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Enhanced Oil Recovery in Unconventional Liquid Reservoir Using a Combination of CO₂ Huff-n-Puff and Surfactant-Assisted Spontaneous Imbibition

Fan Zhang, Imad A. Adel, Kang Han Park, I. W. R. Saputra, and David S. Schechter, Texas A&M University

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Abstract

Field observations, along with experimental laboratory, exhibit evidence that enhancing production by CO₂ huff-n-puff process is a potential EOR technique that improves the, commonly low, ultimate oil recovery in unconventional liquid reservoirs (ULR). As pressure goes beyond the MMP, intermediate components of oil vaporize into the CO₂ and consequently condense at room pressure and temperature. In addition, Surfactant-Assisted Spontaneous Imbibition (SASI) process has been widely believed to enhance oil recovery in ULR, which has been investigated by several laboratory and numerical studies. During the hydraulic fracturing with surface active additives, surfactant molecules interact with rock surfaces to enhance oil recovery through wettability alteration and interfacial tension reduction. The wettability alteration leads to the expulsion of oil from the pore space as well as water being imbibed into the matrix spontaneously. However, the understanding of hybrid EOR technologies, combining both gas injection and surfactant imbibition, to enhance recovery in ULR is not well studied.

In this manuscript, we assess the potential of combining both CO₂ huff-n-puff and surfactant imbibition techniques in optimizing oil recovery in ULR. Sidewall core samples retrieved from ULR were first cleaned utilizing the Dean-Stark methodology and then saturated by pressurizing them with their corresponding oil for three months. CO₂ huff-n-puff experiments were operated on shale core samples under different pressures in a set-up integrated into a CT-scanner. Those cores were then submerged in the surfactant solution, in a modified Amott cell, to observe whether any additional oil is produced through the process of SASI. Total production from these two different methods, which was done sequentially, will provide insight into the possibility of hybrid EOR technology. CO₂ huff-n-puff experiments were performed below and above the MMP which was previously determined by the slim-tube method. Contact angle (CA), interfacial tension (IFT) were also measured on the saturated shale core samples. CT-Scan technology was used to visualize the process of oil being expelled from the core plugs in both CO₂ huff-n-puff and spontaneous imbibition experiments.

Experimental results provide a promising outcome on the application of hybrid EOR technology, CO₂ huff-n-puff and SASI, improving oil recovery from ULR. Oil recovery was observed to reach around 50% of measured OOIP from CO₂ huff-n-puff alone with an addition of 10% recovery from SASI after the CO₂

treatment. A detailed description of the correlated experimental workflows is presented to investigate the hybrid EOR technology in enhancing oil recovery in ULR. In addition, a discussion on the difference in mechanism of oil production from the huff-n-puff and SASI method is also included alongside several additional novel findings regarding the color shift of the produced oil. MMP data of CO₂ and oil measured as well as a change of contact angle (CA) and interfacial tension (IFT) when the surfactant is introduced into the system are also provided to support insight on the mechanism of the production improvement. All measured and compiled data deliver the required information for this study to demonstrate the possibility of combining both CO₂ EOR and SASI EOR, a hybrid EOR, as a practical method to produce a significant amount of oil from unconventional shale oil reservoirs.

Introduction

Unconventional shale reservoirs have played an important role in oil production for a decade, leading the United States to become one of the world's top producers. Multistage hydraulic fracturing treatment triggered the success in developing ULR as well as becoming a necessity due to the characteristic of shale reservoirs possessing low porosity and ultra-low permeability (Zhang et al. 2018b). Hydraulic fractures interact with existing natural fractures to create fracture networks in ULR, which provides sufficient flow paths for oil to be extracted from the tight shale reservoirs. Two of the most prolific shale formations in the United States are the Wolfcamp in the Permian Basin and the Eagle Ford located in the Western Gulf Basin. Combined Wolfcamp and Eagle Ford basin contain nearly 22 billion barrels of unexplored resources, based on the US Geological Survey. The production from the two plays has been growing over a total of 3.7 million b/d (EIA, 2017). However, those astonishing numbers are negligible when it compared to the fact that for most cases the average recovery factors of wells in the area are less than 10%.

Multiple hydraulic fracturing treatments, along with the horizontal wells, significantly enhance communication in the reservoirs, resulting in crude oil production from the matrix. However, a rapid decline of the production rate happens in shale reservoirs due to the poor fluid transport through the shale rock. Typically, the oil production rate decreases to less than 20 % of the original production rate and half of the total oil produced within the first year. The shale reservoir still has 90 % of OOIP when the oil production falls below the economic line. Low efficiency in recovering the oil from these shale reservoirs are mainly caused by the sole reliance of primary depletion. EOR technologies should be applied as it could play an essential role in compensating for the decline and sustain production of unconventional shale reservoirs. Field experience along with laboratory experiments have demonstrated EOR methods can effectively produce additional oil beyond primary depletion in both conventional and unconventional reservoirs. EOR techniques, such as gas injection, chemical methods, and thermal methods, have been applied worldwide to improve the ultimate oil recovery. In the United States, CO₂ injection is the most applied EOR technique in ULR (Zou and Schechter 2017). Additionally, Surfactant-Assisted Spontaneous Imbibition is also the common EOR method used to improve recovery from ULR.

The selection of CO₂ for gas injection EOR in ULR, is due to the high effectivity of CO₂ in improving production in ULR. Compared to N₂ and CH₄, CO₂ possesses a lower MMP to oil. Lower MMP allows CO₂ to be miscible in the corresponding crude oil under lower working pressure. The miscibility of CO₂ into the oil is highly essential as it will cause two essential phenomena which directly relate to the incremental of oil production, the swelling of oil and the reduction of its viscosity. Another strong point for the usage of CO₂ is that it can be in the supercritical state under the typical pressures and temperatures of ULR reservoirs. The supercritical state of CO₂ is highly beneficial due to its liquid-like high-density behavior.

Organic shales rocks contain kerogen in addition to the common inorganic compound as the building blocks of its matrix. The inorganic matrix serves as the larger pore for storage with diameters of its pore above 50 nm, similar to the pore space found in the conventional reservoirs. As expected, it is the pore space from the organic matrix that gives out the typical ultra-low permeability feature due to the size of the pore

openings contained in it. Often classified as mesopores and micropores, these two classes of pore size exist in the organic matrix. Pores with an opening diameter of less than 2 nm are classified as micropores, while those that fall in between the classification of micropores and macropores are grouped as mesopores. Going down to the scale of micropores, Darcy's law becomes obsolete due to the ultra-low physical flow capacity of the pore. While on the other hand, diffusion, governed by Fick's law becomes more and more significant (Civan et al. 2011). The shift of flow mechanisms from pressure difference-driven to concentration gradient-driven could have a notable impact on how an EOR should be applied on these type of plays. To make things even more interesting, several studies have shown the effectiveness of the compartment to PVT properties of the hydrocarbon contained in the pore (Haider and Aziz 2017; Luo et al. 2018; Stimpson and Barrufet 2016). Small pore openings caused a shift in property of the fluids such as bubble point and most importantly, MMP of CO₂ and oil, which could directly affect the mechanism of which the application of gas EOR on ULR undergo.

Laboratory experimental set-up for the study of CO₂ EOR on shale rock sample requires some advance modification from the typical gas EOR set-up on the conventional reservoir system. Two main complications faced when running gas injection in organic-rich rocks are the inability to determine the initial oil saturation, and the injectivity problem as we can not flow gas through the core sample due to its ultra-low permeability. Determination of initial oil saturation can be performed by a cleaning and re-saturation process as proposed by (Tovar et al. 2018). The cores are cleaned using extended Dean-Stark extraction method and are re-saturated to known initial oil in place in the laboratory. The cores are aged in their corresponding reservoir oil 10,000 psig for a period of three months. This methodology enables accurate determination of the original-oil-in-place for each of the core plugs prepared for the further experiment. The inability of continuous injection through the core sample is avoided by treating the process as a huff-n-puff. Experimental set-up of CO₂ EOR in this scheme is achieved by surrounding the core sample with glass beads, which also acts as a representation of a high-permeability hydraulic fracture network (Tovar et al. 2014). On each huff-n-puff cycle, CO₂ is injected into the porous media generated by the glass beads instead of injecting directly into the core, allowing contact of the gas molecules with the surface of the oil-saturated core sample. It has been found that laboratory experimental of CO₂ EOR utilizing this method resulted in significant oil production (Tovar et al. 2018). In addition to the success of producing oil a highly unique phenomenon of pressure-dependent oil recovery on pressure above the MMP was also observed.

