect the next test
10. Finally, a new oil sample was pumped into the slim-tube to repeat the experiment.

Asphaltene Aggregation Experiments Apparatus

After determining the MMP of N_2, asphaltene deposition, and precipitation during N_2 gas injection was evaluated. Asphaltene deposition and precipitation is the most challenging problem during gas injection EOR methods. Asphaltenes can be defined as a high molecular weight substance of crude oil that is soluble in toluene, but insoluble in alkanes. Asphaltene can cause pore plugging in the reservoir and production facilities. A specially designed filtration vessel was used to fit the filter paper membranes and to investigate the effect of immiscible N_2 injection pressure (750 psi) and at miscible pressure (1500 psi) on asphaltene aggregation. Three sets of filter membranes sizes were used (450, 100, and 50 nanometers) to study the impact of varying the filter membrane. The order was 450, 100, and 50 nm from the top to the bottom of the specially designed filtration vessel, as shown in Figure 3. Asphaltene weight percentage is calculated by weighing the filter paper before and after the filtration process. The difference between weights can then be used to determine the asphaltene percentage weight. Following the filtration experiments, asphaltene visualization experiments were conducted to investigate the effect of pressure on asphaltene deposition over time. A solvent of heptane was used to dissolve the oil samples in tubes to quantify the asphaltene weight percent after each experiment. The asphaltene percentage weight was calculated using the following equation:

$$\text{Asphaltene wt}\,\% = \frac{\text{wt asphaltene}}{\text{wt oil}} * 100$$

Where:
Asphaltene wt% Asphaltene weight percentage
Wt asphaltene Asphaltene weight on the filter paper
Wt oil Oil sample weight

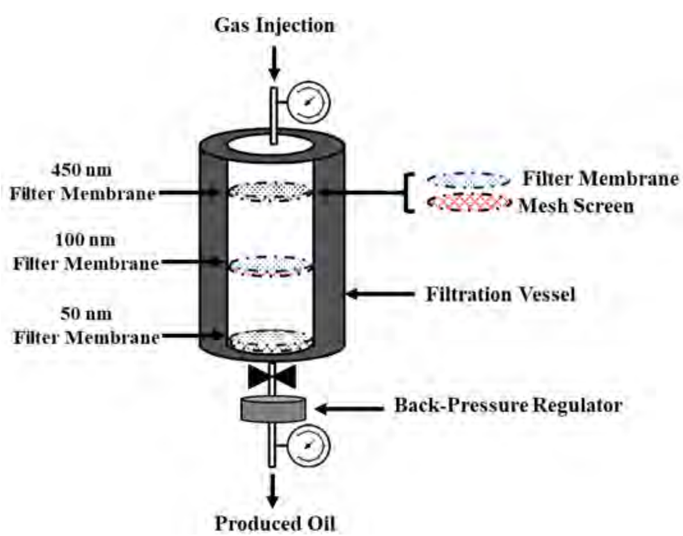

Figure 3—Components of the specially designed filtration vessel.

Filtration Experimental Procedure

The following steps were followed for all filtration experiments:

1. The first set of mech screens, filter membrane papers, rubber O-rings, and spacers were placed inside the filtration vessel, respectively. This step was repeated for the next two sets.
2. The vessel was closed tightly using a specially designed cap to tighten all the sets together and prevent any possible leakage during the experiment.
3. A crude oil of 30 ml was poured into the accumulator. A syringe pump was used to inject the oil into the vessel.
4. N_2 was injected into the cylinder to reach the desired level. The crude oil was then exposed to the gas for a predetermined soaking time.
5. After 2 hours of soaking time, the syringe pump at the outlet was turned on to constant pressure, and then it was adjusted to the required backpressure for each experiment to let the crude oil pass through the membranes.

6. N_2 was injected into the vessel, and the produced oil was collected. The experiment was stopped when no farther oil production was observed.
7. The inlet and outlet pressures were observed and recorded using transducers that connected to a computer. The difference between the two pressures did not exceed 50 psi.
8. After gas injection was completed, the vessel was opened, and the remaining crude oil was collected from each filter membrane for analysis.

