

An Experimental Investigation of Asphaltene Aggregation Under Carbon Dioxide Injection Flow in Ultra-Low-Permeability Pore Structure

Mukhtar Elturki and Abdulmohsin Imqam, Missouri University of Science and Technology, USA

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Abstract

One of the major problems during gas injection in unconventional reservoirs is asphaltene precipitation and deposition. Asphaltenes can reduce the pore throat in the reservoir and plug the surface and subsurface equipment during the production process, thus, result in oil production reduction with significant financial consequences. The impact of carbon dioxide (CO₂) gas injection on asphaltene deposition in unconventional reservoirs still poorly investigated. This research investigates the impact of CO₂ gas injection on asphaltene aggregation in ultra-low-permeability pore structures, mainly present in unconventional shale resources. First, the minimum miscibility pressure (MMP) of crude oil with CO₂ was determined using the slim tube technique. Then, several CO₂ injection pressures were selected to conduct the filtration experiments using a specially designed filtration apparatus. All pressures selected were below the MMP. Various sizes of filter paper membranes were used to study the effect of pore structure on asphaltene deposition. The results showed that asphaltene weight percent was increased by increasing the pressure and a significant asphaltene weight percentage was observed on smaller pore size structures of the filter membranes. The visualization tests revealed the process of asphaltene precipitation and deposition and showed that asphaltene particles and clusters were precipitated after one hour and fully deposited in the bottom of the test tube after 12 hours. High-resolution photos of filter paper membranes were presented using microscopy imaging and scanning electron microscopy (SEM) analysis; these photos highlighted the asphaltene particles inside the filter paper membranes and pore plugging was observed. The study's findings will contribute to a better understanding of the main factors influencing the stability of asphaltene particles in crude oil under immiscible CO₂ injection pressure, particularly in nano pores, which are predominant in shale unconventional resources.

Introduction

Gas injection has been frequently used in the oil sector in order to boost oil recovery. Even though hydraulic fracturing techniques and horizontal wells could increase the oil production for shale reservoir, oil 4 to 6% can be recovered (Unal et al., 2019; Elturki and Imqam, 2020a; Eltaleb et al., 2020; 2021; Awad et al., 2020; Biheri et al., 2020; 2021a; 2021b; 2021c; Elturki et al., 2021). Reduced oil viscosity, oil swelling impact, and gas-oil characteristics, such as lower interfacial tension, are some of the essential processes by gas injection technology (Shuker et al., 2012; Godec et al., 2013; Liu et al., 2017). In unconventional

reservoirs, gas injection EOR technologies have been examined, and the findings have demonstrated an increase in oil recovery. Recently, laboratory experiments have demonstrated a major impact in enhancing oil recovery from conventional and unconventional resources using cyclic and continuous CO₂ injection (Yu et al., 2016; Elwegaa et al., 2019; Fakher and Imqam, 2020a; Zhang et al., 2021; Li et al., 2021; Tovar et al., 2021; Milad et al., 2021). During the production and processing of oil, asphaltene precipitation and deposition present massive issues including pore plugging of reservoirs and wettability change, leading to reduced oil production (Mahdavi et al., 2015). Asphaltene is a high-molecular-weight molecule derived from crude oil that is soluble in toluene but not in alkanes. Because mobility was attained, high molecular weight components in the crude oil, such as asphaltenes, might be precipitated from the oil phase during gas injection and deposited into the pores, resulting in decreased oil recovery (Escobedo and Mansoori 1997; Wang et al. 2016). Saturates, aromatics, resins, and asphaltenes are the significant components of crude oil, with all components held together by resins (Punase et al. 2016). Any change in the equilibrium conditions, such as pressure and temperature, can lead to breakdown of the bonds between the resins and asphaltenes and thus cause asphaltene deposition inside the pores (Zanganeh et al., 2012; Zendehboudi et al., 2014; Tavakkoli et al., 2014).

