Investigation of Oil Recovery in Fractured Carbonate Rock by Equilibrium and Non-Equilibrium Gas in Weakly Water-Wet Condition

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Received: January 4, 2016	Accepted: February 14, 2016	Online Published: February 26, 2016
doi:10.5539/eer.v6n1p14	URL: http://dx.doi.org/10.5539/eer.v6r	11p14

Abstract

The purpose of the three experiments described in this paper is to investigate the efficiency of secondary and tertiary gas injection in fractured carbonate reservoirs, focusing on the effect of equilibrium gas, re-pressurization and non-equilibrium gas. A weakly water-wet sample from Asmari limestone which is the main oil producing formation in Iran, was placed vertically in a specially designed core holder surrounded with fracture. The unique feature of the apparatus used in the experiment, is the capability of initializing the sample with live oil to obtain a homogeneous saturation and create the fracture around it by using a special alloy which is easily meltable. After initializing the sample, the alloy can be drained from the bottom of the modified core holder and create the fracture which is filled with live oil and surrounded the sample. Pressure and temperature were selected in the experiments to give proper interfacial tensions which have been measured experimentally.

Series of secondary and tertiary gas injection were carried out using equilibrium and non-equilibrium gas. Experiments have been performed at different pressures and effect of reduction of interfacial tension were checked by re-pressurization process. The experiments showed little oil recovery due to water injection while significant amount of oil has been produced due to equilibrium gas injection and re-pressurization. Results also reveal that CO_2 injection is a very efficient recovery method while injection of C_1 can also improve the oil recovery.

Keywords: equilibrium gas, fractured reservoirs, non-equilibrium gas, slightly water-wet

1. Introduction

Normally, large amounts of oil may be trapped in fractured reservoirs due to capillary forces and gas injection could be an efficient EOR method for such reservoirs. In fact, gas injection into an oil reservoir, in either equilibrium or non-equilibrium, has been applied for a long time where enhanced hydrocarbon recovery from the matrix-trapped oil is the main goal. The recovery mechanisms in fractured reservoirs depend on the matrix geometry and reservoir fluid properties. The main mechanisms under secondary and/or tertiary gas injection are essentially gas-oil gravity drainage and gas-oil diffusion if the reservoir pressure is above the bubble point pressure.

When a tall and permeable oil saturated matrix block is surrounded by gas in the fracture, oil drains from the matrix as a result of the density difference between oil in the matrix and gas in the fracture. Therefore gravity drainage may take place provided the threshold height is smaller than block height. Recovery mechanism in Asmari reservoirs is essentially gravity drainage since the matrix block is quite high (Saidi, A, M, 1987).

However, performance of gravity drainage is limited by threshold height and matrix block size. This is the case in short matrix block and low permeability chalks of the North Sea; e.g. Ekofisk, implying that gravity drainage may not be an efficient recovery mechanism. In this case, if the reservoir pressure is increased and a non-equilibrium gas is injected to the reservoir, oil recovery will increase. In non-equilibrium gas injection, compositional effect may play an important role leading to additional oil recovery (Saidi, A.M., 1987). A number of researchers have studied non-equilibrium gas injection in fractured chalk focusing on compositional effect and component exchange between matrix and fracture (Øyno *et al.* 1995, Thomas *et al.* 1991, Karimaie *et al.* 2008). However, the recovery mechanisms of high pressure gas injection are still under question and it may depend on the nature of the gas. Among different types of dry gas, CO_2 has a strong attraction to oil and is very effective for displacing oil from the matrix block. CO_2 could offer a good opportunity to recover the remaining oil in the final (tertiary) phase of the reservoir. Holm and Josendal (1974) found that CO_2 has a great depth of vaporization and extraction of hydrocarbons from crude oil. It can extract the heavier components (C_6-C_{30}) and can be miscible with crude oils that have little C_2-C_6 components. This feature makes the CO_2 injection applicable to many reservoirs which are no longer suitable for lean gas injection, but they can still be a good candidate for secondary and tertiary CO_2 injection. Hujun *et al.* (2000) investigated CO_2 gravity drainage in artificially fractured core using dead oil at the reservoir temperature of 58.9 C and concluded that CO_2 gravity drainage could significantly enhance oil recovery after water flooding in the naturally fractured Sprayberry Trend Area. In their experiment artificial fracture was defined as the gap between two pieces of the core.