Surface activity, and its exploitation to improve oil recovery from the unconventional reservoirs, have been investigated in numerous published studies. Multiple methods are available for the purpose of analyzing surface properties alteration such as surfactants (Alvarez et al. 2017b), salt (Valluri et al. 2016), and the combination of the two (Park and Schechter 2018). Changes in the interfacial properties cause the alteration of both wettability and interfacial tension, altering the state of capillary force as a final product. The shift of capillary forces has been observed to improve oil recovery (Zhang et al. 2018a).

Wettability alteration coupled with IFT reduction play a significant role in improving oil recovery in ULR. The addition of surfactant into stimulation fluids have been applied to the oilfield as an EOR method to enhance oil production in unconventional shale reservoirs. Surfactant-assisted spontaneous imbibition can effectively increase the recovery factors, which has been demonstrated in several laboratory studies (Alvarez et al. 2014). Both the Permian Basin and Eagle Ford sidewall core plugs have been tested by the spontaneous imbibition EOR technique. The experimental results revealed that surfactant-assisted spontaneous imbibition could enhance oil recovery in shale cores by wettability alteration and moderate IFT reduction. Adding a proper surfactant to completion fluid can considerably improve the effectiveness of hydraulic fracturing treatments to enhance oil recovery in ULR.

Wettability is commonly defined as either water-wet or oil-wet in petroleum system, depending on the affinity of water or oil to spread onto a rock surface. The fluid having a higher affinity toward the rock surface is called the wetting phase, and the fluid that forms bubble shape droplets on the rock surface

is named as the non-wetting phase (Anderson 1986). Surfactant molecules have an amphiphilic nature with two parts, known as head and tail part, possess both affinities for the aqueous phase and oleic phase. Therefore, surfactant molecules perform as a bridge between water and oil phases in the pore system of reservoirs. Surfactant molecule comprises of a hydrophilic head and a hydrophobic tail, the head part has a higher affinity to the aqueous phase and the tail part towards the oil molecule. Therefore, the water-oil interfacial tension decreases due to an amphiphilic characteristic of surfactant molecules. Additionally, surfactant molecules interact with rock surfaces to alter the wettability of rock from oil-wet to water-wet, resulting in water being imbibed into the pore space spontaneously and oil being expelled out from the matrix.

Spontaneous imbibition is primarily driven by capillary forces which are defined as Young-Laplace equation in Eq. 1, where σ is IFT, θ is contact angle, and r is pore radius. IFT and contact angle are the two dominant parameters in capillary pressure based on Eq. 1. Surfactant molecule can alter the wettability of rock surface and reduce water-oil IFT simultaneously, resulting in complex capillary forces in shale nanopores. In addition, capillary forces in the nanopores are significant due to the very small diameter of those pores (Deng and King 2015, 2018). The extent and magnitude of wettability and IFT alteration are rigorously evaluated to understand the performance of surfactants in enhancing oil recovery in ULR.

$$P_c = \frac{2\sigma \cos\theta}{r} \quad (1)$$

Complete and systematic laboratory workflows were established to evaluate surfactant related properties on performance in spontaneous imbibition by using shale cores (Alvarez et al. 2017b). Wettability and IFT measurements and spontaneous imbibition experiments were performed to evaluate the performance of different surfactants in enhancing oil recovery in ULR cores. Wettability can be measured quantitatively by contact angle, Amott-Harvey index, and US Bureau of Mines (USBM) methods, etc. The saturation, re-saturation, and classic techniques such as the Amott or USBM techniques are not suitable for wettability measurements in ULR cores. Due to low porosity and ultra-low permeability of shale cores, it is challenging to inject liquid breakthrough core plugs. The contact angle method is the ideal method to measure the wettability of shale rock surface. Thus, we measure the static contact angle of crude on aged shale sample surface which has been extensively reported as the quantitative determination of wettability. Interfacial tension of oil and water is defined as the control variable of the magnitude of the capillary driving force. Measurement of IFT can be performed by different methods, such as spinning drop method, sessile drop method, and pendant drop method. For the laboratory workflow constructed by Alvarez, pendant drop method was selected as the method of measurement as it works accurately in determining IFT in the range of IFT found on all surfactant systems tested.

Gas injection and surfactant-assisted spontaneous imbibition techniques are the two effective EOR methods in ULR. Several laboratory and numerical studies investigate the potential of gas injection in improving recovery factors in ULR. In addition, there are a few studies evaluating the effectiveness of surfactant-assisted spontaneous imbibition to enhance oil recovery in ULR. However, there is an insufficient understanding of combining both CO₂ huff-n-puff and surfactant imbibition techniques in optimizing the oil recovery in ULR. To fill the gap about hybrid EOR techniques to achieve the optimum oil recovery in ULR, we investigate the performance of gas injection coupled with surfactant imbibition method in ULR.

In this study, the CO₂ huff-n-puff experiments were performed on the resaturated sidewall cores retrieved from ULR under different pressures in the CT scanner. Then we conducted surfactant-assisted spontaneous imbibition on the core plugs that finished CO₂ huff-n-puff experiments. Next, contact angle, IFT, and CO₂ - oil MMP measurements were performed. Finally, we present all of the experimental results to provide the evidence that combined EOR techniques can achieve optimum oil recovery in ULR.

Methodology

Production from unconventional shale from primary depletion is still a relatively new realm, let alone the study of EOR on this ultra-tight rock. However, as expressed before, the low recovery from primary depletion just emphasizes the need for a new technique to improve the efficiency of recovering oil from shale reservoirs. This study is aimed to provide more insight into new methods of oil recovery from ULR. Studies covering the use of surface active chemicals and CO₂ to improve production have been published and described in the previous section with the implementation of the two methods separately. In this work, an experimental study combining both CO₂ and surfactant is presented. The incorporated procedure, including the measurement of MMP, huff-n-puff experiments under the CT-Scan machine, followed by contact angle (CA) and interfacial tension (IFT) measurements as well as spontaneous imbibition experiments will be briefly detailed in this section.

MMP Measurements

Minimum miscibility pressure (MMP) is one of the most crucial parameters in any gas injection project. The MMP is the lowest pressure at which the injected gas and the reservoir oil become miscible through a multi-contact process at the reservoir temperature (Elsharkawy *et al.* 1992).

The slim tube method is considered the most accurate way to determine the MMP. This method, being one dimensional, avoids the unfavorable conditions such as gravity override, unfavorable viscosity ratios and fingering, making this method the most accurate way to determine the MMP (Adel *et al.* 2018; Adel *et al.* 2016).

All recovery factors are measured at 1.2 PV of injected gas. To standardize the slim tube experiments, long coil lengths and low injection rates have been recommended in literature to avoid composition variations, effects of fingering and to ensure a stable thermodynamic front (Elsharkawy *et al.*, 1992).

The effluents are measured along the duration of the experiment to calculate the recovery factor at each pressure. The resulting recovery factors are plotted as a function of the pressure, and two regions are identified. The immiscible region, where the recovery factor is a strong function of pressure, and the miscible region, where the recovery factor is a weak function of pressure. The MMP of each sample was determined by fitting a straight line through each group of data points representing either the miscible or the immiscible region and solving for the interception point of the two trend lines.

CO₂ Huff-n-Puff experiments

We developed a physical representation of a hydraulic fracture in the laboratory by surrounding an organic-rich shale core plug with 1 mm glass beads inside an aluminum core-holder, as shown in Fig. 1. This set up is mounted into an aluminum core-holder that enables the penetration of X-rays. With this configuration, we closely reproduce field conditions by having a high permeability fracture in direct connection a matrix of permeability in the nano-darcy range. All of the experiments were performed in a core- flooding equipment coupled to a CT-scanner which enabled time lapse, imaging capabilities, and gas injection under reservoir conditions of pressure and temperature, and confining pressure. Once the rock matrix-hydraulic fracture system is packed into the core-holder and connected to the core-flooding equipment, a vacuum is applied to remove the air. Then we inject CO₂ from a floating piston accumulator until we reach the desired experimental pressure. At this point, we start the soak time at constant pressure for 10 hours. During soaking, CT-scans are performed regularly to track the changes in density as a function of time and space.