Results and Discussion

Results of N_2 MMP and filtration experiments are discussed in this section.

Effect of Temperature on N_2 MMP

In this section, the effect of temperature on N_2 MMP is examined. In the oil and gas industry, the temperature has a strong impact in many areas including production, design, and recovery improvements. Three temperatures were selected (32, 45, and 70 °C) to investigate the effect of temperature on oil viscosity of 19 cp in all experiments. The oven was used to adjust the temperature. Oil recoveries were plotted with N_2 gas injection pressures to estimate the MMP. The results demonstrated that at 32 °C, the N_2 MMP was 1600 psi, as shown in Figure 4. Increasing the temperature to 45 °C lead to a slight decrease in MMP, which was 1500 psi, as shown in Figure 5. Figure 6 presents a temperature of 70 °C with lower N_2 MMP determination, which was 1350 psi. The higher the temperature, the lower the MMP because the temperature decreased the oil viscosity; thus enhancing the vaporization. The temperature is inversely proportional to the N_2 MMP due to the N_2 remaining in the gaseous phase at the same conditions.

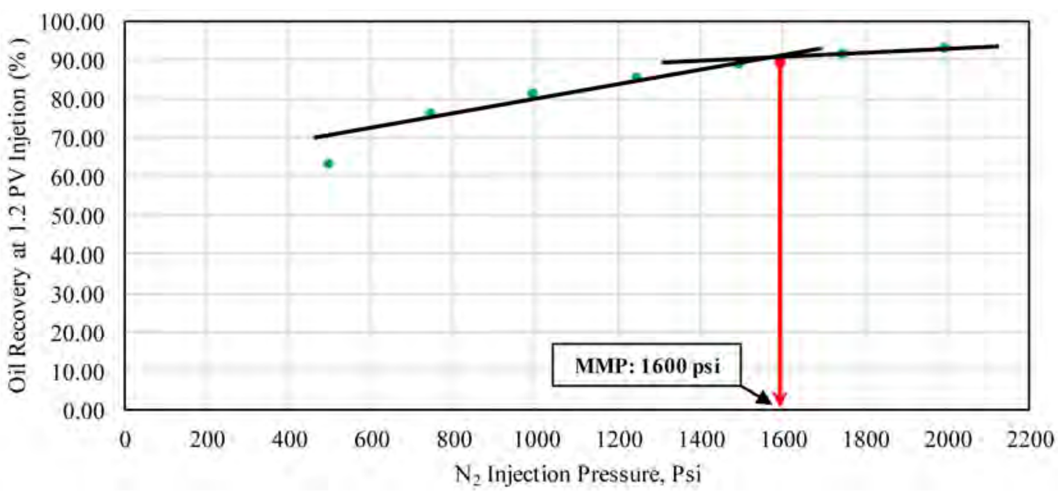

Figure 4—N_2 MMP determination using 19 cp oil viscosity at 32 °C

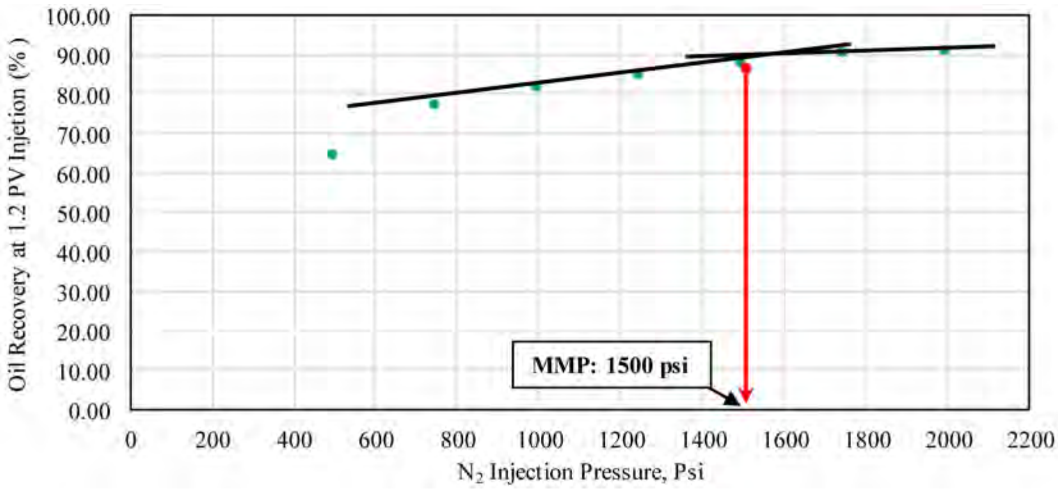