Lean gas injection is also an attractive recovery option. Morel *et al.* (1990) experimentally studied the combined effect of gas diffusion and stripping in the matrix blocks of light oil fractured reservoirs subjected to methane or nitrogen gas flooding. They concluded oil recovery was about twice as fast with methane as with nitrogen. Le Romancer *et al.* (1994) performed similar experiments in 1-D conditions on a chalk core in the presence of different water saturation. The saturation profile in their experiment showed a strong capillary end effect for nitrogen injection with an accumulation of oil near the fracture. Darvish *et al.* (2006) performed CO₂ injection experiments at reservoir conditions using a methodology developed by SINTEF for initialization of the sample with live oil. Karimaie *et al.* (2008) studied injection of nitrogen and CO₂ in fractured chalk at reservoir condition and conclude that oil recovery by N₂ injection is quite low compare to CO₂ injection.

However, very few laboratory works on CO_2 and C_1 gas injection on fractured rock samples using live oil have been reported in the past and as can be seen, the bulk of the experimental studies on gas injection were performed using dead (synthetic) oil in atmospheric condition which is usually not related to reservoir condition. Therefore the scarcity of experimental data and difficulty encountered in obtaining such data, have made laboratory work at reservoir conditions attractive for this process.

After carefully conducting experiments to confirm low oil recovery under water injection for Asmari limestone in atmospheric condition (Karimaie and Torsæter, 2007), gas injection tests were proposed to investigate the efficiency of equilibrium and non-equilibrium gas on this type of rock. In this paper results of experiments to investigate the efficiency of gas injection on oil recovery in secondary and tertiary cases are reported. The present work was begun by water injection followed by equilibrium gas injection and re-pressurization. After carefully conducting equilibrium gas injection tests and obtaining the final recovery, non-equilibrium gas; i.e. CO_2 and / or C_1 was injected to the system to understand the compositional effect.

2. Rock and Fluid properties

Experimental studies have been carried out on Asmari limestone outcrop from southern part of Iran as a representative of weakly water-wet sample with low permeability. However in order to make sure the wettability state of the samples, some preliminary experiments have been performed. Three plugs were prepared from the long cores used in gas injection experiments and results from plug analyses were used to understand the large scale behavior.

Amott wettability test and thin section study was performed and results show weakly-water wetting state of the samples. Detail of the experiments published by Karimaie and Torsæter 2007.

The porous medium used in gas injection experiments were cylindrical core sample with a length of 18-19 cm and 3.7-3.8 cm in diameter. The porosity was in the range of 23-24%, and absolute permeability to liquid, measured with n-heptane at the room temperature, was around 15 mD. Table 1 provides an overview of the core samples properties.

Table 1. Properties of the samples used in secondary and tertiary gas injection experiment

Exp.	Permeability	Porosity	Length
No.	(mD)	%	(cm)
1	15.2	23.0	18.0
2	14.4	23.0	19.0
3	15.0	24.0	19.0

All experiments were performed with synthetic binary mixture with a molar composition of 75.47% with

methane and 24.53% with heptane as live oil. Equilibrated oil and gas at preselected pressures below the bubble point were prepared and transferred to separate cylinders. CO_2 and C_1 were selected as non-equilibrium gases and synthetic brine with 3% NaCl which has density of 1.03 g/cm³ and viscosity of 1.07 mPas was used as water phase in the experiments.