In recent years, the effect of asphaltene precipitation and deposition in reservoir's pores and their impact on oil recovery has gained considerable interest (Negahban et al., 2005; Alizadeh et al., 2011; Ashtari et al., 2011; Temizel et al., 2016; Telmadarreie et al., 2017; Gharbi et al., 2017; Hassanpouryouzband et al., 2017; Mohebbinia et al., 2017; Fakher and Imgam, 2018; Al-Hosani et al., 2020; Fakher and Imgam, 2020b; Fakher and Imqam, 2020c; Carvalhal et al., 2021; Huang et al., 2022). Many studies have been conducted on the impact of asphaltene deposition and precipitation on pore plugging in conventional oil reservoirs and wettability change of the rocks during CO₂ injection (Amin et al., 2011; Zadeh et al., 2011; Mansur et al., 2012; Alta'ee et al., 2012; Taheri-Shakib et al., 2018; Alizadeh et al., 2014; Zanganeh et al., 2018; Mehana et al., 2019; Sarsito et al., 2021; Lo et al., 2022). Others investigated the asphaltene aggregation under nitrogen (N₂) gas injection (Jamaluddin et al., 2002; Wang et al., 2018; Khalaf et al., 2019). Unconventional reservoirs, on the other hand, have seen less investigations (Srivastava et al., 1999; Bagheri et al. 2011; Moradi et al., 2012; Buriro et al., 2012; Buriro et al., 2013; Alizadeh et al., 2014; Tavakkoli et al., 2015; Shen et al., 2016; Luo et al., 2017; Zanganeh et al., 2018; Golshahi et al., 2019; Fakher and Imqam, 2019; Dashti et al., 2020; Elturki and Imqam, 2021a; b; c) Srivastava et al. (1999) studied the impact of CO₂ flooding on asphaltene deposition, and they found that the asphaltene fluctuations increased linearly with the concentration of CO₂ after the onset. Bagheri et al. (2011) conduced an experimental study to compare the effect of lean gas and CO₂ on asphaltene deposition process using different production schemes. Their findings showed that higher mole percentage and higher pressure resulted in an increase in asphaltene precipitation and deposition, especially during lean gas injection. Moradi et al. (2012) conducted an experimental study to evaluate the effect of N₂ gas on asphaltene precipitation and their results showed that pressure and gas concentration both had more impact on asphaltene instability during N₂ compared to methane, and the temperature had less effect. Also, they found that at high concentrations of N₂, the asphaltene redissolution rate decreased below the bubble point pressure. Alizadeh et al. (2014) investigated the effect of miscible CO₂ on asphaltene precipitation behaviors, and they found that for low CO₂ to mixture mole fractions, injecting CO₂ gas into live crude oil led to lower the asphaltene precipitation quantity. Before CO₂ approaches its critical concentration, this tendency will continue. Any further increases in the CO₂ percentage of the crude induce additional asphaltene to precipitate after this point. Tavakkoli et al. (2015) introduced a new technique called the "indirect method" that can be used for both systems of model oil and read crude oil systems. The absorbance of the supernatant fluid following centrifugation of oil and n-alkane mixtures is measured in this approach. The process can be utilized with low to high asphaltene concentration. Zanganeh et al. (2018) compared the effect of Ch₄, CO₂, and N₂ gases on asphaltene stability. They used different mole percentages to

highlight the impact of more fraction on asphaltene deposition at reservoir conditions. They found that when the pressure increased, the deposition of asphaltene increased. CO₂ and methane had higher impact on asphaltene deposition and precipitation compared to N₂. Golshahi et al. (2019) implemented several laboratory tests to investigate the effect of CO₂ on stability and structure of asphaltenes. They found that the structure of asphaltene had changed after the injection of CO₂ and a new functional amine group was created and reduced the asphaltene precipitation onset. Fakher and Imgam (2019) used nano-pore structures and a specially designed filtration apparatus to investigate the asphaltene instability under CO2 injection. Their findings showed that the asphaltene percentage was higher in the oil recovery at higher pressures. Also, less pore plugging due to asphaltene was observed when increasing the nano-pore size structure. Dashti et al. (2020) conducted a visual study of asphaltene deposition under different gas scenarios. They revealed that the rete of asphaltene deposition is 1.23 times faster during CO₂ injection compared to methane injection at 100 bar pressure. Less asphaltene rate deposition was observed during N₂ injection pressure. Elturki and Imqam (2021a; b; c) conducted a laboratory study to highlight the severity of asphaltene deposition under miscible and immiscible N₂ injection. They used filter paper membranes and various N₂ injection pressures to conduct the experiments using filtration technique. Their results showed that the asphaltene weight precent was higher at higher pressures, especially above the MMP, and on smaller pore structures. A few studies have investigated the CO2 and N2 MMP and its impact on asphaltene aggregations and oil recovery (Soroush et al., 2014; Adel et al., 2018). Others claimed that the asphaltene precipitation increased when the pressure increased before reaching the MMP and the severity of asphaltene precipitation was observed with high injection rate of CO₂ (Rong-tao et al., 2021).

