Tertiary Liquid and Supercritical CO_2 Injection in Chalk and Limestone at Strongly Water-Wet and Near Neutral-Wet conditions

Eide, Ø., Haugen, Å., Svenningsen, S., Hoff, K. Ersland, G., Fernø, M., Graue, A.
Department of Physics and Technology, University of Bergen

This paper was prepared for presentation at the International Symposium of the Society of Core Analysts held in Aberdeen, Scotland, UK, 27-30 August, 2012

Abstract

Tertiary liquid and supercritical CO_2 injections were performed for enhanced oil recovery (EOR) in carbonate rocks with different pore structure and wettability conditions ranging from strongly water-wet to moderately oil-wet. Differences in the amount of CO_2 injected to reach ultimate recovery was observed between rock types, where limestone exhibited faster production compared with chalk; however, the ultimate recovery was lower for limestone. This demonstrated that heterogeneity with respect to permeability and pore topology plays a major role during CO_2 EOR efforts, as the CO_2 channels through, leaving large areas unswept for the more heterogeneous cores. No clear trend was made indicating that the rock wettability had an effect on oil recovery, but slightly oil-wet conditions seemed to be the more favorable wetting state for the more heterogenous limestone.

Introduction

CO_2 injection for enhanced oil recovery has previously been used in several on-shore carbonate fields with moderate success, Hustad and Austell [8]. The topic of CO_2 injection into larger off-shore fields has recently become a topic of interest in the North Sea, both due to its ability to recover additional oil from giants like the Ekofisk field, but also as a prospect for climate change mitigation for safe and remote storage of CO_2 from European power plants and other industrial facilities.

CO_2 has several EOR-advantages compared to other solvents like flue gas, hydrocarbon gas and nitrogen gas. Compared to hydrocarbon gas injection CO_2 has a potential to extract heavier components up to C_{30} from the reservoir oil. The minimum miscibility pressure for CO_2-oil systems is usually significantly lower than for hydrocarbon gas, flue gas or nitrogen. However, there are several challenges associated with the use of CO_2, the main challenge being its corroding effect on equipment and piping as it dissolves in water and creates carbonic acid. Supplying sufficient amounts of CO_2 to the field at competitive prices is also a major concern and has halted many promising CO_2 projects. In 2000 Jensen *et al* [9] conducted a screening of EOR methods for the Ekofisk field. He concluded that CO_2 WAG (water-alternating-gas) was one of the most promising EOR techniques with a potential of 5.6% incremental oil recovery from the oil originally in place (OOIP). Challenges related to significant dissolution/compaction concerns were identified, but until significant volumes of CO_2 were accessible at low cost, the project was discarded from further consideration.

Macroscopic sweep efficiency is a major challenge due to high mobility of the injected CO_2 phase. In particular heterogeneous/fractured reservoir systems will suffer with respect to sweep. The common method to reduce mobility and improve the sweep efficiency is the water-alternating-gas method, where alternating slugs of CO_2 and water are injected. CO_2 improves the microscopic displacement efficiency and water the macroscopic sweep efficiency. Research and field applications have repeatedly shown the inadequacy of the WAG process, but owing to a lack of a viable alternative the method is widely used, Kulkarni and Rao [10].

The objective of this work is to investigate the effect of wettability, temperature (liquid or supercritical phase state) and heterogeneity on final oil recovery in chalk and limestone core plugs. The results are a part of an ongoing study with the overall objective to determine the flow and oil recovery mechanisms by use of CO_2 in fractured reservoirs.

Experimental Procedures

Core Material

Two different carbonate outcrop rocks were used in this study. The fairly homogeneous Rørdal chalk which consists mainly of coccolith deposits, with a composition of calcite (99%) and some quartz (1%). The permeability and porosity ranged from 2-5 mD and 43-47% respectively. More information can be found in Ekdale and Bromley [3], Hjuler [7], Odling *et al* [13] and Strand *et al* [15].The second rock type was the more heterogeneous Edwards limestone with trimodal pore sizes, vugs and microporosity, identified by Johannesen *et al* [11] and Riskedal *et al* [14]. The permeability and porosity ranged from 6-33 mD and 19-26%, respectively.