Figure 5—N_2 MMP determination using 19 cp oil viscosity at 45 °C

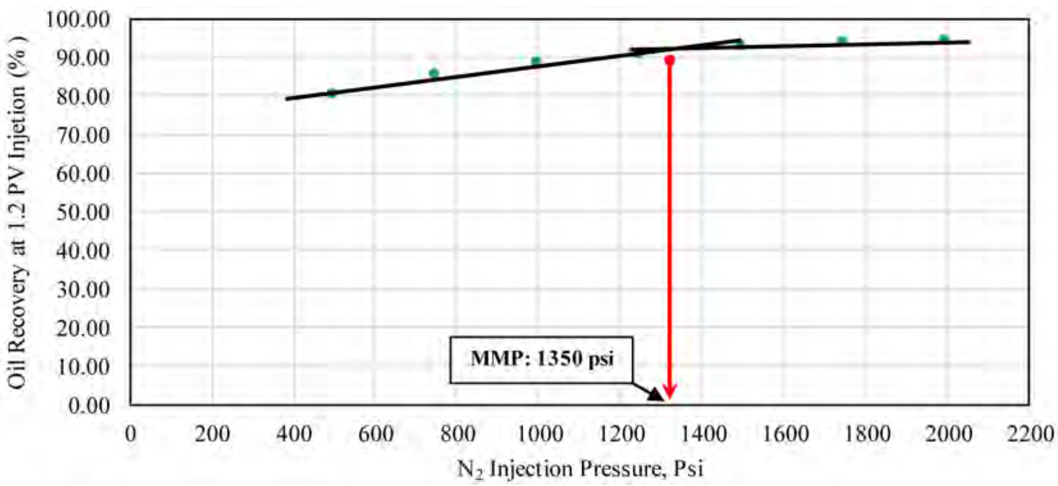

Figure 6—N_2 MMP determination using 19 cp oil viscosity at 75 °C

Effect of Viscosity on N_2 MMP

In this section, two experiments were implemented to investigate the effect of viscosity on N_2 MMP using 32 and 5.7 cp oil viscosities at 32 °C. Oil viscosity is an important factor that affects oil recovery. Changes in oil viscosity will affect the vaporization phenomenon that occurs during the gas displacement process. Studying the effect of viscosity on MMP helps to design the gas injection process. After collecting oil recoveries and plotting them with N_2 injected pressures, the results demonstrated that when using the oil viscosity of 5.7 cp, the MMP was 1100 psi, as shown in Figure 7. Increasing the oil viscosity to 32 cp resulted in lower oil recovery, which was less than 90% of OIIP. Various studies stated that MMP can only determine if the oil recovery is more than 90%. The higher pressure that was implemented in the laboratory was 2000 psi; thus, the determination of N_2 MMP using 32 cp was not achieved, and the MMP was determined to be >2000 psi (Figure 8). Figure 9 summarizes the N_2 MMP using different temperatures and oil viscosities.