Despite the fact that several investigations on the behavior of asphaltene deposition and precipitation under CO₂ injection have been conducted in conventional reservoirs, the impact of CO₂ on asphaltene stability in crude oils in shale unconventional resources is yet relatively limited. The goal of this study was to determine the degree of asphaltene fluctuation under CO₂ injection in ultra-small-permeability pore structures. This research will help minimize asphaltene deposition and precipitation and its related problems in future CO₂ injection applications by analyzing the influence of various parameters on asphaltene-related formation damage.

Asphaltene Thermodynamic Flocculation Mechanism

Multiple compounds can be found in crude oil based on its composition. These compounds can be found in three phases: gases, liquids, and solids phase (Mullins, 2008). However, the liquid phase consists of saturates, aromatics, and resins. The asphaltenes are the common solids that exist in crude oil composition (Elturki and Imqam, 2020b; 2021d). Asphaltenes are the main polar components (which contain heteroatoms such as N₂, sulfur, or oxygen) of crude oil which can be soluble in aromatic solvents, but insoluble in paraffinic liquids (Goual and Firoozabadi, 2002; Speight, 2014; Soleymanzadeh et al., 2019). However, saturates and aromatics are nonpolar compounds. Resins can be a good bridging agent to hold the polar and nonpolar components which resins have both polar and nonpolar sites (Alimohammadi et al., 2019). Asphaltene precipitation and deposition is a major problem during gas injection methods which causes serious issues during oil production and its processing. Plugging the pores of the reservoirs and changing the formation wettability are common issues (Shen and Sheng, 2017). Figure 1 presents formation wettability alteration due to asphaltene deposition and its impact on oil recovery.

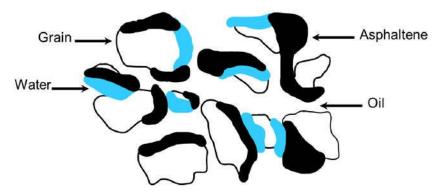


Figure 1—Formation wettability alteration due to asphaltene precipitation (Elturki and Imqam, 2021b)

Asphaltenes are high molecular weight, black to brown in color, organic fraction of crude oils which soluble in solvents of aromatic such as toluene but insoluble in n-alkenes such as n-heptane (Mohammed et al., 2021). Crude oil is classified as a colloidal system, whereas asphaltenes are classified as dispersed phases. Under reservoir conditions of temperature and pressure, asphaltene continues to stay in solution or in colloidal suspension stabilized by resins (Leontaritis and Mansoori., 1987; Jamaluddin et al., 2002), as shown in Figure 2. Asphaltenes carry an intrinsic charge of positive or negative depending on oil composition (Leontaritis, 1989). Resins have a strong tendency to associate with asphaltenes due to their opposite charge and act as protective bodies for asphaltene particles (Al-Kafeef et al., 2005). Colloidal asphaltene can be naturally or artificially precipitated if the resin protective shield is removed from asphaltene particle surfaces. According to Al-Kafeef et al. (2005), the flocculation of colloidal asphaltenes in oil-production flowing systems is essentially caused by the breakdown of attraction force balances between the absorbed resin molecules and asphaltene particles.

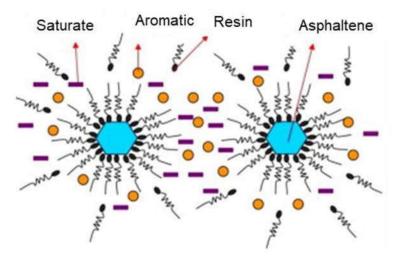


Figure 2—Resins attached with asphaltene (Ashoori et al., 2017).

As gas is injected into the reservoir, it contacts and interacts with the reservoir oil and thus changes the reservoir equilibrium conditions and fluid properties. Therefore, asphaltene may start to precipitate and flocculate because of colloidal suspension instability (**Rashid et al., 2019**). SARA (saturate, aromatic, resin, and asphaltene) analysis is widely used to identify the fractions of crude oil that affect asphaltene stability. Saturate includes the straight or branched saturated hydrocarbon chains and cyclic saturated hydrocarbon. Aromatic includes hydrocarbon with one or more unsaturated ring structures. Resin and asphaltene are large molecules with 3 to 10 or more ring structures per molecule (**Bahman et al., 2017**). Before further discussing the main factors that could affect the asphaltene flocculation, it is important to note that flocculation

and deposition are two separate phenomena. It is possible to have flocculation without substantial insitu deposition. Figure 3 shows a simple sketch showing the asphaltene precipitation, flocculation, and deposition in porous media. Flocculation is when the colloidal-fine particles in the formation comes out (precipitate) from a liquid phase, aggregation into larger particles (flocs). These flocs could break up into fines again totally or partially, which is called the process of flocs dissociation. While the deposition is described as flocs that accumulate onto the rock surface. The process of flocculation is primarily a function of pressure, temperature, and oil composition, while the deposition depending upon its attraction of large particles to adsorb onto the rock surface. The flocs could adsorb on the rock as static deposit, could block the pore throat (plugging) due to their bigger size compared to pore diameter or could be entrained and returned into fines (dissolved in oil phase) due to high shear rate (Leontaritis 1989).