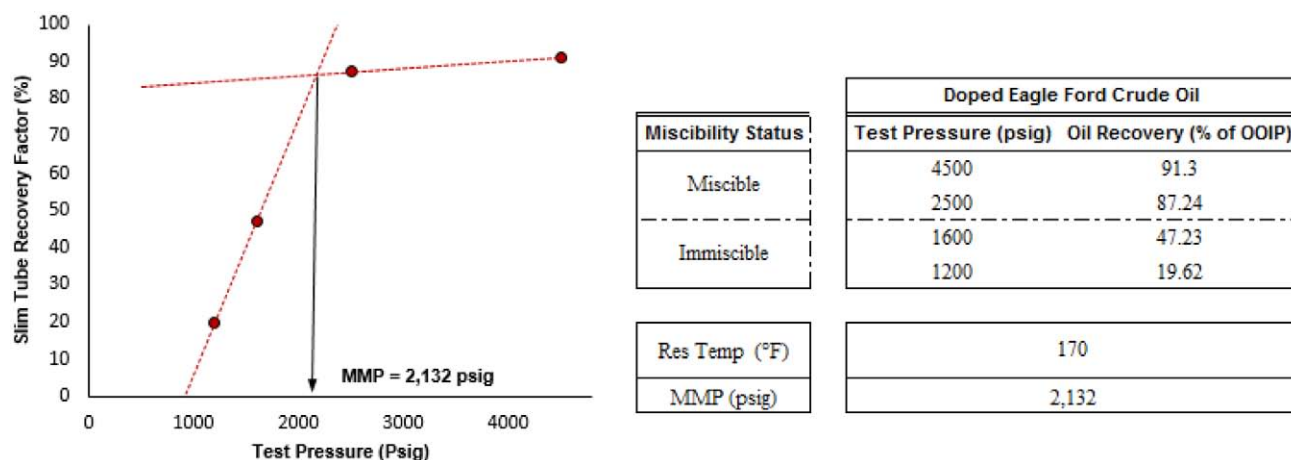


Figure 1—(a - left) MMP plot showing the recovery as a function of pressure for the doped Eagle Ford oil. (b - right) Slim tube recovery factors as a function of pressure for the MMP determination

When soak time is completed, the CO₂ is displaced by fresh CO₂ at a low flow rate, and the effluents are collected. The described procedure constitutes one huff-and-puff injection cycle; the process is repeated for the subsequent huff-and-puff cycles until no more oil is produced and ultimate recovery is achieved.

Contact Angle and IFT Measurements

Surfactant addition to the system of oil, water, and rock affects two critical parameters that are directly correlated to the production of oil from shale reservoir, wettability, and interfacial tension. Therefore it is essential to provide the measurement of the two parameters on both the initial and surfactant-affected state to present a comprehensive result on the application of surfactant to improve oil recovery from shale.

In this study, wettability was analyzed by measuring contact angle under three-phase conditions. Captive bubble method was utilized to determine the contact angle which then would determine wettability. Aged rock chip gathered from sidewall core plugs were used as the rock medium of which the contact angle is measured. The chip was submerged in the aqueous-phase and was brought up to the reservoir temperature. Oil bubble was then placed on the bottom-facing surface of the rock by dispensing 0.15-0.20 μ L of oil through a J-shaped needle. The picture of oil bubble placed on the rock surface then was captured using a high-speed camera where the contact angle data was gathered from. Reed and Healy's convention is often used as the classification of contact angle to different wettability. Due to the set-up of the captive bubble method, contact angle values measured from this experiment must be first converted to be able to use the Reed and Healy convention by deducting the measured contact angle from 180°.

On the interfacial tension side, inversed pendant drop method was chosen to measure the oil-water IFT. Oil bubble was slowly dispensed upward into a cuvette filled with the aqueous-phase which previously heated up to the reservoir temperature. An identical hi-speed camera from the contact angle measurement was utilized to capture the image of the oil bubble as it was precisely about to detach from the needle and by also feeding in the density data of both fluids on the measurement temperature, the oleic- and aqueous-phase IFT is calculated. On this measurement, the density data were obtained by direct measurement using Anton Paar DMA-4100. This machine possesses the capability of density measurement on high temperature, suitable for the data required on the IFT measurement.

Further details about the procedure of contact angle and IFT measurement performed in this study are available in (Alvarez et al. 2014; Alvarez and Schechter 2017; Alvarez et al. 2017c).

Surfactant-assisted Spontaneous Imbibition Experiment

Modified Amott cells were used for carrying out the surfactant-assisted spontaneous imbibition experiments. Core plugs were submerged into the aqueous phase solution for ten days, or until the oil production

stabilizes; produced oil is recorded using graduated tube located at the top of the modified Amott cells with time. Shale cores were placed horizontally on the bottom of Amott cell to minimize the effect of gravity on the experiment. The entire spontaneous imbibition experiments were performed at reservoir temperature within an oven. The experimental setup was periodically scanned in a CT-scanner during the whole test. More details of surfactant-assisted spontaneous imbibition experiment are in the paper (Alvarez and Schechter 2015; Saputra and Schechter 2018).

To observe the effectivity of combining gas and surfactant to recover oil, core plugs used on the SASI experiments went through the CO₂ huff-n-puff tests. After the last cycle on the CO₂ experiment, the plug was retrieved and directly placed in the modified Amott cell set-up. For simplification reason, on all core plugs from CO₂ experiments under different operation pressure, one surfactant was used for the imbibition experiment. Further study will be done to investigate the effect of different type of surfactants on the recovery from this combined EOR technique.

Results and Discussion

The results and observations from the correlated set of experiments are presented in this section. The potential of CO₂ EOR technique in ULR is validated by performing CO₂ huff-n-puff experiments. Also, the addition of surfactants into completion fluid is validated as an effective EOR method by results of surfactant-assisted spontaneous imbibition experiments. Measurement results including MMP, contact angle, and IFT are presented to evaluate the efficiency of surfactant in altering wettability and IFT as well as determine the pressure range for CO₂ huff-n-puff experiments. The laboratory results reveal that this hybrid technique of combining gas injection and surfactant imbibition EOR methods possess the capability of improving oil recovery in ULR.

Minimum Miscibility Pressure Determination

We examined the relationship between the operating pressure and the slim tube MMP. The purpose is to investigate how externally controlled parameters such as the operating pressure can be optimized to maximize recovery from unconventional liquid reservoirs.

The slim tube method is considered the most accurate way to determine the MMP. It simulates the 1D displacement of the reservoir crude oil by the injected CO₂ through a porous medium and fully accounting for the thermodynamic phenomenon, the effect of displacement efficiency and phase behavior taking place in the CO₂ - oil system inside a sand packed stainless steel coil. This method, being one dimensional, avoids the unfavorable conditions such as gravity override, unfavorable viscosity ratios and fingering, making this method the most accurate way to determine the MMP.

The recovery factor as a function of pressure for the doped Eagle Ford crude oil that is used to re-saturate the core plugs is presented in Fig. 1 The MMP was determined to be 2,132 psig.

Results of Gas Injection Related Experiments

Four experiments using re-saturated Eagle Ford core plugs were conducted (Table 1). The high permeability media was then saturated with CO₂ at constant pressure and reservoir temperature. The production was monitored using a CT scanning technology throughout the length of the experiments to track changes in composition and saturation as a function of time and space. Soak time was maintained constant and the experimental pressures were selected above and below the slim-tube MMP to show the effect of MMP on recovery.

