

SPE-182567-MS

Hybrid EOR Technology: Carbonated Water and Smart Water Improved Recovery in Oil Wet Carbonate Formation

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This paper was prepared for presentation at the SPE Annual Caspian Technical Conference & Exhibition held in Astana, Kazakhstan, 1-3 November 2016.

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Abstract

Search for an alternative, cheap and environmental friendly EOR method has resulted in the development and subsequent field implementation of Smart Water Injection (SWI) and CO₂ injection (COI). Though miscible COI has multi-fold advantages, carbonated water injection (CWI) may surpass COI in terms of volume requirement and efficiency. This research work is focused on finding the benefits of combination of SWI & CWI in terms of reduced residual saturations in oil wet carbonate formation.

In order to measure the recovery efficiency, core flooding experiments are conducted with SWI as a tertiary recovery following sea water secondary recovery. This is followed by quaternary recovery using smart water saturated with CO_2 (CSWI). Three high potential smart water compositions are screened and the best one is chosen for oil recovery studies. Effluent samples collected at pre-defined pore volume injection intervals are analyzed in ppm level using ICP-MS. The analysis is supported by NMR porosity distribution studies, interfacial tension and zeta-potential studies.

Core flooding results showed that tertiary recovery with SWI resulted in 4.8 - 9.5% recovery, whereas the quaternary recovery with CSWI resulted in 5.7% to 13.6% additional oil. The underlying physical processes and the pore-scale mechanisms of fluid-fluid and fluid-solid interactions during SWI-CWI are explained in terms of surface potential, electrical double layer, oil swelling factor, droplet coalescence, wettability alteration and flow diversion due to fluid redistribution within the pore network.

From this study it is established that CSWI could be a novel and promising EOR technique among the latest EOR methods. CSWI has an advantage over COI due to the fact that it exists as a dissolved gas instead of free phase. It eliminates the requirement for high pressure system, the problems of gravity segregation and poor sweep efficiency. Combination of CWI with SWI can make it an effective EOR solution with significant cost advantage, higher oil recovery in addition to the environmental benefits of CO_2 sequestration.

Introduction

Smart Water Flooding is the process that has been recently established in the oil industry as an effective EOR process. This method has been verified in laboratory (Webb et al., 2004) and also through a few successful field trials (Lager et al. 2008). Miscible CO_2 at high saturation may also enhance the oil recovery over the

conventional water flooding due to its higher solubility in oil thus reducing the oil viscosity, increasing the density, swelling of oil and increasing viscous force of the injecting fluid (Yongmao et al., 2004).

Most researchers (Lager et al. 2008) relate the main mechanism of smart water EOR to multicomponent ion exchange (MIE), where originally oil-wet or mixed-wet reservoir rocks tend to change to water-wet because of the desorption of electrically polar oil compounds from the rock surface. The divalent ions of injected smart water are exchanged with either cations of the rock surface, thus changing the surface charge of the rock (Yousef et al. 2011). There are other possible mechanisms such as IFT reduction, rock dissolution and electrical double layer expansion. However, none of the mechanisms has been universally accepted as the most predominant mechanism so far.

Miscible CO_2 injection into the reservoir is a well established EOR technique, which contributed to significantly volume of oil recovery over the past decades. However recently the benefits of injecting dissolved CO_2 with brine (CWI) has been seen to have merits over injecting miscible CO_2 as supercritical fluid. Due to better mass transfer mechanism, oil recovery by means of CWI can be more efficient in terms of reducing the oil density and viscosity, oil swelling factor and by creating higher viscous forces than CO_2 alone (Riazi et al. 2009). Moreover, this technique does not require a huge amount of CO_2 and more CO_2 can be stored in the subsurface (Sohrabi et al. 2009) at significantly lower pressure.

Understanding the importance and simplicity of these two EOR methods gives very reasonable justification to apply them sequentially or simultaneously. This novel EOR technique does not require high capital and operational cost, contrary to other EOR methods.

Investigation on the effect of carbonated smart water on carbonate cores is the objective of this study as very little or no work is done so far in this area. This objective is met thorugh a systematic laboratory investigations starting with screening the best brine and to establish the procedure of dissolving CO_2 of known quantity in brine for further core flooding studies. The possible pore-scale mechanisms behind carbonated smart water flooding is studied based on ICP-MS, NMR and core flooding studies.