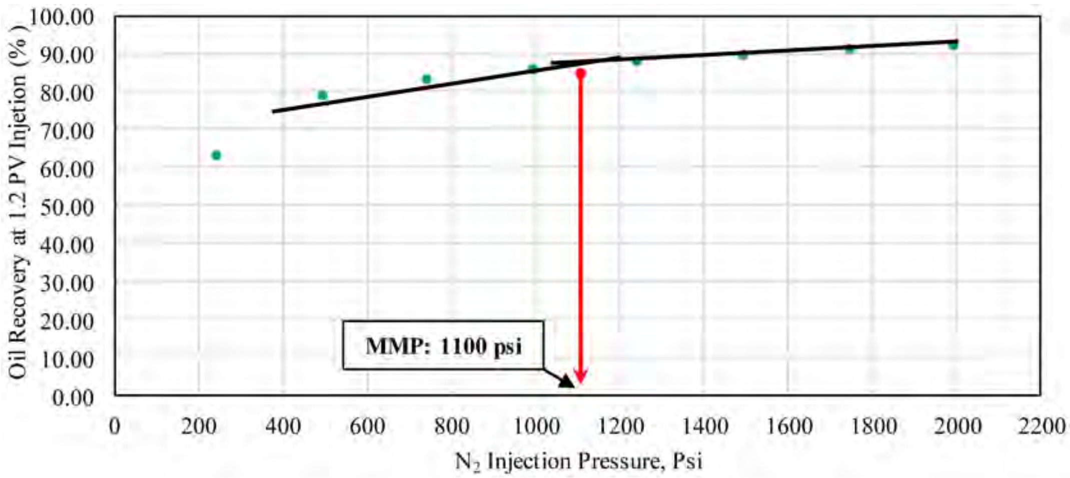

Figure 7—N_2 MMP determination using 5.7 cp oil viscosity at 32 °C

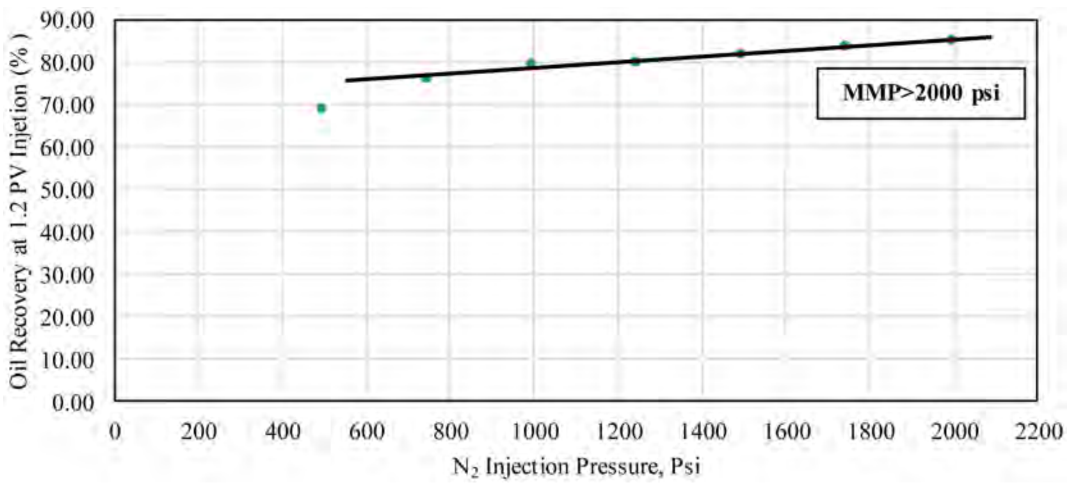

Figure 8—N_2 MMP determination using 32 cp oil viscosity at 32 °C

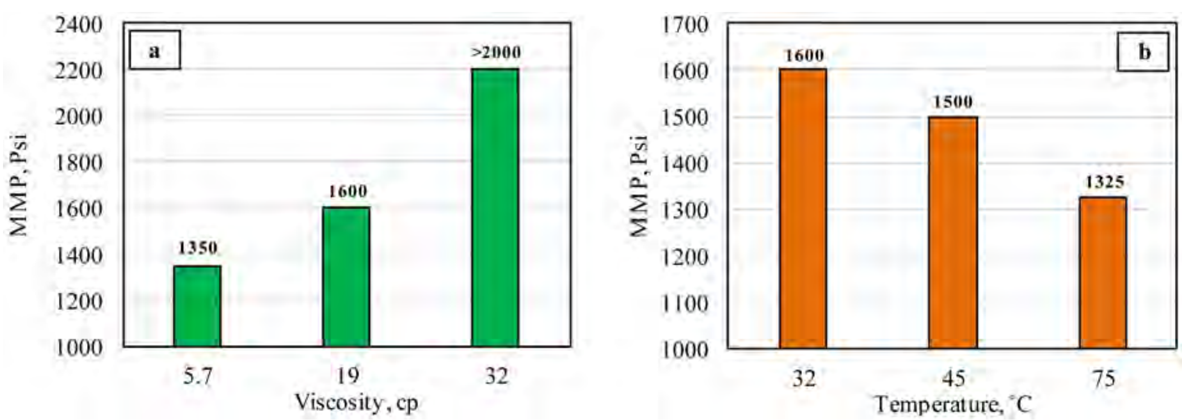

Figure 9—N_2 MMP summary using different a) Oil viscosities at 32 °C b) Temperatures using 19 cp oil.

Figure 10 reveals the cumulative oil recoveries collected from all the experiments. More than 50% of oil inside the slim-tube was recovered using 500 psi in all experiments. By increasing the pressure, the produced oil became much lower than the oil recovery from the original injection pressure. For an instant, the recovery factor increased from 108.5 ml to 110.5 ml (91.03% to 92.71% of OOIP), as shown in Figure 11, when the pressure increased from 1750 psi to 2000 psi, respectively, at 32 °C using 19 cp crude oil. The temperature had a significant impact