TABLE 1—Core plugs injected volume and experimental pressure

Core #	Core length (in)	Injected Oil Volume (ml)	Experimental Pressure (psig)
Core 1	2.3	2.900	2,500
Core 2	2.0	1.785	3,000
Core 3	2.0	1.744	3,500
Core 4	1.5	1.129	1,400

Effect of pressure on recovery factor. The oil recovery (% OOIP) with time for all four CO₂ huff-n-puff experiments are plotted in Fig. 2 and Fig. 3. In each figure, we marked stages for each of the huff-n-puff cycle. On the first experiment, Core 1, experimental pressure was set above the MMP to be 2,500 psi. Fig. 2 (a) shows that the ultimate oil recovery for this set-up was observed to reach 31% of OOIP. As can be seen, five huff-n-puff cycles were executed on Core 1. Significantly higher recovery was observed in the first cycle compared to the preceding cycles. It is important to note that the reduction of recovery from each cycle did not come from the decrement of CO₂ quality on each cycle as fresh supercritical CO₂ was injected before each cycle started. On the next experimental pressure of 3,000 psi, core 2 was set inside the core-holder, and three huff-n-puff cycles were executed. The result from this experiment can be seen in Fig. 2 (b). An ultimate recovery factor of 41% OOIP was observed which is higher compared to the previous set-up. On the last working pressure tested, which was done on Core 3, six cycles were executed resulted in an even higher final recovery factor of 49% OOIP. A similar trend was observed in the last two experiments, core 2 and core 3, of where the recovery dropped from earlier cycle to the later. It is also important to note how the recovery factor from the three experiments kept increasing even though the working pressure was already well above the MMP.

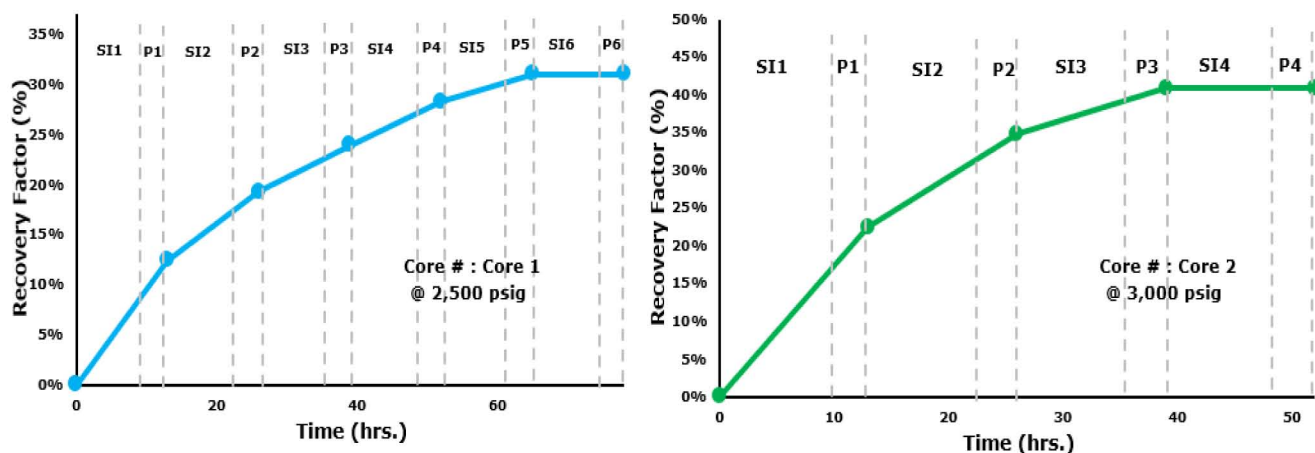


Figure 2—Recovery factors as a function of time for Core 1 (a - left) and Core 2 (b - right) at pressures of 2,500 and 3,000 psig.

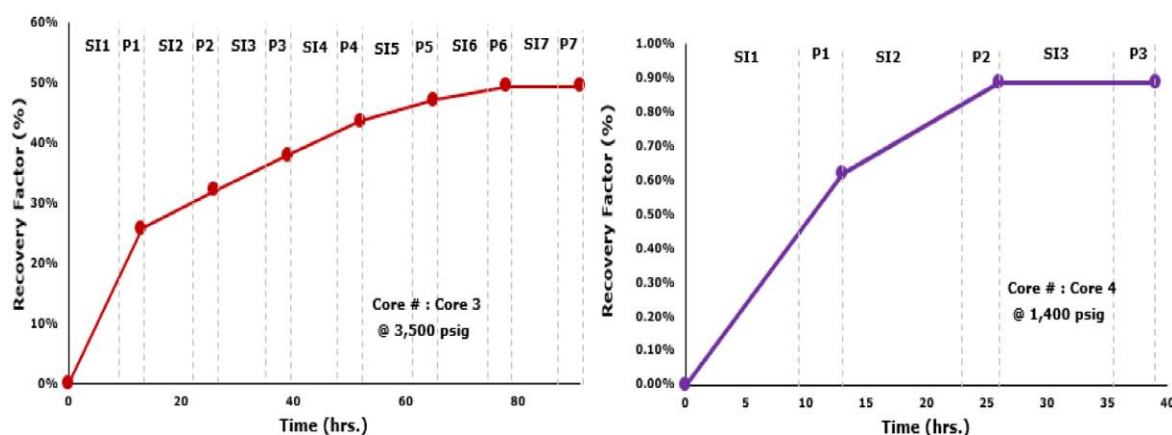


Figure 3—Recovery factors as a function of time for Core 3 (a - left) and Core 4 (b - right) at pressures of 3,500 and 1,400 psi

Recovery factor was 49.31% at the highest pressure of 3,500 psig. This is higher than the maximum of 40% we previously observed in the Wolfcamp (Tovar et al. 2018), and the reason is the higher concentration of intermediate hydrocarbon components in the Eagle Ford, and the higher experimental pressure. Higher pressures could not be achieved due to the corrosive nature of CO₂, which destroys Viton sleeve used in the core-holder set-up. However, higher recovery factors are expected at higher pressures.

Time-frame for recovery. The time frame in which the high recovery factors are achieved during huff and puff is critical. The feasibility of gas EOR process is questioned due to the slow transport in organic shales. The experimental results presented in this section illustrate that recovering oil from organic-rich shales during CO₂ huff-n-puff is not a lengthy process. Recovery from core plugs that takes several months to saturate is extremely rapid compared to the expectations from nano-Darcy rock samples.

The first CO₂ huff-n-puff cycles, composed of a soaking interval (SI1) and a production interval (P1), deplete the cores the fastest and produced about 50% of the ultimate recovery. The oil recovery rate starts decreasing after the first cycle until it levels off by reaching the maximum recovery.

The oil recovery curves of the CO₂ huff-n-puff experiments conducted above MMP pressure have a similar trend, comprised of three parts. Almost half of the total oil production was achieved from the first huff-n-puff cycle. As experiments develop, the volume of recovered oil decreases and maintain at a stable value during the middle of experiments, then they decrease to zero. During the first part, at the beginning of the experiment, a significant amount of oil is recovered as CO₂ soaks the fully saturated core plug, the lighter components of oil vaporize into CO₂ phase. In the second part, the volume of produced oil maintains at a constant value. As the 'easy' produced oil recovered from the first part, oil recovery rate is limited by the rate of CO₂ penetration into the core plug. By the time of the last part, CO₂ had already penetrated to the center of the core plug causing the oil recovery rate to decrease to zero.

Figure 3 (b) depicts the oil recovery with time for the CO₂ huff-n-puff pressure below MMP. In this experiment, oil was produced only in the first two huff-n-puff cycles. The oil recovery factor for Core 4 is less than 1%. The performance of CO₂ with experimental pressure below and beyond MMP was found to be entirely different. We can safely conclude that only insignificant amounts of oil can be extracted through CO₂ injection in ULR cores when the pressure of the system is less than MMP. For a successful CO₂ Huff-n-puff process, the operating pressure should be above MMP,

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This work confirms our previous findings, which challenge the paradigm that establishing miscibility is enough to achieve the highest recovery factors during CO₂ flooding, as is the case in conventional reservoirs. The best way to operate gas injection processes in organic rich shales is by designing a huff-and-puff project at the highest pressure possible, regardless of the crude oil - gas MMP.