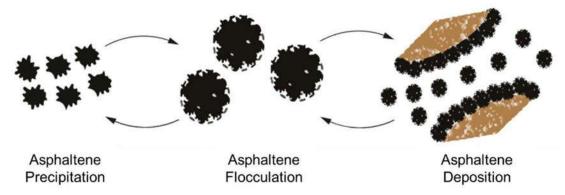


Figure 3—Asphaltene precipitation, flocculation, and deposition in porous media (Jiang et al., 2021)

Experimental Apparatus and Materials Description

MMP Experiment

At reservoir temperature, the MMP is the lowest pressure at which a gas will mix with the stored oil. When the interfacial tension between oil and gas vanishes after several contacts, MMP is the minimum pressure at which the miscibility between the injected gas and the reservoir oil is achieved (Ekundayo et al., 2013). The MMP experiments were carried out to verify that all following filtration tests would be below the MMP limit. Figure 4 shows the main components of the slim-tube test, which include a syringe pump, three accumulators, gas cylinders, a stainless-steel slim-tube full of sand, and a pack pressure controller. Several steps were implemented during the slim tube test, starting with the preparation for a pre-test where the slim tube was filled with sand then saturated with water at a rate of 1.5 PV. This phase is essential in calculating the weight of water-saturated slim-tube after filling with sand to determine the volume of pores. The second step was filling the slim tube with crude oil at a low rate of 0.5 PV to ensure a 100 % saturation of the pores inside the slim tube after pumping. The final phase was comprised of various experiments in controlling the temperature to the predetermined level, filling the gas into the gas cylinder, and pumping the gas at the rate of 1.2 PV during the injection. A regulator for backpressure was installed at the outlet for the slimtube and used to control the pressure by using a different water pump as the back-up reservoir for pressure. The test was stopped when the CO₂ gas broke through or when 1.2 PV of gas injected into the slim tube. Plotting CO₂ injection pressures against cumulative oil recoveries yields the MMP. The pressures used in the test to determine the MMP of CO₂ were 400, 600, 800, 1000, 1200, 1500, 1750, 1850, and 2000 psi. A crude oil with viscosity of 19 cp was used in all slim tube experiments. Figure 5 shows the results of MMP experiments. It was found that the MMP of CO₂ at 32°C was 1450 psi when using 19 cp viscous oil.

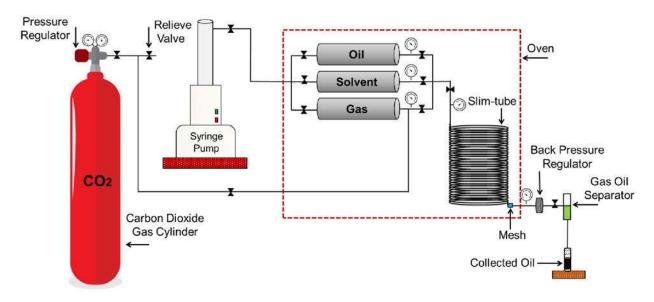


Figure 4—Schematic diagram of the slim-tube apparatus.

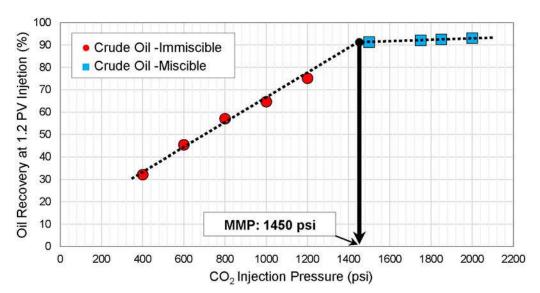


Figure. 5—CO₂ MMP determination using an oil viscosity of 19 cp at 32°C.

Filtration Experiments Apparatus:

The filtration experiments were conducted in order to highlight the severity of pore plugging on nano filter membranes due to asphaltene clusters under CO₂ gas injection; the main components of these tests, as shown in Figure 6, include the following:

- **Filtration Vessel:** A specially designed filtration vessel which can accommodate three filter membranes, as shown in Figure 7.
- **Filter Membranes:** Filter paper membranes of 450, 100, and 50 nanometers.
- **Mech Screens:** Three mesh screens to prevent the filter membranes from folding under high pressure.
- Carbon Dioxide Cylinder: A high purity CO₂ cylinder with a pressure regulator to control the cylinder pressure.
- **Spacers:** As a support, spacers were inserted between each mesh screen to keep them in place.
- **O-Rings:** Rubber O-rings were placed above and below each spacer to prevent leaking and to ensure oil flow through the filter paper membranes.