Constant composition expansion experiment using a PVT cell shows a bubble point pressure of the mixture around 229.5 bar at 85 °C. Phase densities, oil formation volume factors and interfacial tension between the phases at different pressures close to the bubble point and constant temperature (85 °C) are shown in Table 2. Readers may refer to the published paper by Karimaie and Torsæter (2010) for additional PVT information and more details on experiments.

Table 2. PVT properties at different pressure and constant temperature (85 °C) (Karimaie Torsæter 2010)

Pressure (bara)	Oil	Gas density	Bo	IFT
	Density	(g/cm^3)	rv/stc v	mN/m
	(g/cm^3)			
220	0.407	0.223	2.28	0.15
210	0.433	0.198	2.1	0.374
200	0.452	0.178	1.98	0.686

Table 3. Summary of the experiments

	Water	Equilibrium gas	Equilibrium gas	CO ₂	C ₁
Experiment	Injection	injection at 210 bar	injection at 220 bar	injection	injection
1- Asmari		\checkmark	\checkmark	√	
limestone					
2- Asmari	\checkmark	\checkmark	\checkmark	\checkmark	
limestone					
3- Asmari	\checkmark	\checkmark	\checkmark		\checkmark
limestone					





- 1- Quizix pump.
- 2, 3-Isolated cells for oil and gas.
- 4- Isolated tube for transferring the oil and gas.
- 5- Pressure transmitter.
- 6- Steel tube containing matrix and fracture.
- 7- By-pass system.

- 8- Sealing material accumulator.
- 9- Back pressure regulator.
- 10- Condenser
- 11- Separator.
- 12- Gas wet test meter.
- 13- Gas chromatograph

3. Description of Equipment and Experimental Procedure

Initializing the sample with live oil and maintaining the pressure to prevent compositional change in fractured system, is very important and challenging issue in gas injection experiments. To overcome this problem, a method for initialization of the core with live oil using an experimental set-up has been developed to ensure the homogeneous saturation of the core plug. A schematic of the experimental equipment is given in Figure 1. It was designed for water and gas injection in fractured porous media under high pressure and temperature. A unique feature of the apparatus used in gas injection experiments, was the capability for initializing the sample with live oil and create the fracture to surround the sample afterwards.

The methodology of the experiments were based on placing the core sample inside a steel tube which has an inner diameter larger than core diameter so that the annular space between the core and tube could simulate the fracture space. Experiments were based on filling the fracture space with a meltable alloy so called "woods metal" with a melting point of around 67 °C which is an eutectic alloy of 50% bismuth, 26.7% lead, 13.3% tin, and 10% cadmium by weight, saturate the sample with dead C₇, measuring permeability, displace dead C₇ with equilibrated live oil, and draining the woods metal to create the fracture space around the core sample. After initialization and draining the woods metal from the bottom of the system, core sample was saturated with equilibrated live oil and surrounded with fracture. Readers may refer to previous published paper by Karimaie, et al (2008) and Karimaie and Torsæter (2010) for more detail on experimental methodology.

The first experiment; secondary gas injection; was started by injecting equilibrium gas at 220 bar in which IFT was equal to 0.15 mN/m. The rate of injection was 5 cm³/min at the beginning of the experiment to drain all the oil in the fracture and then decreased to 0.1 cm³/min. Gravity drainage would take place resulting in oil recovery from the matrix block. After reaching to final oil recovery, CO_2 was injected as a non-equilibrium gas to the system.

The second and third experiments were performed in order to investigate the efficiency of tertiary gas injection by equilibrium and non-equilibrium gas and effect of re-pressurization. After draining of woods metal by live oil, water injection tests were performed in both experiments using synthetic brine. Water was quickly injected from the bottom of the system at a rate of 5 cm³/min to drain all existent oil in the fracture so that core sample experience counter-current imbibition. Injection rate was then decreased to 1 cm³/min and continued for 1-2 days, to attain ultimate oil recovery. Thereafter equilibrium gas at 210 bar (IFT=0.374 mN/m) was injected at high rate (5 cm³/min) from the top of the system to drain all the water in the fracture very quickly. Injection rate was then decreased to 0.1 cm³/min and process was continued until reaching to the ultimate recovery. The system was re-pressurized to 220 bar (IFT=0.15 mN/m) to study the effect of IFT reduction on oil recovery. In the final stage, non-equilibrium gas; CO₂ for Exp. #2 and C₁ for Exp. #3 were injected to the system.