Results of Surfactant-Assisted Spontaneous Imbibition Related Experiments

The surfactant-assisted spontaneous imbibition experiments were performed on the core plugs that finished the CO₂ huff-n-puff experiments. In this study, we used one type of surfactant on all spontaneous imbibition to reduce the variability of surfactants in recovery factors in ULR cores. The results of contact angle and IFT measurement are used to demonstrate the efficiency of surfactants in altering wettability and reducing interfacial tension between oil and aqueous phase. The spontaneous imbibition experimental results can verify the feasibility of combining gas injection and surfactant imbibition EOR techniques to achieve optimum oil recovery from ULR.

Contact angle measurements were conducted on the aged shale chips which were retrieved from the same reservoir as broken sidewall core plugs. IFT measurements were performed using corresponding oil. The contact angle and IFT measurements results are presented in Fig. 4. As shown in Fig. 4, the initial wettability of the samples was found to fall in the oil-wet region which is commonly found in ULR (Alvarez and Schechter 2016).

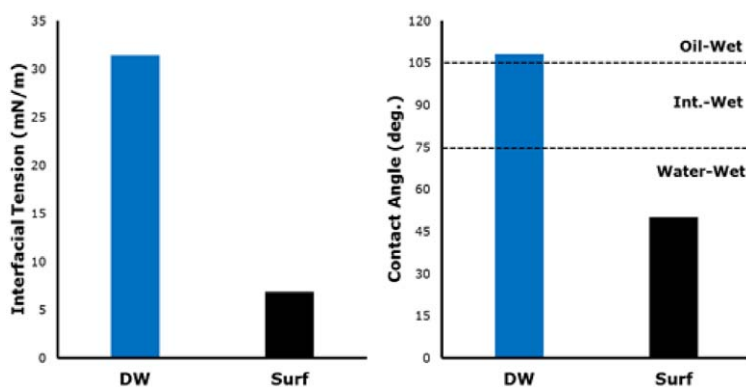


Figure 4—Results of contact angle and IFT measurements.

The wettability of shale samples was altered from oil-wet to water-wet as shale trims submerged into surfactant solution. The IFT measurement results indicated that the surfactant decreases the IFT from 31 mN/m to 6 mN/m. The surfactant showed an ability to reduce contact angle to a small value as well as IFT to a moderate range. Based on our previous work, surfactants with the ability to effectively alter the wettability to a low value and maintain the capillary pressure relatively high most likely recover more oil from ULR cores. Therefore, this surfactant type should have a good performance in improving oil recovery in ULR cores.

Surfactant-assisted spontaneous imbibition experiments were run ten days, and the ultimate oil recovery factors of all the tests were recorded as shown in Fig. 5. Consistent with the previous studies of surfactant-assisted spontaneous imbibition, the addition of surfactant into aqueous phase increases the oil recovery from spontaneous imbibition in ULR. Table 2 presents that similar volume of oil produced from spontaneous imbibition experiments. The maximum oil recovery obtained from spontaneous imbibition is 11.5%, which was performed on Core 4. Although up to 49% oil has been recovered from CO₂ huff-n-puff experiments, around additional 10% oil still can be extracted from surfactant-assisted spontaneous imbibition experiment.

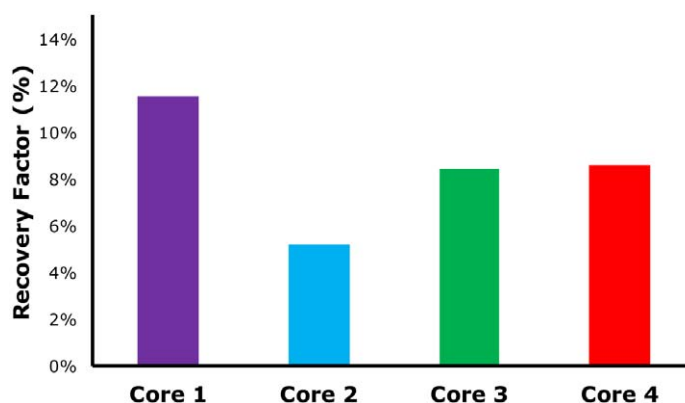


Figure 5—Oil recovery after CO₂ injection through Surfactant-Assisted Spontaneous Imbibition

Another fascinating observation was found from spontaneous imbibition experiments. During surfactant-assist spontaneous imbibition experiments, we took the pictures to record the production of a graduated cylinder of modified Amott cell. Fig. 6 presents the production of SASI at 12 hours and 240 hours. At the beginning of SASI, we observed many foams were gathered in the graduated cylinder before oil extracted from the core plugs. Typically, we cannot observe foam during spontaneous imbibition experiments. This observation reveals CO₂ was penetrated and adsorbed into the shale core plugs during CO₂ huff-n-puff experiments. In addition, it also evidenced water imbibed into the shale core and replaced air phase fluid out.

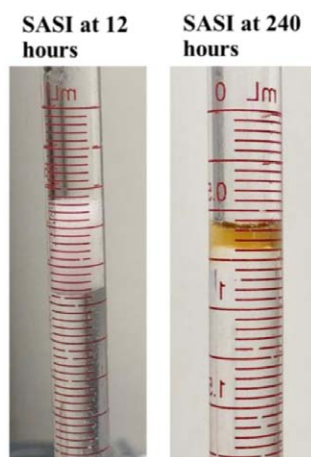


Figure 6—Observations during Surfactant-Assisted Spontaneous Imbibition

Combined CO₂ and Surfactant Results

The results of CO₂ huff-n-puff experiments and surfactant-assisted spontaneous imbibition experiments are gathered and plotted in Fig. 7–10. We present the ultimate oil recovery of combining these two EOR techniques in ULR cores. The maximum oil recovery is reached by the highest pressure performed using Core 3. Fig. 9 shows that 49% of OOIP was recovered from CO₂ huff-n-puff experiment and spontaneous imbibition contributed to additional 8.6% oil recovery, totaling to a 57.6% OOIP recovery. Core 2, Core 1, and Core 4 are sitting in the order of ultimate recovery from largest to smallest after Core 3. Recovery factors from each production mechanism of all the four cores are presented in Fig. 11 with the production from CO₂ presented as the horizontal pattern and the production from surfactant presented as the diagonal pattern. Comparing the case of Core 4 to the other three cases, it can be seen that the contribution of production from SASI to the total production was significantly more dominant compared to production from CO₂ on Core 4 compared to Core 1, 2, and 3. Core 4 was the only experiment in which the working pressure for the CO₂ was below the MMP.

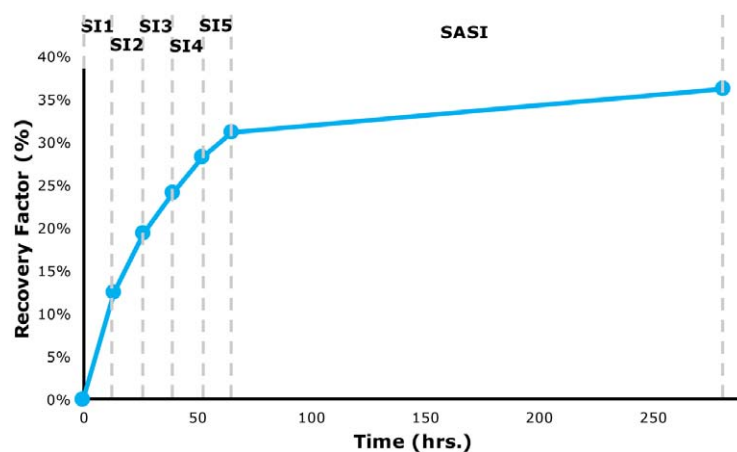


Figure 7—Oil recovery combining gas injection and spontaneous imbibition experiments for Core 1.