Core Preparation and Assembly

The cores were cut from larger slabs of rock and dried at 80 °C for several days before standard porosity and permeability measurements. Irreducible water saturation was established by a bi-directional flood with oil under constant injection pressure using the same differential pressure for all core plugs of same rock type. S_{wi} for samples CHR 1 and 2 was considered too high, thus all subsequent samples were established using a higher applied pressure. Several core samples were aged to less water-wet, near neutral-wet and slightly oil-wet wettability conditions. The core samples were aged by exposing the rock surface to a North Sea crude oil at elevated temperature after irreducible water saturation was established. Oil was then continuously injected into the core to expose the core sample to fresh oil over an extended period of time (1-6 days). The flooding direction was reversed after half the aging time to create a uniform wetting distribution. Further details can be found in Graue *et al* [6], Aspenes *et al* [1] and Fernø [5]. The rock properties are shown in Table 1, where the Rørdal chalk samples are denoted CHR_# and the Edwards limestone samples are denoted EDW_#. Wettability was measured using the Amott-Harvey method. Aged Rørdal chalk samples exhibited less water-wet characteristics, with little or no spontaneous water imbibition and no spontaneous oil imbibition. Aged Edwards limestone core samples exhibited slightly oil-wet conditions, with spontaneous imbibition of oil and no spontaneous imbibition of water. Wettability was not measured on untreated rock samples, as they have previously been shown to be strongly water-wet, Johannesen [10]. Before being mounted in the biaxial Hassler type core holder the core plugs and end pieces were wrapped in aluminum foil to prevent the CO_2 from corroding and dispersing through the rubber sleeve. The core holder was mounted horizontally during all experiments. With the small height and density difference in the experiments and the strong capillary forces, the effects of gravity segregation should be negligible.

Waterflooding and CO_2 injection

The cores were waterflooded to S_{orw} before CO_2 was injected in tertiary mode. Experiments were performed with both liquid and supercritical CO_2, and decane was used as the oil phase. At supercritical conditions the core holder was mounted inside a heating cabinet at 46 °C, i.e. above the critical temperature for CO_2 (31.1 °C). Liquid CO_2 was injected at ambient temperature (21 °C). In both types of experiments the line pressure was 82 bars. First contact miscibility between CO_2 and decane occurs at 80 bars and 37.8 °C, Ayirala *et al* [2]. At 20 °C the MMP is approximately 54 bar, whereas at 46 °C the MMP is approximately 95 bars. The experimental setup is shown in Figure 1. The volumetric injection rates used is shown in Table 1, where decrease in CO_2 density at elevated temperature has been accounted for. A temperature increase from 20 °C – 46 °C at 82 bars leads to a volumetric expansion by a factor of 3.1. Note that the front velocity was lowered by the increased diameter of the core samples used at elevated temperature (2” vs. 1.5” at ambient temperature), so the front velocity during supercritical CO_2 injection was only somewhat higher than during liquid CO_2 injection.

Fluid Properties

To alter the core wettability a North Sea crude oil was used. To avoid asphaltene precipitation and to stop the aging process, the crude oil was replaced with decahydronaphthalene which in turn was displaced by n-decane, which was used as the oil phase. At liquid CO_2 conditions the CO_2 and oil phase have almost identical density (0.83 and 0.74 respectively), while at supercritical CO_2 conditions the CO_2 is 3.1 times less dense than the oil phase (0.24 and 0.72, respectively).

Results

In all graphs the results are shown as oil recovery versus time. All results are summarized in Table 2.

Rørdal chalk

Figure 2 shows the recovery factor during waterfloods in eight Rørdal chalk cores. Waterfloods performed at ambient temperature are indicated with squares, whereas elevated temperature is indicated with crosses. Core samples with altered wettability have dotted lines, whereas strongly water-wet cores have solid lines. All cores at strongly water-wet conditions (core plugs CHR_1-5 and CHR_7) demonstrated a clean breakthrough of water with no two-phase production. Less than 0.5 PVs of water was required to reach end point oil saturation in these core plugs. Waterfloods at less water-wet conditions (core plugs CHR_6 and CHR_8) demonstrated a transient period with simultaneous water and oil production. In these core plugs between 1.3 and 2.25 PVs of water was required to reach end-point oil saturation. The final oil recovery after waterfloods ranged between 30-55% OOIP, leaving a significant target for EOR efforts.