Figure 2. Production as a function of time (Exp. 1). Secondary gas injection, start with equilibrium gas @ 220 bar and followed by non-equilibrium gas

4. Results and discussion

The first experiment has been performed to investigate the potential of secondary recovery by gas injection. In other words, no water injection has been performed in the test. The oil recovery vs. time for the first experiment is shown in Figure 2. 47% of oil was recovered during equilibrium gas injection part which is very promising. Injection of CO_2 as a non-equilibrium gas also leads to 17.5% additional oil recovery. Additional oil production resulting from non-equilibrium gas injection was significant which may be due to participation of all mechanisms in this process.

The second and third experiments were carried out in order to investigate the efficiency of tertiary gas injection using equilibrium and non-equilibrium gas. It is a common belief that if a formation is not water wet, the matrix will retain oil by capillarity during water flooding ends to low oil recovery. This is the case for Asmari limestone which is confirmed by our previous results on long stack of blocks (Karimaie and Torsæter, 2007).



Figure 3. Production as a function of time (Exp. 2). Tertiary gas injection, start with water injection followed by equilibrium gas, re-pressurization and non-equilibrium gas.



Figure 4. Production as a function of time (Exp. 3). Tertiary gas injection into Asmari limestone sample. C₁ injection at the final stage

Results of the second and third experiments are shown in Figures 3 and 4. Less than 8% of the original oil in place was produced which is in line with previous published results and indicates the extents of weak water wetness of the matrix block. More than 90% of the original oil was left after water injection, resulting in a very high potential for oil recovery by gas injection. Equilibrium gas was then injected at high rate (5 cm³/min) from

top of the system to drain all the water in the fracture very quickly. The rate was then decreased to $0.1 \text{ cm}^3/\text{min}$ while the pressure was constant at 210 bar (IFT=0.37 mN/m). The oil production was started and continued for almost 1.5 days to reach to ultimate recovery. Around 40% and 33% of the original oil in place was produced during this step in experiments 2 and 3 respectively. Afterwards, the system was re-pressurized to 220 bar which in turn cause to reduction of the IFT to 0.15 mN/m. Oil production was then started so that 12% and 17% of the original oil in place was produced during experiments. The equilibrium gas injection continued until reaching to ultimate recovery. Increase of oil recovery as pressure increases could be attributed to two processes, increase of oil formation volume factor (B_o), and a lower residual saturation due to the reduction of gas-oil interfacial tension. However, the contribution of B_o is minor due to small residual saturation and the change in IFT is more important since the recovery curve has a sharp increase. Bardon and Langeron (1980) also found a decreasing residual oil saturation using C₁-C₇ mixtures at high pressures when measuring gas oil relative permeability.

Significant oil recovery efficiency achieved during the two successive stages of gravity drainage is mainly due to the low IFT gravity drainage. The ultimate recovery in both experiments clearly indicates almost the same recovery efficiency under gravity drainage. Based on the results of the experiments, it is confirmed that tertiary gas-oil gravity drainage in can be utilized for higher oil recovery.