- **Backpressure Regulator**: A regulator for backpressure was installed at the filtration vessel outlet and used to control the pressure using a syringe pump.

- Oven: An oven was utilized to control the temperature of the filtration vessel during the experiments.
- **Transducers:** To monitor the pressure changes, two transducers were positioned at the inlet and outlet of the filter vessel and connected to a computer.
- **Crude Oil:** A crude oil with viscosity of 19 cp was used in all the experiments and Gas Chromatography-Mass Spectrometry was used to determine the composition of the crude oil, as shown in Figure 8.
- **Accumulators:** One oil accumulator is used to inject oil into the vessel, while the other is used to pressurize gas to achieve higher pressures.

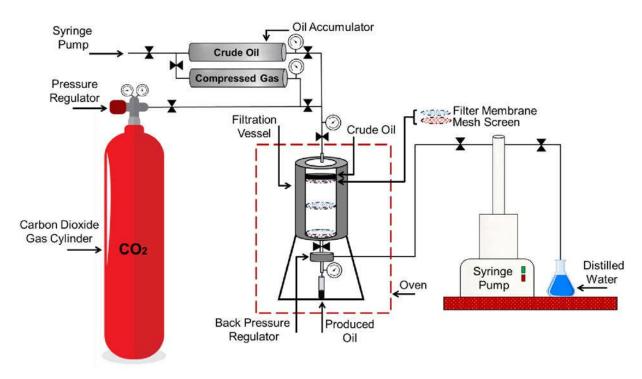


Figure 6—Setup of filtration experiments.

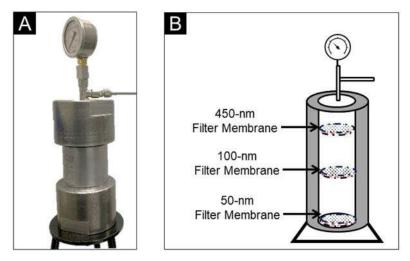


Figure 7—Filtration vessel (a) Real filtration vessel. (b) Sketch filtration vessel.

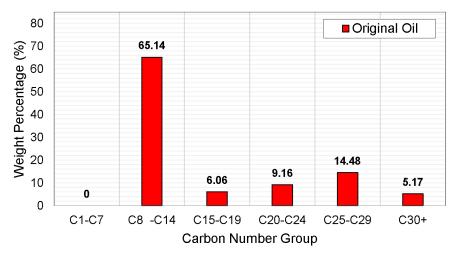


Figure 8—Crude oil composition

After preparing all the needed materials and the setup is ready, the filtration process began with placing the first set of mesh screen, filter paper membrane, rubber O-rings and spacers inside the vessel, respectively. This step was repeated twice for the next two sets. The filter paper membranes were put in the following order: 450-nm in the top, 100-nm in the middle, and 50-nm in the bottom of the filtration vessel. Then, the vessel was firmly sealed with a specially constructed top to keep all of the sets together and prevent leaking during the experiment. A crude oil of 30 ml was injected into the vessel using a syringe pump. CO_2 was injected into the vessel and the oil was then exposed to the gas for 2 hours soaking time to let the gas to interact with the asphaltene inside the oil. The syringe pump at the outlet was turned on to perform a predetermined constant pressure (equal to the injection pressure of the test) which presents the reservoir pressure and to let the oil to pass through the membranes. The oil was collected at the outlet and the experiment was stopped when no oil was observed. Finally, the oil was collected from all the filter membranes and the filter membranes were retrieved for further analysis. The asphaltene percentage weight was calculated using the following equation:

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

where Asphaltene wt%=asphaltene weight percentage, wt asphaltene=asphaltene weight on the filter paper, and wt oil=oil sample weight.