It is envisaged that, if successful in field pilot, this EOR technique may gain huge importance in the Middle East oil and gas industry since a huge amount of oil is expected to be left behind in the carbonate formation after secondary recovery processes.

Experimental Procedure

Initial preparation of core plugs, brines and oil

The core plugs that are received from one of the carbonate oil formations of Abu Dhabi, shown in Table 1, are subjected to standard procedure of cutting, cleaning, measuring dimensions, determination of helium porosity and air permeability and finally saturation with synthetic formation brine. Different brines, including sea water, formation water and smart brines are prepared, filtered and their density and viscosities are measured. Oil sample is received from the same oil reservoir of Abu Dhabi, which is de-gassed, de-watered filtered through 0.45 micron filter. After preliminary preparation procedures, all core plugs are saturated with formation water at pressure followed by vaccum saturator.

Core number	Length (cm)	Diameter(cm)	Bulk Volume (cc)	PoreVolume(cc)	Porosity %	k (md)	S _w (%)
41	8.55	3.81	97.87	6.75	6.89	0.04	
33	6.72	3.81	76.65	16.98	22.15	20.25	99.9
403	6.54	3.81	74.79	10.30	13.77	7.38	53
404	6.52	3.81	74.64	8.93	11.97	4.35	68.7
	(continued on next page.)						

Table 1—Parameters of core plugs

Core number	Length (cm)	Diameter(cm)	Bulk Volume (cc)	PoreVolume(cc)	Porosity %	k (md)	S _w (%)
190	6.50	3.79	73.74	12.42	16.84	338.45	90
198	6.54	3.79	73.99	12.94	17.48	1.50	98.4
52	5.40	3.82	62.18	8.48	13.64	0.19	
200	6.57	3.79	74.44	12.39	16.64	8.16	98
411	6.12	3.81	70.03	11.17	15.95	1.40	70.3
415	6.51	3.82	74.66	8.97	12.02	5.52	50.7
409	6.52	3.82	74.82	6.45	8.62	1.16	83.16

Table 1—(continued).

Table 2—Oil density and viscosity

Temperature °C	Density g/cc	Viscosity cP
20	0.8364	5.34
40	0.8219	3.44
60	0.8074	2.26
80	0.7991	1.61
90	0.7919	1.43

NMR porosity studies

Pore size distribution (PSD) of the core plugs are measured through NMR Rock Core Analyzer before and after coreflood recovery studies in order to see the change in pore size distribution and cumulative porosity. In both cases, the core plugs are cleaned and fully saturated with formation brine.

IFT studies

Interfacial tension studies between different brines and oil were done at 90 °C on Spinning Drop Tensiometer. The pH of brines used in this study were brought down using hydrochloric acid in order to create the acidic environment similar to carbonated water. This study was not done using carbonated water due to pressure limitation of the equipment.

Zeta Potential studies

Zeta Potential surface charge measurements are conducted on both rock/brine and rock/brine/oil systems. As in IFT studies, the brines used in this study are with reduced pH using hydrochloric acid again due to pressure limitation of the equipment.

Microemulsion studies

Different brines (with the pH values ranging from 14 to 1) and oil were placed in glass pipette in order to see if there is a possibility of forming microemulsion due to brine composition and pH variation. The observation was done for one week, in order to see if there is a micro emulsion generation at oil/brine interface. As in IFT and surface charge studies, these studies are also were done using brines with reduced pH using Hydrochloric acid.

Dissolving CO₂ in brine

In order to study the recovery efficiency of carbonated water flooding, CO_2 must be dissolved in brine. Dissolution of CO_2 in brine is performed at 350 psi and 100°C. At this condition, 6 cc of CO_2 can be dissolved in I cc of water, as shown in Figure 1 (Wiebe and Gaddy 1940). Solubility of CO_2 in water is converted to solubility in brine using Equation 1 (Kechut et al. 2010).