The fourth part of the production curve in Figures 3 and 4 portrays the non-equilibrium gas injection stage. To better evaluate the performance of the type of dry gas injection, oil recovery efficiency for the two experiments are shown in Figure 5, which are calculated, based on original oil in place. Compared to the CO_2 injection experiment, lower oil recovery was noticed during C_1 injection. The recovery may be due to one or a combination of different effects: Gravity drainage, swelling of oil and diffusion. However, due to the nature of the apparatus, it was impossible to distinguish these recovery mechanisms. Therefore, swelling test have been performed using a PVT simulator. Results are given in Figures 6 and 7. Figure 6 clearly demonstrate that oil swollen volume due to CO_2 injection is more than for C_1 injection which could be a reason for higher oil recovery by CO_2 . Figure 7 also shows the bubble point pressure variation for CO_2 and C_1 vs added gas mole %. It is clear that bubble point pressure decreases when CO_2 mole% increases and opposite behavior is shown for C_1 . Therefore, it may conclude that system experiences a single-phase region and swelling may play an important role for CO_2 injection, while in case of C_1 injection the system would be in a two-phase region and swelling will be negligible.



Figure 5. Comparison between CO2 and C1 injection in the last stage of the experiment



Figure 6. Swollen volume of the oil by CO₂ and C₁ injection



Figure 7. Bubble point variation of the oil by CO₂ and C₁ injection

5. Uncertainities and sources of errors

It is clear that no physical quantity can be measured with perfect certainty and there are always errors in any measurement. In our experiment core diameter varied from 3.7 cm to 3.8 cm along the core height. Core diameter variation caused uncertainty in core and fracture pore volume, justify that matrix and fracture porosity as uncertainties in the experiments. However, in order to reduce these uncertainties, matrix pore volume was measured by the amount of fluid injected to the core sample, i.e. saturation method. The fracture pore volume was also measured by two methods: mathematical calculation based on inner diameter of the tube and outer diameter of the core sample (53 cm³ for sample 1 and 55 cm³ for samples 2 and 3) and by weighting the amount of woods metal before and after each experiment. The calculated value and measured value had about 10% difference which may be due to variation of core diameter along the core height and/or due to not draining all alloy from the fracture. However, based on weight difference on the amount of woods metal used to seal the fracture and the one drained out of the fracture space, we found that about 15% of the woods metal remained in the space between the core and steel tube. Reduction of fracture pore volume may have effect on fracture permeability, which in turn may change the recovery mechanism from gravity-dominated flow to viscous-dominated flow.

6. Conclusions

The main objective of this research was to gain more knowledge on recovery efficiency of water and gas injection in fractured reservoirs in weakly-water wet state using a series of experiments. Numerous experimental studies of water injection have been presented in the literature which in most of them water-wet samples were examined under atmospheric condition. Similar detailed laboratory experiments have not been reported for oil-wet or weakly water-wet system at reservoir condition. In this research, water injection tests have been performed in fractured porous media at reservoir condition prior to gas injection and less than 10% recovery was achieved. The recovery of trapped oil in matrix block after water injection was targeted by employing equilibrium and non-equilibrium gas injection and the present study addresses the efficiency of oil recovery in water flooded reservoirs with particular emphasis on low IFT equilibrium gas, re-pressurization and non-equilibrium gas injection. Based on the results, injection of equilibrium gas at low interfacial tension leads to significant oil recovery. Experiments also clearly shows that gas injection could be an efficient method in Asmari reservoirs and water injection is rather inefficient.

Generally when injected gas is not miscible, gas invasion into the pores is opposed by the threshold capillary pressure results in trapped oil in the reservoir. The amount of such trapping can be reduced by increasing the reservoir pressure which in turn decreases interfacial tension. The hypothesis was verified in this study by re-pressurization during gas injection, the results show a favorable effect on oil production due to reduction of interfacial tension. Experiments also reveal that injection of non-equilibrium gas, in secondary and tertiary cases leads to additional oil recovery. However, efficiency of the process is strongly dependent on the type of gas. When a lean gas such as methane is injected, ultimate recovery is lower than CO2 injection.

Acknowledgment

We acknowledge the financial support provided by NIOC and Statoil. All PVT experiments in this paper were performed at SINTEF petroleum research. We gratefully acknowledge the help of all laboratory personnel. Some part of the paper was presented at the SPE International Conference on CO2 Capture, and Utilization, USA, 2010 (SPE 139703). Thanks to SPE for permission to publish the paper.