on oil recoveries, and this was obvious when using 75 °C, which the recovery was 95.4 ml (78% of OOIP) using 500 psi. This can be explained by the increase in temperature turned to reduce the oil viscosity; thus, increasing the oil recovery. Generally, higher viscosity reduces the oil recovery, and a higher portion of crude oil was trapped between the sand grains inside the slim-tube. The oil produced when using crude oil with a viscosity of 32 cp is higher than the recovery from oil with viscosity of 19 cp, when the N_2 is injected at 500 psi. With 2000 psi injection pressure, the oil recovery of 32 cp was much lower than the 5.7 and 19 cp. This can be explained because heavy oil requires higher pressure to produce and to achieve the miscibility, as explained in the previous section.

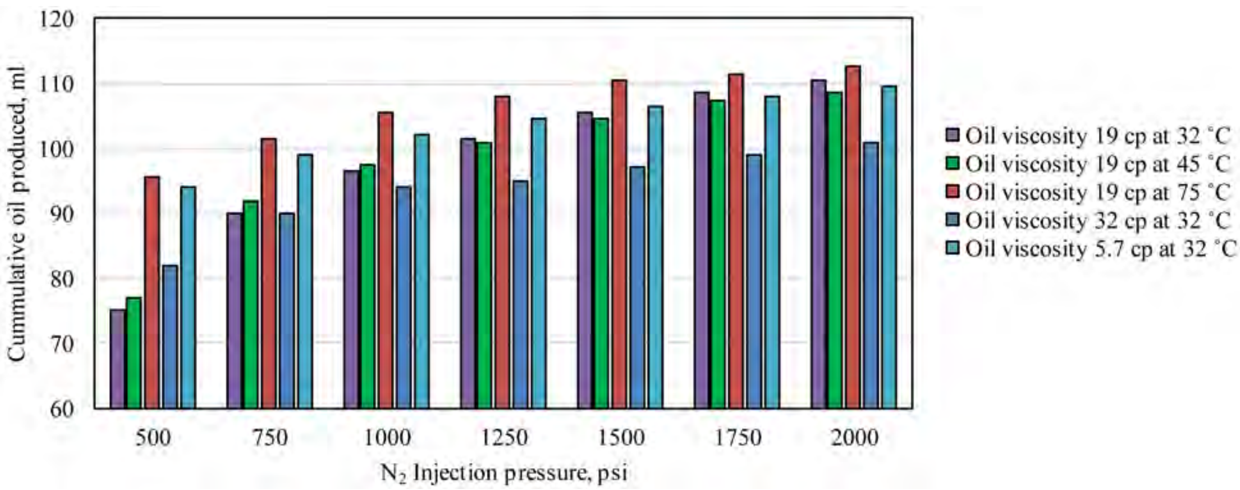

Figure 10—Cumulative oil recoveries vs. N_2 injection pressure for all experiments.