Figure 1—Solubility of CO₂ in pure water as a function of Temperature and Pressure (Wiebe and Gaddy 1940)

$$\log\left(\frac{R_{sb}}{R_{sw}}\right) = -0.028 * S * T^{-0.12} \tag{1}$$

The dissolution of CO_2 in brine is performed by the following steps:

- 1. Collect required volume of brine into cylinder and maintain required pressure and temperature.
- 2. Start injecting CO_2 into the same cylinder at the same pressure as for brine using gas booster.
- 3. Start decreasing the pressure in the brine cylinder so that, the CO_2 can enter it because of the pressure difference. Control the volume in the cylinder until required volume of gas is collected.
- 4. Once the required volume of CO₂ and brine are in cylinder, keep two fluids in the cylinder under required pressure and temperature

Core flooding

Core plugs that are selected for the core flooding studies are brought to S_{or} prior to core flooding studies. After dissolving CO₂ in brine, the core flooding process is started at 350 psi backpressure and 100°C.

Three core flooding studies were performed by the following steps:

- 1. Secondary recovery using sea water
- 2. Tertiary recovery with sea water with four times sulfate (SW4S), since this smart water is found to be the best in terms of electrical double layer expansion obtained from zeta potential studies.
- 3. Quaternary recovery with sea water with four times sulfate saturated with CO₂ (SW4S+CO₂).

Results and discussions

Microemulsion studies

Figure 2 and 3 display the microemulsion study results on day one and day seven. Even at the end 7th day there is no change at the oil-brine interface, thus it can be concluded that, no microemulsion is formed between oil and different brine samples at 95°C.



Figure 2-Microemulsion phase behavior study between crude oil and brines at different pH values (Day 1)



Figure 3—Microemulsion phase behavior study between crude oil and brines at different pH values (Day 7)

IFT studies

Figure 4 to 8 are showing the results of IFT studies between oil and five brines including DI and SW. The IFT values are plotted versus pH of brine (from 7 to 1) for each brine.



Figure 4—IFT between DI water and oil



Figure 5—IFT between 0.1SW and oil









Figure 8—IFT between SW4S and oil

From the above figures it can be seen, that IFT between oil and different brines are reducing with reducing brine pH. At pH 7, the IFT for all brines are showing around 25 mN/m, while at pH around 1, the IFT reduced till almost 12 mN/m. From reservoir engineering point of view a reduction of 13 mN/m IFT is

insignificant in order to produce any significant immobile oil from the pore throats. Thus reduction of IFT is considered as a significant mechanism for additional oil recovery.

Zeta Potential studies

Surface charge studies through Zeta Potential measurements are performed on the rock/brine (R-B) and rock/brine/oil (R-B-O) systems. Table 3 shows the surface charge of both systems. The values in the table are the average of five measurements for each brine-rock system. Rock-brine system for all brines are showing positive surface charge, except the DI water. This is because the carbonate rock surface is generally positively charged. However, when oil is added into the system, which has negatively charged acidic molecules, the surface charge is change to negative. For DI water/rock/oil system the charge down to -37.9 mV. The idea behind injecting smart water is that it should bring the surface charge of the system to positive value, closer to the R-B system, signifying that the stuck oil is released from the rock surface due to the ion exchange and double layer expansion phenomena.

Fluid Type	R-B in mV	R-B-O in mV
DI	-2.27	-37.9
SW	2.45	-0.75
0.1SW	0.635	-6.19
SW4S	1.0025	1.63
0.1SW4S	1.1125	-1.65

Table 3—Surface charge of rock brine and rock/brine/oil systems

It is evident from Table 3 that all four brines brought the surface charge of the rock powders close to neutral, but only SW4S has actually resulted in positive surface charge. Therefore, in terms of surface charge modification and oil releqase aspect, SW4S seems to be the best brine of choice. The reason behind the higher efficiency of SW4S is the presence of four times increased sulfate ions in the sea water along with higher concentration of Ca & Mg ions. Since both $SO_4^{2^-}$ in the brine and Ca^{2^+} on the carbonate rock surface are divalent ions, the negatively charged sulfate ion is relatively strongly attracted to the carbonate rock surface than the carboxylic acids. Therefore, when $SO_4^{2^-}$ is adsorbed on the rock surface, it becomes a catalyst for Ca^{2^+} and Mg^{2^+} ions, so they will be co-adsorbed on the stern layer of $SO_4^{2^-}$ (Yi and Sarma, 2012). When PDIs (Ca^{2^+} , Mg^{2^+} and $SO_4^{2^-}$) forms electrical double layer on the carbonate rock surface, the attached oil phase will be released from the rock surface and free to flow through the pore throats.