Nomenclature

 $B_o = Oil$ formation volume factor, rv/stc v

EOR = Enhanced Oil Recovery

EOS = Equation of state

IFT = Interfacial tension, mN/m

 P_b = Bubble point pressure, bar

OOIP = Original oil in place

References

- Bardon, C., & Langeron, D. G. (1980). Influence of Very Low Interfacial Tensions on Relative Permeability. Paper SPERE October, 391-401. http://dx.doi.org/10.2118/7609-PA
- Darvish, G. R, Lindeberg, E., Holt, T., & Utne, S. A. (2006). Reservoir Conditions Laboratory Experiments of CO₂ Injection Into a Fractured Core. Paper SPE 99649 presented at the 2006 SPE/DOE Symposium on Improved Oil Recovery in Tulsa, Oklahoma, U.S.A., 22-26. http://dx.doi.org/10.2118/99649-MS
- Holm, L. W., & Josendal, V. A. (1974). Mechanism of Oil Displacement by Carbon Dioxide. J. Pet. Tech. Dec.1427. http://dx.doi.org/10.2118/4736-PA
- Hujun, L., Putra, E., Schechter, D., & Grigg, R. (2000). Experimental Investigation of CO₂ Gravity Drainage in a Fractured System. Paper SPE 64510 presented at the SPE Asia Pacific Oil and Gas Conference and Exhibition held in Brisbane, Australia, 16-18 October 2000.
- Karimaie, H. and Torsæter, O. (2010). Low IFT gas-oil gravity drainage in fractured carbonate porous media. Journal of Petroleum Science and Engineering (JPSE), 70(2010) 67-73.
- Karimaie, H., & Torsæter, O. (2007). Effect of Injection Rate, Initial Water Saturation and Gravity on Water Injection in Slightly Water-Wet Fractured Porous Media. *Journal of Petroleum Science and Engineering* (JPSE), 58, 293-308.
- Karimaie, H., Darvish, G.R., Lindeberg, E., and Torsæter, O. (2008). Experimental Investigation of Secondary and Tertiary Gas Injection in Fractured Carbonate Media. *Journal of Petroleum Science and Engineering*

(JPSE), 62(1-2), 45-51.

- Le Romancer, J. F. X., Defives, D. F., & Fernandes, G. (1994). *Mechansim of Oil Recovery by Gas Diffusion in Fractured Reservoir in Presence of Water.* Paper SPE 27746 presented at the SPE/DOE ninth Symposium on Improved Oil Recovery held in Tulsa, U.S.A., 17-20 April. http://dx.doi.org/10.2118/27746-MS
- Morel, D. D., Bourbiaux, B., Latil, M., & Thiebot, B. (1990). *Diffusion effects in gas flooded light oil fractured reservoirs*. Paper SPE 20516, presented at the 65th SPE fall meeting, New Orleans, September. http://dx.doi.org/10.2118/20516-PA
- Øyno, L., Uleberg, K., & Whitson, C. H. (1995). Dry Gas Injection in Fractured Chalk Reservoirs- an Experimental Approach, Paper SCA 1995-27 presented at the International Symposium of the Society of Core Analysts held in San Francisco, California.
- Saidi, A. M. (1987). Reservoir Engineering of Fractured Reservoirs. Total Edition Press.
- Saidi, A. M. (1996). *Twenty years of Gas Injection History into Well-Fractured Haft Kel Field (Iran)*. Presented at the International Conference & Exhibition of Mexico held in Villhemosa, SPE 35309. http://dx.doi.org/10.2118/35309-MS
- Thomas, L. K., Dixon, T. N., & Pierson, R.G. (1991). *Ekofisk Nitrogen Injection*. SPE Formation Evaluation, June, 151-160. http://dx.doi.org/10.2118/19839-PA

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