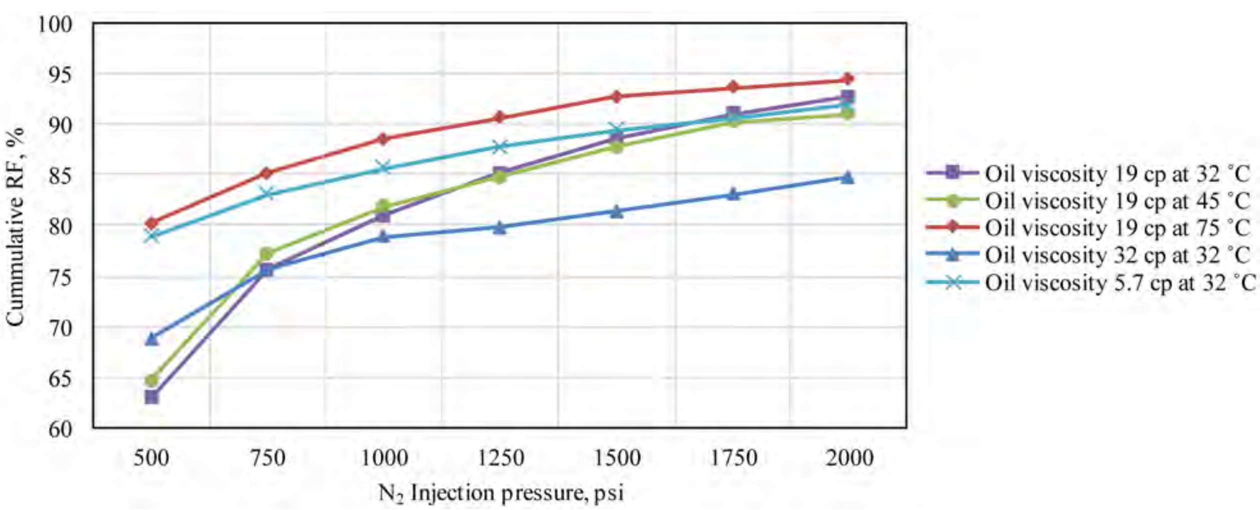

Figure 11—Cumulative recovery factor vs. N_2 injection pressure for all experiments.

Asphaltene Aggregation Experiments Results

In this section, filtration and visualization experiments are discussed.

Filtration Results

Using the data obtained from the miscibility pressure experiment (Figure 5), two N_2 injection pressures of 750 (below miscible pressure), and 1500 psi (at miscible pressure) were investigated. Two hours of soaking time were implemented to let the oil inside the vessel be exposed to N_2 gas for enough time. Heterogeneous order was selected to investigate the effect of N_2 gas injection on pore size structures. The upper part of the filter membrane was 450nm, followed by 100 nm, and the bottom filter membrane was 50nm. Figure 12 shows the asphaltene weight percent when a heterogeneous paper membrane distribution was used during various N_2 injection pressures. The results demonstrated that the asphaltene weight percent increased when the pressure increased. This is due to the injection N_2 pressure aggregates the asphaltene resins; thus, the bonds between asphalting molecules and resins weakened. This resulted in asphaltene deposition on the filter membranes. As the pore size of filter membranes decreased, the oil could not pass through pores easily; thus, an increase in the asphaltene deposition resulted. The molecules of asphaltene that had a size of more than 450 nm plugged the pores of the 450 nm filter membrane. The asphaltene molecules that had sizes smaller than 450 and larger than 100 nm precipitated on a 100 nm filter membrane. Thus, the asphaltene that participated on the filter membranes of 450 nm, 100 nm, and 50 nm had sizes larger than 450 nm, 450-100 nm, and larger than 100 nm, respectively. Given these observations, asphaltene could result in pore plugging in the reservoir during production and cause severe issues during production operations.