Results and Discussion

Effect of Porous Media Pore Size

To investigate the impact asphaltene precipitation and deposition under CO₂ injection on ultra-small pore size structures, three sets of filter paper membranes (450, 100, and 50 nanometers) were selected in this research. All the tests were conducted at a temperature of 32°C. The filter paper membranes were placed in a heterogenous order: The 450-nm filter was in the top of the vessel, followed by the 100-nm filter paper, with the 50-nm filter paper in the bottom. Asphaltene weight percent in each filter paper membrane was investigated using two different pressures (i.e., 750 and 1250 psi). Figure 9 reveals the findings for the asphaltene weight percentage in all the filter membranes for both pressures. The asphaltene weight percents under 500 psi of CO₂ pressure were ranged from 7.45% to 9.36% in 450-nm and 50-nm filter membranes, respectively. A higher percentage of asphaltene weight percent was observed on 50-nm due to small pore size structure. By comparing the results to asphaltene weight percent under 1250 psi of CO₂, higher asphaltene weight percent was determined to be a result of pressure increase. The asphaltene weight precent was 13.87% on 450-nm filter and increased up to 16.97% on 50-nm filter membrane. These observations showed

that higher pressure impacted negatively the bonds between the asphaltene particle and resins inside the crude oil and thus resulted in an increase in the asphaltene deposition and precipitation. Also, smaller pore size of the membrane's structure led to prevent more asphaltene particle to pass through the pores easily and more asphaltene clusters deposited. Asphaltene particles greater than 450-nm, for example, will not pass through a 450-nm filter membrane.

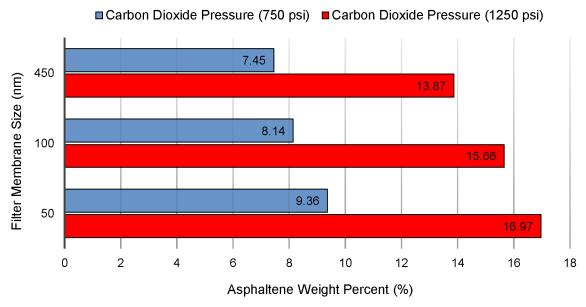


Figure 9—Asphaltene weight percent using a heterogenous filter paper membranes distribution under 500 and 2000 psi of CO₂ injection pressures.

Effect of Carbon Dioxide Injection Pressure

An oil sample was collected from the remining oil and produced oil after each experiment to highlight the impact of gas injection on the asphaltene instability. The oil that remained above the upper filter membrane after the experiment is referred to as the remaining oil. The produced oil is the oil that passed through all the filter membranes during the experiments and was collected at the outlet of the filtration vessel. As a consequence, the filter pore structure impacted the produced oil, and the findings of asphaltene weight percent will be discussed. The tests were carried out with a heterogeneous filter membrane distribution, a temperature of 32°C, and a soaking length of 2 hours. The asphaltene weight percent was higher in the remaining oil when using high pressure of 1250 psi of CO₂ compared to 750 psi injection pressure. Even though the remaining crude oil did not pass the filter membranes, the asphaltene weight precent was higher when using high pressure. On the other hand, the asphaltene weight precent was smaller in the produced oil because the filter membranes prevented most asphaltene weight precent was smaller in the produced oil because the filter membranes prevented most asphaltene particle from passing through and reaching the outlet. The same observation on the produced oil was noticed in which higher pressure led to more asphaltene weight percent. During the higher pressure, more asphaltenes passed through the filter paper membranes. These findings highlight the severity of high pressure of CO₂ on asphaltene deposition and precipitation in real reservoirs, especially in unconventional shale resources.

Visualization of Asphaltene Deposition Process

In this section, the process of asphaltene life cycle will be presented. After each experiment, the remaining oil was collected, and a sample of remaining crude oil (1 ml) was added to the solvent of n-heptane (40 ml) in a test tube and vigorously shook to make sure a complete dissolution of crude oil. Chemically, asphaltenes are insoluble in alkanes such as n-heptane. Thus, n-heptane was selected to show the asphaltene process over time until fully deposited. Figure 11 shows a simple sketch of test tube and the process of asphaltene precipitation and deposition after mixing 1 ml of crude oil with 40 ml of n-heptane (ratio of 1:40). Figure

12 presents the process of asphaltene deposition of a remaining oil sample after the filtration experiment during 750 psi injection pressure. At zero time, no asphaltene can be found in the test tube which was fully dissolved in the solvent of n-heptane. After 1 hour, the asphaltene particles started to precipitate and form small clusters in the mixture. After 4 hours, the fine clusters produced from the precipitation process started to combine and form bigger clusters (flocs), a process referred to as flocculation. These flocs started to gather and after 12 hours started to deposit in the bottom of the test tube. Deposition is the process of transferring flocs from the oil phase to the rock surface (Al-Ghanem, 2017). These findings provided additional understanding into how asphaltene developed in a real reservoir.