Figure 3 shows the development in oil recovery during eight CO_2 injections in Rørdal chalk cores. Liquid CO_2 injections are indicated with squares, whereas supercritical CO_2 injections are indicated with crosses. Core samples with altered wettability have dotted lines, whereas strongly water-wet cores have solid lines. The injected CO_2 recovers additional oil after the waterfloods, with total recovery factor ranging between 62-77% OOIP. During supercritical CO_2 injections core plugs CHR_5-8 reached their end point oil saturation after 1.5-3.2 PVs injected. Final oil recovery for these plugs ranged between 62-73% OOIP. In contrast, at liquid CO_2 conditions, core plugs CHR_1-4 did not reach their end point saturation during the injections. After an initial period of rapid oil recovery, the rate of oil production slowed down, but oil was still mobilized after 4-14 PVs injected. Final oil recovery (when experiments were terminated) in these plugs ranged between 67-77% OOIP.

Figure 4 shows development in oil recovery during eight waterfloods in Edwards limestone core plugs. Waterfloods performed at ambient temperature is indicated with squares, whereas elevated temperature is indicated with crosses. Core samples with altered wettability have dotted lines, whereas strongly water-wet cores have solid lines. The waterfloods display the same wetting characteristics as in the Rørdal chalk, with a clean water breakthrough in the strongly water-wet cores, and two-phase production of oil and water in the slightly oil-wet cores. The oil recovery after the waterflood ranged between 13-50% OOIP.

Figure 5 shows development in oil recovery during eight CO_2 injections in Edwards limestone core samples. Liquid CO_2 injections are indicated with squares, whereas supercritical CO_2 injections are indicated with crosses. Core samples with altered wettability have dotted lines, whereas strongly water-wet cores have solid lines. The final oil recovery after the CO_2 injection ranged between 29-73% OOIP. Unlike the Rørdal chalk, most samples reached their end point oil saturation before 4 PV had been injected. After supercritical CO_2 injection, the core plugs generally had lower oil saturation than the experiments with liquid CO_2 injection. After liquid CO_2 injection the final oil recovery ranged between 29-44% OOIP, and after supercritical CO_2 injection the final oil recovery ranged between 53-73% OOIP. During liquid CO_2 injection there was a clean breakthrough with no transient production, unlike during supercritical CO_2 injection where there was a period of transient production.

Discussion

Due to space limitations, the authors have chosen to focus on CO2 work, and the waterfloods have been omitted from the discussion.

Effect of heterogeneity

During the CO2 flood there was a large difference in behavior between the chalk and the limestone: the average end point oil saturation for chalk was 0.22, compared to 0.34 for the limestone samples. Also, the chalk samples did not appear to reach their endpoint saturation, but had continued transient production until the experiments were terminated above the MMP. On the other hand, in most of the limestone samples the oil was recovered quickly with short transient production, but with low overall recovery from CO2 injection. This indicates poor sweep efficiency where the highly mobile CO2 channels through the large pores. The large permeability contrast in the Edwards limestone could explain why the CO2 breaks through after only a short time and leaves large areas untouched by CO2. Poor core sweep using CO2 may also be explained by the discontinuous nature of oil after waterfloods, making it harder for the CO2 to mobilize the oil. This effect was not as visible in the chalk samples, an explanation could be the small spread in pore size, and small pores, in the chalk samples compared with the limestone samples. The small spread in pore size means that the CO2, which is non-wetting to the oil, is less likely to finger through as there is no path of large pores. The small pores mean that the water films are thinner and the CO2 can more quickly diffuse through and contact the oil. It was also observed that most of the water which was produced during CO2 injection was produced shortly after injection start. This initial water production was followed by an oil bank, after which the water production was very low. This was most likely due to the low solubility of CO2 in water, compared with oil. This is illustrated in by Brautaset et al. [3], where it is visible that areas that were poorly swept during waterflooding was the first areas to be swept by oil due to the affinity CO2 has for oil.

The large heterogeneity contrast in the Edwards limestone has proven to be a problem when trying to conclude regarding other parameters as initial saturations or wettability. One remedy for this is to use the same core for several experiments by cleaning it.