Core flooding studies

Three core flooding experiments on three different cores are conducted in order to study the efficiency of carbonated smart water flooding in carbonate rocks. Figure 9, 10 and 11 are showing the plots of displacement efficiency of brine flooding versus pore volume (PV) injected for three core flooding experiments conducted, using three different cores with permeability values of 1.5 md, 8.167 md and 20.25 md respectively. This selection was made with the intention of studing the effect of permeability on recovery, in addition to the brine properties. Identical flood parameters for secondary, tertiary and quaternary are adopted for all the three core flooding studies. Secondary and tertiary stages are performed at increasing flow rates from 0.1 cc/min to 0.5 cc/min as shown in the plots. Flow rate is changed each time when no more oil production is observed for at least one pore volume of brine injection. Each stage of flooding is continued until 0.5 cc/min flood rate is reached, which is seen to be close to the critical velocity of these core plugs. When there was no more oil production at 0.5 cc/min, next stage of core flooding is started. Quaternary recovery stage was done at 0.5 cc/min only in order to avoid the hydrodynamic effect and to

be able to understand the effect of carbonated smart water only. When there was no more oil production observed during carbonated smart water flooding, the injection is stopped and the core is allowed to to soak in CW for 24 hours. After 24 hours of static condition, the injection process is started again. This step was taken in order to see if there is any possibility of further oil production due to reaction in the pore throats.



Figure 9—Core flooding 1



Figure 10—Core flooding 2



Figure 11—Core flooding 3

Table 4 shows the results of three core flooding experiments along with core properties. Core 198 is the least permeable core with 1.5 md permeability and highes water saturation (S_{wi} 35.1 %), while core 200 is 8.167 md with S_{wi} 30% and core 33 is 20.25 md with S_{wi} 32.1%. The secondary recovery stage resulted in 52.7% for core 198, 69.3% for core 200 and 63% for core 33. The recovery efficiency from tertiary and quaternary stages resulted in reverse order. Core 198 showed highest recovery, while core 200 showed lowest recovery. Interestingluy, the overall recovery for all cores is around 76 %. The saturation and recovery percentages seem reasonable since it can be correlated with NMR porosity data.

	Core 198	Core 200	Core 33
k (md)	1.509	8.167	20.25
S _{wirr} (%)	35.1	30	32.1
Oil in place, cc	9.6	7.8	10.6
Secondary recovery, cc	6.66	4.11	6.7
Secondary recovery, %	52.7	69.3	63
Tertiary recovery, cc	0.74	0.19	0.07
Tertiary recovery, %	9.4	1.9	0.66
Quaternary recovery, cc	1.08	0.55	1.18
Quaternary recovery, %	13.9	5.7	11.8
Cumulative recovery	76	76.9	75.46

Table	4—Core	flooding	results
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In all the three core flooding studies, significant amount of oil is produced from quaternary recovery compared to tertiary recovery, which proves the high efficiency of Carbonated water injection in recovering trapped oil blobs, which could not be recovered even after smart water EOR.

The fllowing could be attributed as the possible mechanisms behind improved oil recovery from carbonated smart water flooding. There are three main mechanisms that can happen when carbonated water is injected in the pores: swelling and coalescence of trapped oil ganglia, local flow diversion and oil viscosity reduction. Therefore, the macroscopic sweep efficiency of the brine will improve significantly due to the

swelling and coalescence of trapped oil particles and reduction of oil viscosity. With brine saturtated with CO_2 , the injected fluid becomes more viscous, while with mass transfer, oil phase becomes less viscous. The reduction of oil viscosity results in better mobility ratio between oil and water phases. The swelling and coalescence is considered to be main mechanism in the reservoirs containing light oil due to the higher solubility of CO_2 in lighter oil. However, viscosity reduction of oil is considered to be the main mechanism in the reservoirs with viscous oil, the viscosity reduction will reduce the interfacial tension between oil and injected brine leading to the improvement of oil recovery significantly. This mechanism behind carbonated smart water flooding might be the dominating mechanism behind improved oil recovery.