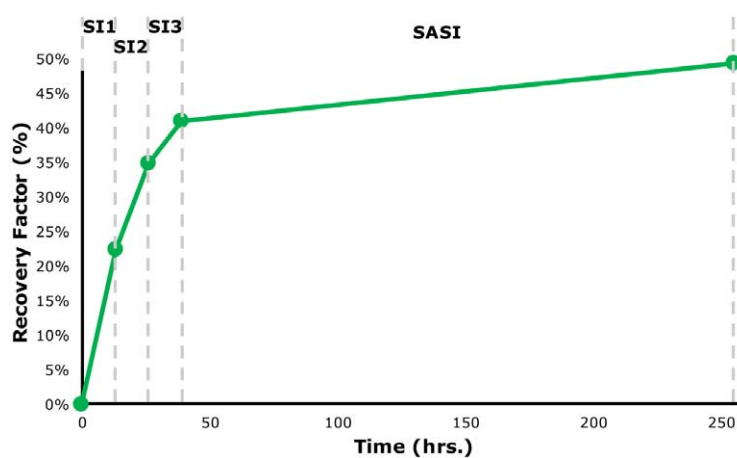


Figure 8—Oil recovery combining gas injection and spontaneous imbibition experiments for Core 2.

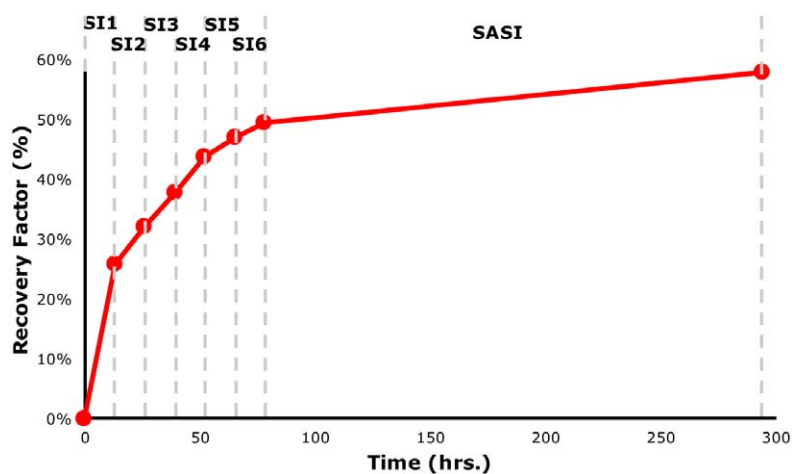


Figure 9—Oil recovery combining gas injection and spontaneous imbibition experiments for Core 3.

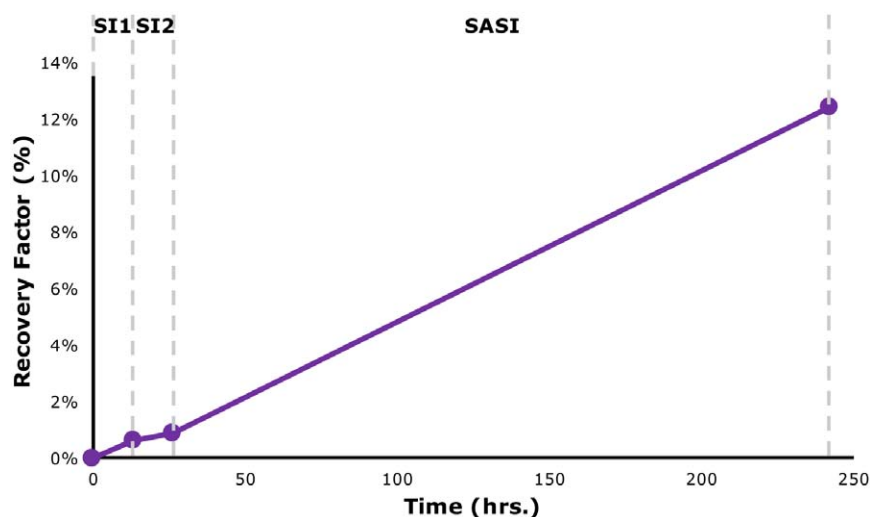


Figure 10—Oil recovery combining gas injection and spontaneous imbibition experiments for Core 4.

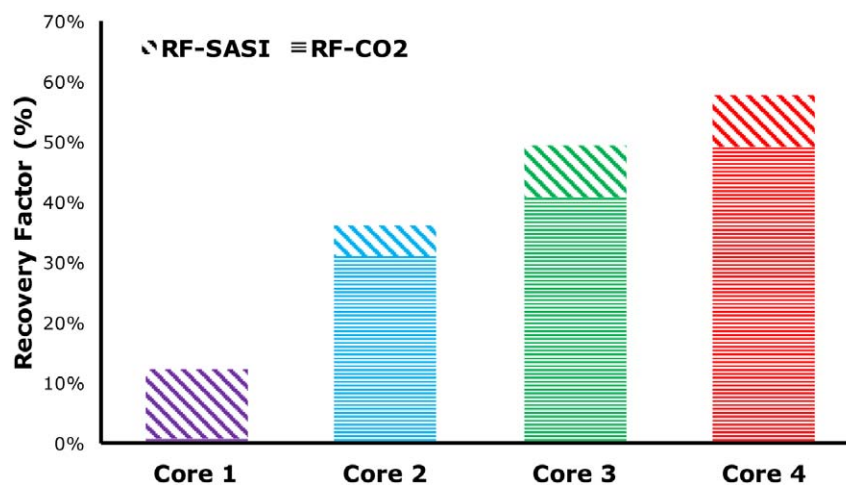


Figure 11—Summary of oil recovery of combining EOR techniques.

Figure 7–10 indicates that extra oil can be extracted from ULR cores that finished CO₂ huff-n-puff experiments, even though 50% of OOIP produced from gas injection experiment. It means that different components of oil were produced by gas injection EOR technique and surfactant imbibition EOR technique. The details of components produced by two methods are presented in the following part that discusses the color difference of the produced oil from the two experiments. These two EOR methods can work perfectly together; higher oil recovery from the gas injection technique does not mean less oil produced by spontaneous imbibition. In addition, for CO₂ huff-n-puff experiments, experiments should be performed under pressure higher than MMP to achieve economic oil production. The recovery factors increase as the pressure increases. Overall this combine EOR technique recovered a significant volume of oil from ULR, which is much more than the depletion case in ULR.

To evaluate the efficiency of this hybrid EOR technique in ULR, we investigate the oil saturation with time through the aging process, CO₂ huff-n-puff experiments, and surfactant-assisted spontaneous imbibition experiments. The aging process costs typically three months to restore the core plug to its original reservoir state. A significant volume of oil is produced from CO₂ huff-n-puff experiments within six huff-n-puff cycles. Spontaneous imbibition experiments recover extra oil from ULR cores by using less than ten days; commonly no more oil is retrieved from spontaneous imbibition experiments after three days. We present the oil saturation of all the shale cores for this whole procedure in Fig. 12.

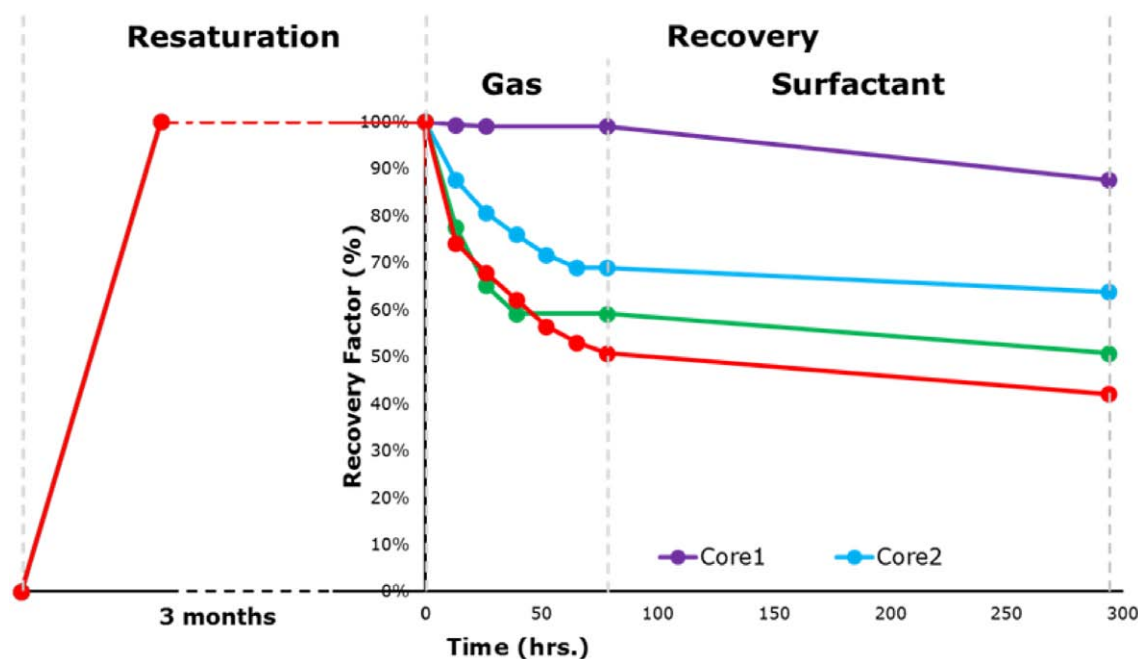


Figure 12—Oil saturation of the core through the aging process, gas injection experiments, and spontaneous imbibition experiments.