<u>Effect of temperature</u>

Final oil recoveries after CO_2 injections in chalk demonstrated difference between liquid and supercritical CO_2 phase. While oil production from supercritical CO_2 injection in core plugs CHR_6-8 ceased after 2-3 PVs were injected, the oil production from liquid CO_2 injected did not cease, even after 3-16 PVs injected for core plugs CHR_1-4. This difference may be explained by supercritical injections being below the MMP. Experiments conducted at liquid conditions the CO_2 and oil were first contact miscible. This may explain why the oil production from liquid CO_2 injection did not cease, oil was continuously being contacted by CO_2 that could diffuse through the water and mix with the oil. The solubility of CO_2 in water is quite small, and will further decrease when increasing the temperature from 20 to 46 degrees, meaning that this effect will be more pronounced at lower temperature. However the decrease in solubility is relatively small. This means that very little CO_2 will dissolve in the water phase.

The experiments demonstrate the necessity of identifying the MMP accurately. In experiments performed just above the MMP in chalk the average oil recovery was 74% OOIP, versus 67% OOIP just below the MMP. This does not take into account the difference in mobility ratio when increasing the temperature, but that effect will be minor as both the CO_2 and decane have reduced viscosity at elevated temperature. That effect is also countered by reduced interfacial tension at elevated temperature, which will improve the microscopic sweep efficiency. In both the CO_2 injections above and below MMP, the pressure was only a few bars from MMP.

In contrast to Rørdal chalk, supercritical CO_2 injections produced more oil compared to liquid CO_2 injection in Edwards limestone. The development during liquid CO_2 injection behaved like a miscible flood with high continuity in the oil phase, as oil was rapidly mobilized and there was a short transient period. However the final recovery is very low compared to what could be expected from a miscible displacement. One possible explanation is that the high initial water saturation isolated much of the oil from the injected CO_2. The experiments conducted at supercritical conditions behaved like drainage by an immiscible fluid with transient production of both oil and CO_2. The final oil recovery from these experiments was higher than during liquid CO_2 injection. One explanation may be that this was caused by slightly different injection rate. During the supercritical CO_2 injection the velocity of the advancing CO_2 front was slightly higher than during liquid CO_2 injection. However, there is a wide spread in the data from the limestone core samples. The very heterogeneous nature of the Edwards limestone makes it very difficult to suggest the mechanisms that cause the difference in oil recovery between the two CO_2 phase states.

Effect of wettability

No impact of wettability on final recovery or production profile was observed during CO_2 injections in chalk. However, lower residual oil saturation was observed in the oil-wet limestone samples EDW_6 and EDW_8 compared with the water-wet limestone core plugs EDW_4-5 (all performed at supercritical CO_2 conditions). The oil production also takes place within a shorter time frame than the water-wet core plugs. A possible explanation is the larger surface area and the more continuous residual oil saturation at slightly oil-wet conditions compared to water-wet conditions, allowing the CO_2 to displace and diffuse the oil throughout the core more efficiently. In the water-wet samples the oil will be more discontinuous, meaning that the CO_2 will have to diffuse through the water to contact the oil, which is a slower process. However, based on the large spread in the data set for Edwards limestone due to heterogeneities these arguments are hampered with uncertainty, as well as the fact that these plugs are only slightly oil-wet. EDW_7 is an exception from EDW_4 and 5, as it is strongly water-wet, yet has the lowest residual oil saturation of all core plugs. One possible explanation for this could be the high initial water saturation (0.40) coupled with an effective waterflood. After the waterflood the remaining oil will primarily be located in the center of the large pores with very little residual oil the smaller pores due to the inefficient primary drainage. Because CO_2 is the non-wetting phase it will travel in the middle of the large pores displacing the oil present, resulting in a high recovery factor.

Conclusions

- CO_2 injection enhanced oil recovery in all experiments
- Lower residual oil saturation was achieved during CO_2 injection in homogeneous chalk compared with more heterogeneous limestone, the higher residual saturation in the heterogeneous limestone is attributed to channeling and loss of sweep efficiency.
- Oil continued to be produced from chalk samples until the experiments were terminated when injecting above the MMP, unlike the limestone samples where the additional oil was recovered within a short period of time, with very little transient production of oil and CO_2 ,indicating that heterogeneity has a large impact on CO_2 floods.
- Slightly oil-wet conditions appeared to be the most favorable wetting condition during tertiary CO_2 injection, however large spread in the data set from Edwards limestone due to core heterogeneities makes this generalization uncertain.
- The large heterogeneity contrast in the Edwards limestone has proven to be a problem when trying to conclude regarding parameters such as initial saturations or wettability. A remedy for this is to use the same core for several experiments by cleaning it.
- A large number of experiments are needed to provide statistics, especially when working with heterogeneous materials where it is hard to control parameters.