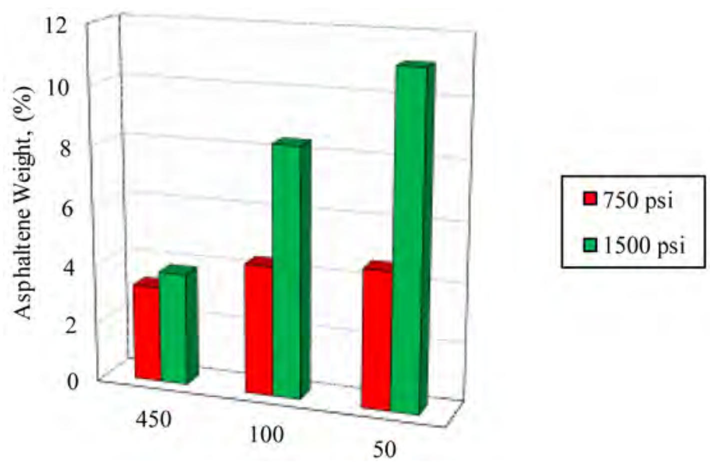

Figure 12—Asphaltene weight percent during 750 and 1500 psi of N_2 injection

Visualization of Asphaltene Deposition Results

The asphaltene disposition processes over time is presented in this section. The remaining oil above each filter membrane was collected after each experiment and dissolved in heptane at a ratio of 1:40. The pictures were captured at various times including 0, 2, 4, and 12 hours to investigate and visually capture the asphaltene deposition process. With 0 hours of visualization time, all the test tubes showed almost the same colors, and no asphaltene particles were observed. After 2 hours, the observed results were that most of the asphaltene particles were in the colloidal condition in the solvent of heptane. The asphaltene started to form and slowly deposit. After 4 hours, the asphaltene fluctuations started to deposition faster, and the suspended particles of asphaltene started to deposition down of the test tube in all filter membrane samples. This can be observed clearly in the bottom of the test tube. As time progressed, the asphaltene particles deposited more in the test tube. After 12 hours, it was not clear in the above part of the test tube the asphaltene particles because most of the particles were deposited. Although the lowest part of test tubes looked the same, the asphalted weight was different. These results showed that asphaltene deposition takes time until fully deposited. Also, higher pressure showed slightly faster deposition and precipitation, especially in the 50 nm filter membrane. The results give more understanding and indications of how the asphaltene deposition in a real reservoir. Better understanding of how the asphaltene deposit and precipitate in the real reservoir can lead to avoiding related problems of asphaltene before they occur in oil production or operation processes.