NMR porosity studies

NMR porosity distribution and cumulative porosity for the cores 33, 198 and 200 are measured before and after core flooding. According to T2 distribution curves of pre-flooding condition, core #200 is mostly composed of macro pores and almost no micro pore. Core #200 is having mostly macro pores and little micro pores. Also, core #33 is having mostly macro pores and little micro pores.

From NMR studies of core 198 it can be seen that there is not much change in porosity distribution curve. Both PSD and cumulative porosity are showing almost same result with some noticeable change at the micro and macro-region. The incremental porosity at the macro-region can be attributed to carbonate dissolution, as major flow occurred through these paths, whereas the reduced porosity at the micro-region is possibly due to blockages of porosity due to deposition of sulfate scales. The unchanged PSD in the meso-region might be due to the balance between rock dissolution and precipitation of ions.

From Figure 13 it can be seen that the overall porosity is increased by 0.5 %, which proves that the carbonated dissolution due to the flow of carbonated water is more than the pore blockage caused by sulfate scale deposition.



Figure 12—Core 198 NMR Porosity distribution comparison: before and after CF



Figure 13—Core 198 NMR Cumulative porosity comparison: before and after CF

The core 200 has become more porous after core flooding. This change in porosity can be seen from cumulative porosity and PSD curve. The cumulative porosity before core flooding was 15.3%, while after flooding it is estimated as 17.25 %. The PSD has also became higher in macro porosity.



Figure 14—Core 200 NMR Porosity distribution comparison: before and after CF



Figure 15—Core 200 NMR Cumulative porosity comparison: before and after CF

Additional porosity in macro-region is possibly due to carbonate rock dissolution. While slight reduction of PSD in micro-region is due to blockage of pores by precipitation of sulfate ions. Overall porosity after CF is increased due to the dissolution of calcium and magnesium ions.



Figure 16—Core 33 NMR Porosity distribution comparison: before and after CF



Figure 17—Core 33 NMR Cumulative porosity comparison: before and after CF

Post core flooding NMR plots of core 33 are showing a significant difference compared to NMR plots before CF. The cumulative porosity of the core is increased to 22.76% compared to 20.45% before CF, while PSD shifted towards more macro porosity.

PSD plot is showing changes both in micro and macro-region. The incremental porosity in macro-region is due to the dissolution of calcium and magnesium ions, since it is main flow path for the injected fluid. There is minor reduction in micro region due to the precipitation of sulfate scale which blocks the pores.

ICP Analysis

Effluents from the core flooding studies are tested for the ionic composition in order to compare pre flooding and post flooding concentration in the brine. This comparison can give idea of rock dissolution due to carbonated water flooding (Kono et al., 2014). ICP studies are done for PDI's (Magnesium, Sulfate and Calcium) which are potentially affecting the recovery efficiency of either brine. ICP analysis from three core flooding studies at different stages with different brines are presented in the below figures.

Effluent analysis from core flooding 1

Figures 18 to 26 are showing Mg, S and Ca concentrations in injected brine and effluent from different stages of flooding. During the secondary recovery stage, there is an increase in Mg concentration in produced brine compared to the injected brine. Increase in concentration might be due to the dissolution of Mg ions from the rock surface as a result of ionic reaction. Different scenario is observed during the tertiary and quaternary recovery stages. Slight decrease in magnesium concentration is observed during tertiary recovery stage and after keeping the SW4S with CO_2 in the core plug at a static condition for 24 hours. When the fluid and rock are at static condition, more reaction might happen which leads to more dissolution of Ca and Mg ions.