Nomenclature

PV = Pore Volumes
MMP = Minimum Miscibility Pressure

Acknowledgements

Four of the authors are indebted to The Norwegian Research Council for financial support from the Petromaks and Climit programs. The authors would like to thank Jim Stevens and James Howard at ConocoPhillips BTC for their assistance with the MRI experiments. ConocoPhillips, DONG Energy and BP are greatly acknowledged for financial support.

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Tables and Figures

Table 1. Rock properties

Core name	Injection after water	I_{A-H}	Length [cm]	Diameter [cm]	Porosity [%]	Permeability [mD]	Pore volume	Inj. Rate [ml/h]
CHR_1	Liq. CO_2	1 a)	6.0	3.8	47	5.7	32.7	2.0
CHR_2	Liq. CO_2	1 a)	6.0	3.8	47	6.2	32.1	2.0
CHR_3	Liq. CO_2	1 a)	6.0	3.8	46	5.0	31.8	2.0
CHR_4	Liq. CO_2	1 a)	6.0	3.8	46	4.6	31.8	2.0
CHR_5	SC. CO_2	1 a)	8.1	5.1	43	2.5	71.4	2/6.2 b)
CHR_6	SC. CO_2	0.4	6.0	5.1	43	2.7	52.5	2/6.2 b)
CHR_7	SC. CO_2	1 a)	6.0	4.9	45	4.6	50.6	2/6.2 b)
CHR_8	SC. CO_2	0.2	6.0	5.0	44	2.4	51.0	2/6.2 b)
EDW_1	Liq. CO_2	1 a)	5.9	3.8	26	15.2	17.2	2.0
EDW_2	Liq. CO_2	1 a)	6.4	3.8	23	9.3	16.5	2.0
EDW_3	Liq. CO_2	1 a)	7.2	3.8	21	9.5	17.6	2.0
EDW_4	SC. CO_2	1 a)	6.1	4.9	26	31.1	29.7	2/6.2 b)
EDW_5	SC. CO_2	1 a)	6.4	4.9	24	27.7	29.4	2/6.2 b)
EDW_6	SC. CO_2	-0.1	6.1	4.9	22	15.9	25.9	2/6.2 b)
EDW_7	SC. CO_2	1 a)	7.5	4.9	24	16.7	33.4	4.5/14.0 b)
EDW_8	SC. CO_2	-0.3	7.0	3.8	20	6.3	15.5	3/9.3 b)

a) Not explicitly measured, measured on sister plugs
b) Corrected for density difference at elevated temperature, shown as volumetric water rate/CO_2 rate

Table 2: Summary of experimental results

Core name	CO_2 state	Amott-Harvey index	S_{wi}	$S_{or,w}$	$S_{or,CO2}$	Recovery waterflood [% OOIP]	Recovery CO_2-flood [% OOIP]	Recovery at water breakthrough [% OOIP]	Recovery at CO_2 breakthrough [% OOIP]
CHR_1	Liq.	1 a)	0.37	0.43	0.15	32	76	32	46
CHR_2	Liq.	1 a)	0.32	0.48	0.23	30	67	27	39
CHR_3	Liq.	1 a)	0.27	0.46	0.17	37	77	37	45
CHR_4	Liq.	1 a)	0.29	0.41	0.19	47	75	47	55
CHR_5	SC.	1 a)	0.22	0.43	0.28	45	64	45	52
CHR_6	SC.	0.4	0.20	0.34	0.28	55	71	41	68