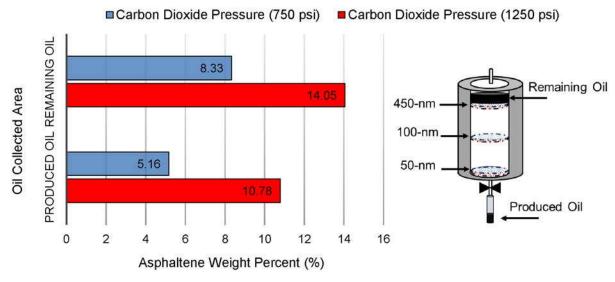


Figure 10—Asphaltene weight percent in remaining and produced oil under 750 and 1250 psi of CO2 injection pressures.

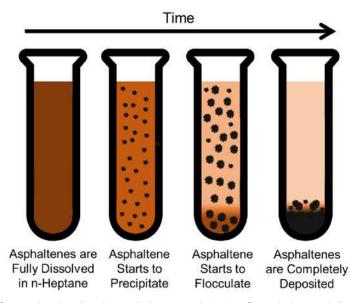


Figure 11—Simple sketch of test tube showing the asphaltene precipitation, flocculation, and deposition in n-heptane solvent.

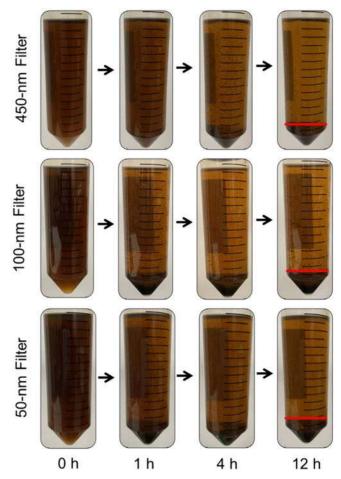


Figure 12—Asphaltene precipitation and deposition visualization process using 750 psi CO2 injection pressures.

SEM and Microscopy Imaging Analysis

Microscopy imaging and scanning electron microscopy (SEM) provides high-resolution images of the filter paper structures in order to highlight the asphaltene particles and pore plugging. Figure 13-A shows asphaltene particles and clusters flowing in the supernatant of the mixture (1 ml of oil and 40 ml of nheptane) after injecting 750 psi of CO₂ gas. This indicates that the bonds between asphaltene particles and resins in crude oil was weakened due to CO₂ gas pressure. These findings strongly suggest that CO₂ injection influenced the stability of the asphaltene in crude oil. The photo was processed to highlight the asphaltene particles in red color, as shown in Figure 13-B. The mixture was then filtered using a 0.45-µm filter paper to collect the asphaltene particles for asphaltene weight percent determinations. This filter paper was analyzed under the microscope to show how asphaltenes combined on the filter paper, as shown in Figure 14-A. The asphaltene particles was highlighted in red, as shown in Figure 14-B. For more clarity of how asphaltene plugged the filter paper membranes, the 50-nm filter paper membrane was selected for SEM analysis. Figure 15 shows the 50-nm filter paper membrane after filtration experiment using 750 psi of CO₂ gas injection, and the filer paper membranes was cleaned using n-heptane. The filter paper membrane was processed using scanning electron microscopy and the asphaltene particles clearly plugged the pores of the filter paper membrane. These results depict that asphaltene accumulated in the filter membrane's pores, obstructing the crude oil's flow. This phenomenon could be encountered in real reservoirs and lead to several issues, especially in unconventional reservoirs with ultra-small permeability and small porosity.

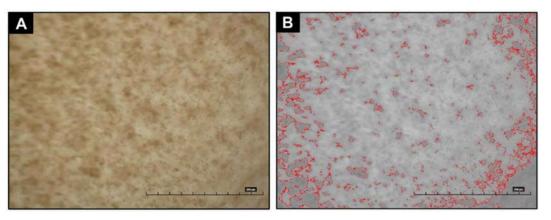


Figure 13—Mixture Supernatant of the crude oil and n-heptane when using 750 psi of CO₂ gas injection (A) under the microscope and (B) after detecting the asphaltene clusters shown in red using powerful photo-processing software

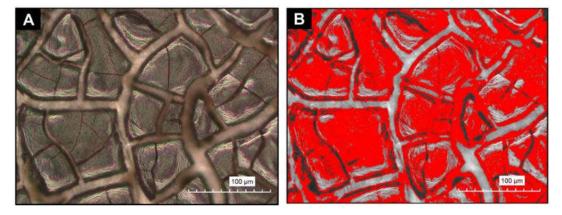


Figure 14—Filtration paper under a microscope characterizing residual deposition of a crude oil and n-heptane mixture when 750 psi of CO₂ gas injection was used (A) asphaltene particles in dark-brown color and (B) highlighted-asphaltene particles in red using powerful photo-processing software