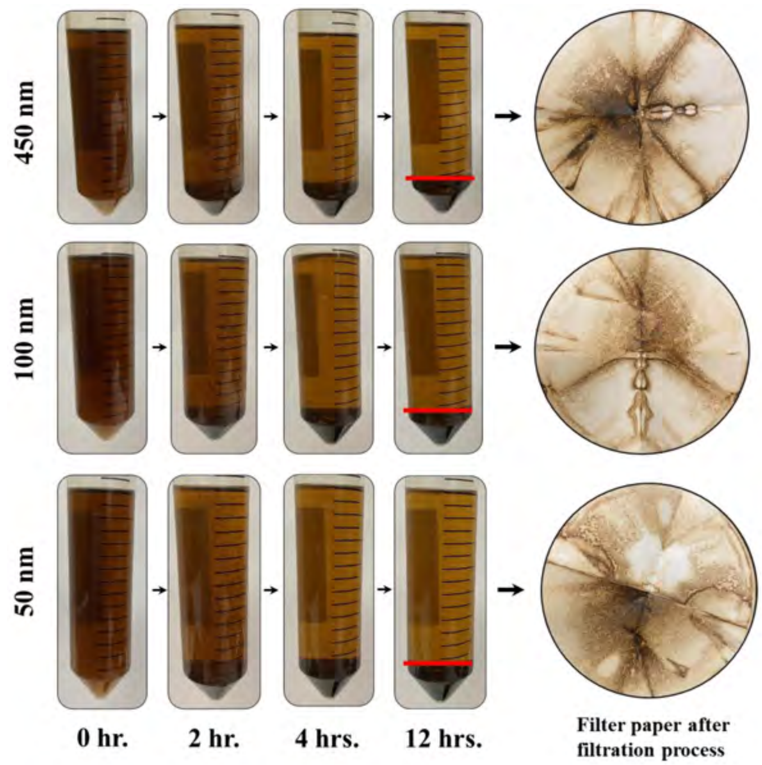

Figure 13—Visualization of asphaltene precipitation and deposition at 750 psi

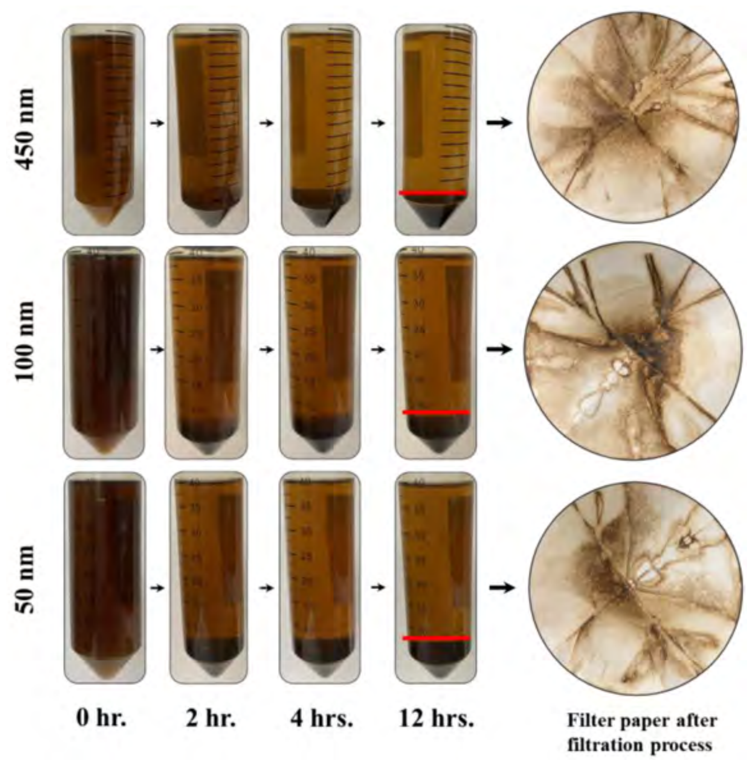

Figure 14—Visualization of asphaltene precipitation and deposition at 1500 psi

Conclusions

Three sets of experiments were conducted in this research, which investigated the MMP of N_2 and filtration and visualization of asphaltene. The MMPs of N_2 were determined using a slim-tube as a function of temperature and viscosity of crude oil. The filtration experiments were conducted using various filter membranes to study the effect of N_2 injection on asphaltene aggregation. The following conclusions were drawn:

- The results demonstrated that increasing temperature resulted in a decrease in N_2 MMP. The MMP decreased with the decrease in oil viscosity, due to the reduction in interfacial tension between the fluids when the oil viscosity was decreased. Higher pressures are required to determine the MMP for high oil viscosities.
- The filtration experiments showed that as the pore size decreased of membranes, asphaltene clusters were unable to pass. As a result, an increase in asphaltene precipitation and deposition was observed.
- The filtration experiments showed that at a pressure below the MMP (750 psi) the asphaltene weight percent was lower than the pressure at MMP (1500 psi). This indicates that miscibility leads to more asphaltene issues during production processes.
- Asphaltene visualization experiments demonstrated that asphaltene particles started to precipitate after 2 hours. After 12 hours, the colloidal asphaltenes was fully precipitated.

Nomenclature

MMP	Minimum Miscibility Pressure
PV	Pore Volume
ml	Milliliter
cp	Centipoise
nm	Nanometer
°C	Celsius

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