CHR_7	SC.	1 [a)]	0.21	0.38	0.22	53	73	53	70
CHR_8	SC.	0.2	0.24	0.43	0.22	43	62	29	58
EDW_1	Liq.	1 [a)]	0.27	0.52	0.41	28	44	28	36
EDW_2	Liq.	1 [a)]	0.30	0.55	0.45	22	35	22	30
EDW_3	Liq.	1 [a)]	0.35	0.57	0.47	13	29	13	26
EDW_4	SC.	1 [a)]	0.14	0.66	0.40	24	53	23	39
EDW_5	SC.	1 [a)]	0.27	0.49	0.31	33	58	33	42
EDW_6	SC.	-0.1	0.23	0.42	0.25	45	68	43	58
EDW_7	SC.	1 [a)]	0.40	0.30	0.16	50	73	48	70
EDW_8	SC.	-0.3	0.29	0.51	0.26	27	64	27	45

a) Not explicitly measured, measured on sister plugs

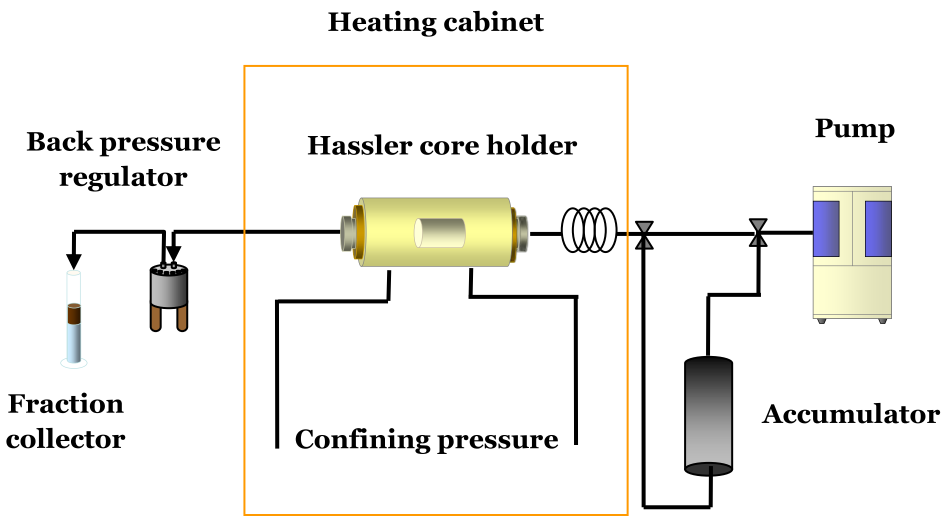