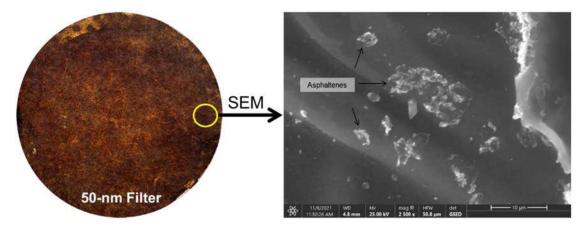


Figure 15—Filter membrane of 50-nm under light and after cleaning using n-heptane when 750 psi of CO₂ gas injection is used (left) and under scanning electron microscopy showing asphaltene particles (right)

Discussion

To provide a comprehensive picture of how gas injection might affect asphaltene instability in crude oil, the findings of this study will be discussed in association with earlier work on asphaltene deposition under N₂ injection (**Elturki and Imqam, 2021a**; **Elturki and Imqam, 2021b**). Figure 16 clearly shows the findings of asphaltene weight percent at immiscible injection pressures of 750 and 1250 psi for both CO₂ (present study) and N₂ (previous work). The asphaltene weight percent on 450-nm was found to be substantially

lower (i.e., 2.00 percent) when using N₂ compared to 7.45 percent when using CO₂ at 750 psi injection pressure. For N₂ 750 psi injection pressure, the asphaltene weight percent increased slightly from 2.00 percent to 3.13 percent on 450-nm and 50-nm filter paper membranes, respectively. This demonstrated that N₂ has slight effect on the stability of asphaltene in crude oil. The asphaltene weight percent, on the other hand, was significantly greater when CO₂ was used, ranging from 7.45% on 450-nm filter to 9.39% on 50-nm filter. When utilizing CO₂, a higher-pressure of 1250 psi contributed to a dramatic increase in asphaltene weight percentage in all filter membranes compared to a moderate increase with N₂ injection. The asphaltene weight % was higher on 50-nm in all filter paper membranes due to the small pore structure (asphaltene clusters cannot efficiently pass through in smaller pores). CO₂ dissolves more easily in crude oil than nitrogen and may rapidly reach supercritical levels. As a result, CO₂ has a very great mass transfer ability than N₂. Also, the ability of CO₂ to extract light hydrocarbon components from the crude oil led to higher asphaltene wight percent (Chung, 1992; Tovar et al., 2021). According to these results, implementing CO₂ gas injection could be challenging in terms of asphaltene deposition than N₂. These conclusions were achieved based on laboratory studies, and further research is needed to apply these findings in the field.

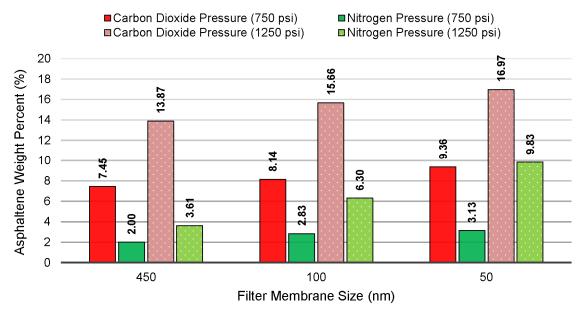


Figure 16—Comparison of asphaltene weight percentages under immiscible CO₂ and immiscible N₂ (Elturki and Imqam, 2021a; Elturki and Imqam, 2021b) gas injection pressures.

Conclusions

This research investigated experimentally the impact of CO₂ gas injection on the stability of asphaltenes in crude oil. Filtration experiments were carried out using specially designed vessel and filter paper membranes with ultra-small pore structures to filter the crude oil under gas pressure in order to highlight the severity of asphaltene particles on pore plugging of the filter papers. Based on our findings, we propose the following conclusions:

- Increasing the pressure of CO₂ during the filtration experiments resulted in an increase in asphaltene weight percent in all filter paper membranes.
- As the pore size decreased of the filter paper membranes, the asphaltene weight percent was increased especially in 50-nm filter membrane due to small pore size structure.
- The visualization experiment revealed the process of the asphaltene life cycle starting with precipitation, fluctuation, and then deposition process. At zero time, the asphaltenes were fully

dissolved in the solvent of n-heptane. After 12 hours, the asphaltene clusters and particles were fully deposited.

- SEM and microscopy imaging provided a clear picture on how asphaltene particles plugged the pore structure of the filter paper membranes.
- In the same injection pressure conditions, the ability of CO₂ to extract light hydrocarbon components from crude oil is substantially greater than in our previous study (which employed N₂ gas injection pressure) and hence resulted in a considerable asphaltene weight percent.

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