Figure 12 presents the evolution of oil saturation throughout the whole experimental steps. Three months are required to re-saturate the core plugs to reservoir conditions, but more than 50% of OOIP were recovered within ten days, which is less than 10% of saturation time. This observation shows that aside from the tremendous effect on quantity as demonstrated by the amount of oil recovered, implementation of this hybrid EOR also provides the ability to recover oil in a significantly shorter time when compared to solely relying on the pressure difference. The experimental results open the possibility of utilizing combined EOR techniques when chasing maximum oil recovery in unconventional shale reservoirs.

CT-Scan Images

All of the experiments were performed in core-flooding equipment coupled to a CT-scanner which enabled time lapse, imaging capabilities, and gas injection under reservoir conditions of pressure and temperature. The CT-scanning technology enables the monitoring of production to track changes in composition and saturation as a function of time and space.

CT-scan technology is used to study the fluid movement inside shale core plugs. The CT-scan machine generates a 3D image of an object by using the attenuation of X-rays. We scanned the core plug periodically during both CO₂ huff-n-puff experiments and surfactant-assisted spontaneous imbibition experiments as shown in Fig. 13. Images produced by the CT machine are in the form of 3D distribution of a variable named CT-number which could be used to identify the shift of oil, water, or supercritical CO₂ saturation in each corresponding area. Typically, water has the highest density followed by supercritical CO₂, and oil, meaning that the CT-number for each phase decreases from water, supercritical CO₂, then oil. On this experiment, inert doping agent was added to the oil-phase which resulted in the amplified CT-number for the oil phases to be the highest compared to the other two phases. On each CT files generated from each scan, one slice on fixed location was selected instead of showing the entire 3D image for the purpose of simplification. Consistent color coding was also applied to all the images to enhance the visibility of the scan results. For CT-scan images, color corresponds to specific CT number range, such as brighter color correlates to higher CT-number. The legend for the color code is shown on the right-side of Fig. 13.

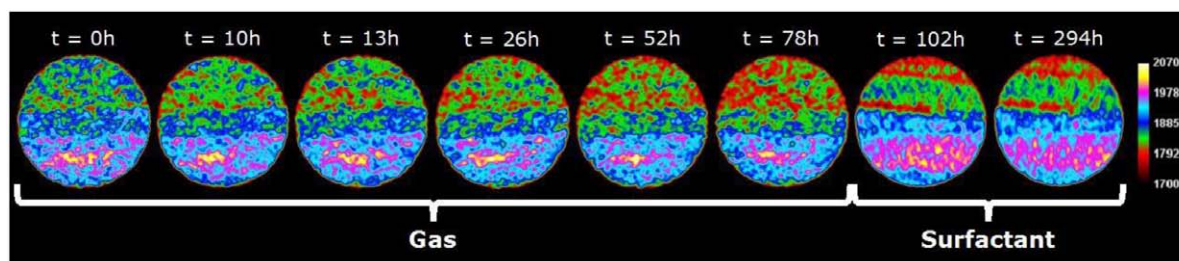


Figure 13—CT scan images from the gas injection experiment and spontaneous imbibition experiment on Core 3.

Looking at the images generated from the CT, it can be seen that the core plug used in the experiment has three different bedding layers as three different color themes are observed in the image. However, a similar trend was found in all three regions. As we go through the time-step shown in the figure attached, it can be seen that a color shift of bright to dark was observed, indicating that less and less oil was available in the core. The color shift is in agreement with the oil production observed on the measuring cylinder. It is also important to note that the color change throughout the whole part of the core plug indicates that the change of oil and supercritical CO_2 saturation occurred in the majority of the core sample, including the center. This observation is highly important as the center region of a shale core plug sample will not be accessible if one solely depends on applying pressure differential on the plug.

On the next part of the images, CT-scan results from the SASI process are presented. Due to the relatively insignificant volume of oil produced by this method, only the initial and final timesteps are shown. As mentioned before, the CT-number of water is lower than the CT-number of the doped oil used in this experiment. Therefore, a change of color to a darker color should be observed on the CT images as a specific volume of oil was produced. As expected, color shifts from green to red, purple to blue, and so on were observed.

Discussion of Color Change of Produced Oil from both EOR Techniques

During the experiment, an unexpected phenomenon was observed in the produced oil obtained from each step of the experiments. Different colored oils produced from gas and surfactant were observed, where oil produced from surfactant was found to be darker compared to oil produced from gas. The color of produced oil from CO_2 huff-n-puff experiment and spontaneous imbibition experiment are dissimilar due to the different mechanisms of these two EOR techniques. To compare the color difference of the oil recovered from the two tests and discuss the mechanism behind each technology, we plotted the produced oil images in Fig. 14. The color of the original oil is presented to make a comparison with the color of produced oil from both CO_2 and imbibition processes.

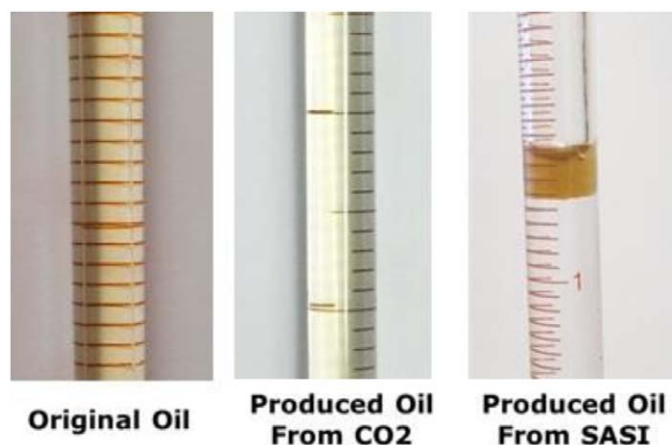


Figure 14—Change in the color of oil observed from original oil to produced oil from CO₂ process and oil recovered from surfactant-assisted spontaneous imbibition experiment

Figure 14 presents a color comparison plot of original oil and produced oil. The color of produced oil from CO₂ huff-puff experiments was light yellow which is very similar to the original oil color. However, the color of produced oil from surfactant-assisted spontaneous imbibition experiments was much darker, which shows a brownish color. Typically heavier components of oil lead to a darker color. As the system pressure went beyond MMP, gas phase can be miscible into the oil phase that results in oil swelling and viscosity reduction. The lighter and intermediate components of the oil vaporized into the gas phase and consequently expelled out from the shale core plug. The produced oil from gas injection process consists of these lighter and intermediate components of the original oil. Thus, the produced oil has a lighter color.

The primary mechanism of surfactant-assisted spontaneous imbibition experiments to enhance oil recovery in ULR is wettability alteration. Surfactant molecules interact with the wall of rock surface to form a stable double layer. During this process, the layer of oil attached to the rock surface was replaced by the surfactant molecules. The heavier components of oil have a preference to attach close to the shale core surface. Therefore, the oil produced from spontaneous imbibition experiments contained a significant portion of heavier components of oil which shows a brownish color.