Figure 19—ICP analysis of CF1: Sulfur concentration











Figure 22—ICP analysis of CF2: Sulfur concentration







Figure 24—ICP analysis of CF3: Magnesium concentration



Figure 25—ICP analysis of CF3: Sulfur concentration



Figure 26—ICP analysis of CF3: Calcium concentration

Sulfur concentration in the effluent of core flooding 1 is decreased at all stages of flooding compared to the concentration in injected brine. This behavior can be best explained in view of the precipitation and deposition of sulfate ions in the pore space. However, calcium concentration in the injected brine and effluents is showing increased trend in all stages of flooding, which means that Ca ions on the rock surface is dissolved both with smart water and carbonated smart water. Overall, from the ionic concentration studies of injection brine and effluents from CF 1, it can be seen that both precipitation and dissolution of ions are occurring at the same time. The effect of ionic reaction from CF1 can be confirmed with NMR plots before and after core flooding. The ICP analysis helps to explain why there is no significant change in porosity and porosity distribution of the core.

Effluent from core flooding 2

In CF 2 plots, trends are showing different behavior compared to CF 1. Mg concentration is increasing compared to injected brine during SW flooding and slightly increased during SW4S flooding. There is also a slight increase while flooding with carbonated smart water during quaternary stage and after aging. This could be due to higher local dolomite present in this specific core. However it is difficult to explain this observation unless a complete compositional analysis of the rock is available.

Sulfur concentration is seen to be decreasing at all stages of core flooding 2 compared to the concentration in the injected brine. Thus adsorption and precipitation of sulfate ions is evident. It is also evident that sulfate precipitation is continuing even at quaternary recovery stage. Concentration of calcium ion is increasing compared to injected brine indicating rock dissolution. Overall, during core flooding 2, more ionic dissolution happened and less precipitation took place. Dissolution of ions from the rock surface can be compared on NMR studies before and after core flooding studies. The porosity and porosity distribution is increased after core flooding.

Effluent from core flooding 3

ICP effluent studies of CF 3 is similar to core flooding 2. Mg concentration in the effluent from SW is increased compared to injected SW and slightly increased during tertiary and quaternary stages. In effluent from carbonated water the sulfur concentration is decreased compared to the injected brine. Calcium concentration in CF 3 is increased in the effluents during all stages of core flooding.

Effluent analysis from all the three core flooding show that calcium dissolution happens at all stages of core flooding. Interestingly, 24 hours of aging the core with carbonated water at static condition leads to the same increase in Ca concentration. Both smart water and carbonated smart can dissolve the calcium ion while flooding. However, the concentration of sulfate ion is decreasing in the effluent compared to injected brines which indicates to the deposition of sulfate ions. There is not much difference in the concentration

of magnesium ions between the injected brine and the effluents. Thus, the ionic reaction of magnesium on the rock surface is not affecting the porosity of the core significantly.

Conclusion

From the studies conducted and the results obtained the following conclusions can be made:

- Among the three different smart waters (0.1SW, 0.1SW4S and SW4S) initially chosen for screening, SW4S is found to be the most potent brine in terms of IFT reduction and EDL expansion. Thus SW4S is chosen as the smart brine for further recovery studies.
- Saturation of the smart water with CO₂ can recover significant amount of immobile oil left after tertiary recovery stage. This is attributed to the impact of CO₂ mass transfer from brine to oil.
- The possible mechanism behind additional oil recovery after carbonated water flooding is rock dissolution, in addition to viscosity ratio improvement, and oil swelling and reconnection of oil ganglia. The pores size distribution and ionic composition studies of the injected brine and effluent indicates that the rock dissolution happened after flooding the core with carbonated smart water.
- From the three coreflood experiments conducted in three different permeability core plugs, it is seen that permeability has very little significance in terms of recovery efficiency.

Nomenclatures

- CF Core flooding
- CO₂ Carbon Dioxide
- CW Carbonated Water
- CWI Carbonated Water Injection
 - DI De Ionized water
- EDL Electric Double Layer
- EOR Enhanced Oil Recovery
- FW Formation Water
- IFT Interfacial Tension
- MIE Multicomponent Ionic Exchange
- NMR Nuclear Magnetic Resonance
- *OOIP* Original Oil In Place
 - PDI Potential Determining Ions
 - PV Pore Volume
 - R_{sb} CO₂ solubility in brine
 - R_{sw} CO₂ solubility in water
 - *S* Brine salinity
 - SW Sea Water
- SW4S Sea water with four times sulfate

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