Similar color shift on the produced oil was also observed on surfactant-assisted spontaneous imbibition experiments conducted with samples from Permian Basin. Same experimental setup and procedures were applied, but at different reservoir temperature of 155°F and with different materials. Core samples went through the aging process with crude oil from the same formation under the reservoir temperature for 3 months to restore their original reservoir conditions. By using various surfactants and salt-blended aqueous phase solutions, the volume of produced oil was recorded and analyzed periodically to calculate the ultimate recovery factor and to identify any significant phenomenon.

Noticeable change in color of produced oil was also observed throughout the experiment with most cases of aqueous phase solutions, and the difference was the most significant when surfactant was present. As shown in Fig. 15, all aqueous phase solutions were effective in producing extra oil through spontaneous imbibition tests. However, distilled water without surfactant additives produced strikingly lighter colored oil for the entire duration of the experiment, compared to oil produced by solutions with surfactants. In addition, imbibition rate and color change of produced oil during the experiments varied by the type of surfactant. Surfactant 02 and 03 started producing lighter colored oil in the early time but resulted in darker black oil at the end of experiments, while Surfactant 01 produced dark-colored oil even in the very early stage of the experiment and continued until the end.

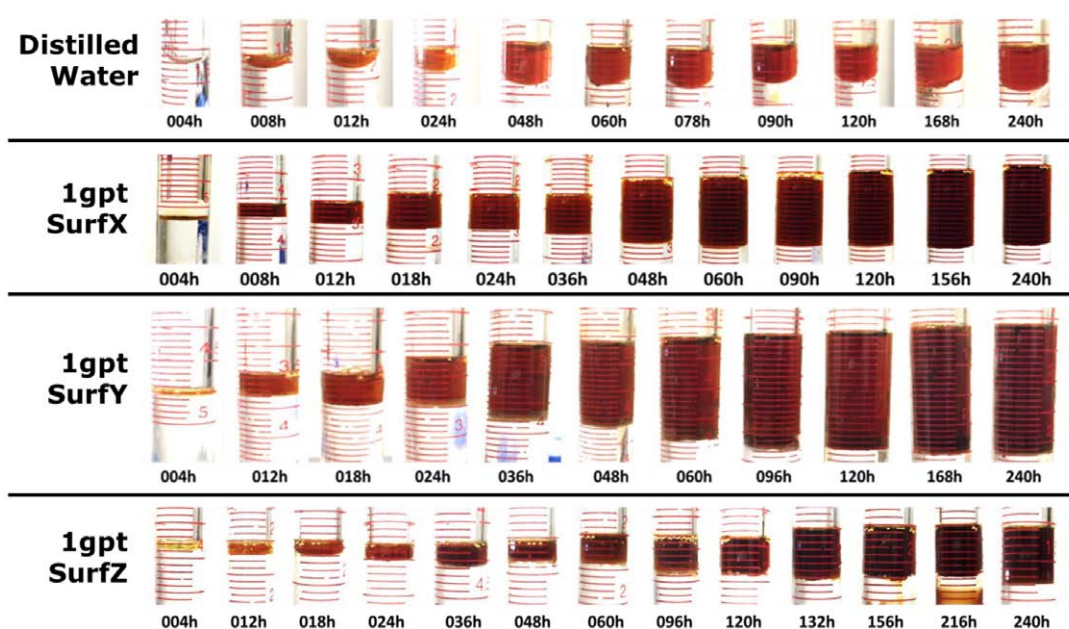


Figure 15—Physical changes of produced oil during spontaneous imbibition experiments

Based on these phenomena and observations, it can be concluded that there are possible compositional changes of produced oil taking place during spontaneous imbibition. All aqueous phase solutions seemed to access bigger pores first and extract easier and lighter component within the matrix. Then, with the support of wettability alteration, IFT reduction, and surfactant ionic interactions, solutions slowly penetrated into smaller pores and displaced heavier components of hydrocarbon. More detailed analysis of oil, such as periodic gas chromatography, is required to accurately determine and justify the actual compositional change during the production, but this strengthens the ability and effectiveness of surfactants on improving oil recovery regarding not only volume but also the composition of crude oil.

Summary

In this study, we presented a thorough evaluation of a hybrid EOR technique of combining gas injection and surfactant-assisted spontaneous imbibition in unconventional shale reservoirs. The correlated experimental workflow and set up were demonstrated at the beginning of this manuscript. The CO₂ huff-n-puff experiments were performed at the different pressure on the re-saturated sidewall core from ULR to investigate the potential of gas injection enhancing oil recovery in ULR. Then, we conducted surfactant-assisted spontaneous imbibition experiments by using the core plugs which have finished gas injection experiments to study the feasibility of this EOR technique in ULR as well as verify the efficiency of this hybrid EOR in liquid-rich shale reservoirs. MMP, contact angle, and IFT measurements were performed to determine the experimental pressure range of gas injection test and evaluate the effectiveness of surfactant in altering wettability. Next, we scanned the core plugs periodically during both gas injection and spontaneous imbibition experiments. By using CT-scan technology, we study the fluid movement inside the core plugs and visualize oil extraction process through CT images. Finally, the color change of produced oil from CO₂ huff-n-puff experiments and spontaneous imbibition experiments is investigated to reveal the mechanism of the two EOR techniques in ULR.

The results of CO₂ injection experiments demonstrated that CO₂ - EOR in unconventional liquid reservoir through a hydraulic fracture could lead to high recovery of the oil naturally stored in the rock matrix. The recovery factors increase as the experimental pressure of CO₂ huff-n-puff experiments increase. The maximum recovery factor is achieved from the experiment under 3,500 psi that 49% of OOIP recovered by CO₂ huff-n-puff process in ULR core plug.

In addition, surfactant-assisted spontaneous imbibition is also a practical EOR technique in ULR, which has been validated by the experimental observations and field experience. Extra 8.6% of oil recovered by spontaneous imbibition test by using the core plug that has produced 49% of OOIP from gas injection experiment. Based on the experimental results, these two EOR techniques can be active together and open the possibility to realize the optimum oil recovery by utilizing combine EOR techniques in ULR.

The color of the produced oil from CO₂ huff-n-puff experiments is much lighter than the color of the oil produced from surfactant-assist spontaneous imbibition. The injected gas can effectively penetrate further into the shale formation and extract the lighter components of oil out from the reservoir matrix. Due to a different mechanism of the surfactant imbibition EOR to gas injection EOR, heavier parts of hydrocarbons are produced from surfactant-assisted spontaneous imbibition processes. These observations also verify the applicability of combining EOR technique in ULR.

Conclusions

1. CO₂ - EOR in unconventional liquid reservoir through a hydraulic fracture can lead to high recovery of the oil naturally stored in the rock matrix. This is observed in the huff-n puff experiments in re-saturated Eagle Ford core plugs.
2. The recovery factors increase as the experimental pressure of CO₂ huff-n-puff experiments increase. The maximum recovery factor is achieved from the experiment under 3,500 psi that 49% of OOIP recovered by CO₂ huff-n-puff process in ULR core plug.
3. Our results show that the operating pressure strongly affects the recovery factor, and that further increasing pressure beyond the MMP results in higher recovery factors.
4. Surfactant-assisted spontaneous imbibition is a practical EOR method in ULR. More oil can be recovered utilizing this technique by wettability alteration and IFT reduction.
5. Additional 8.6% oil recovered through spontaneous imbibition experiment on the core plug that has produced 49% of OOIP from CO₂ huff-n-puff experiment.
6. The two EOR techniques of gas injection and surfactant imbibition effect together and open a possibility to achieve an optimum oil recovery by using a hybrid EOR technique in ULR.
7. The color of the produced oil from gas injection experiments is lighter than that from spontaneous imbibition that heavier components of hydrocarbons are extracted from spontaneous imbibition.

Acknowledgments

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Nomenclature

P_c	= Capillary pressure
r	= Pore radius
θ	= Contact angle
σ	= Interfacial tension (IFT)
CA	= Contact angle
CT	= Computed tomography
DSA	= Drop shape analyzer
EOR	= Enhanced oil recovery
MMP	= Minimum miscibility pressure
$OOIP$	= Original oil in place

PI = Producing interval
SASI = Surfactant-assisted spontaneous imbibition
SI = Soaking interval
ULR = Unconventional Liquid Reservoir

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