Figure 1. Experimental setup

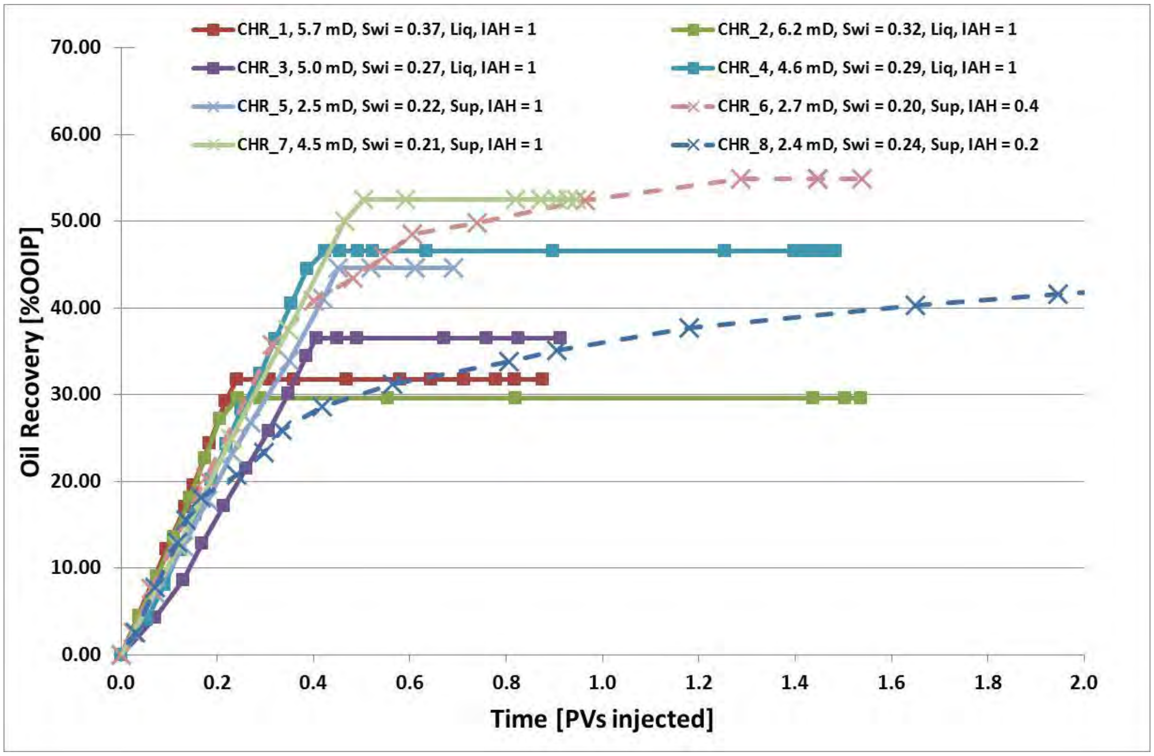

Figure 2. Oil recovery in % OOIP versus pore volumes injected during waterflood in 8 Rørdal chalk cores. Note the x-axis has been cropped at 2 PVs injected, final recovery CHR_8 = 43 % OOIP

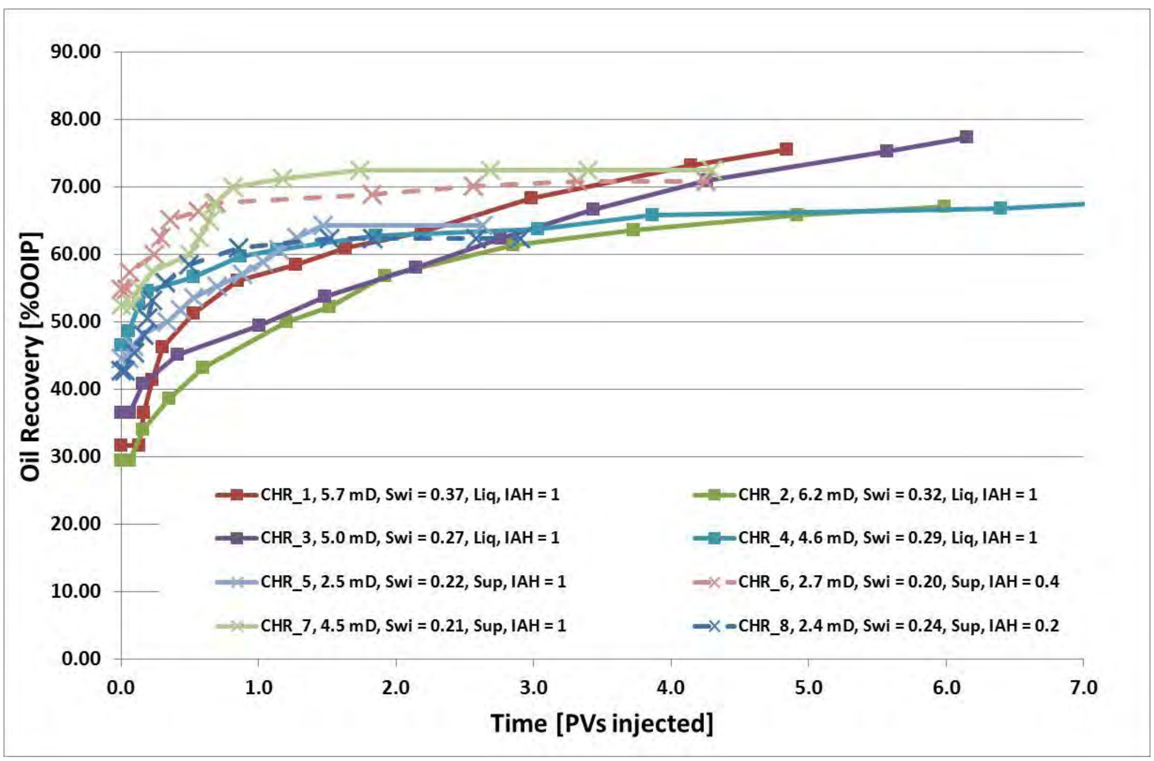

Figure 3. Oil recovery in % of OOIP versus pore volumes injected during CO_2-flood in 8 Rørdal chalk cores. Note the x-axis has been cropped at 7 PVs injected, final recovery CHR_4 = 75% OOIP.

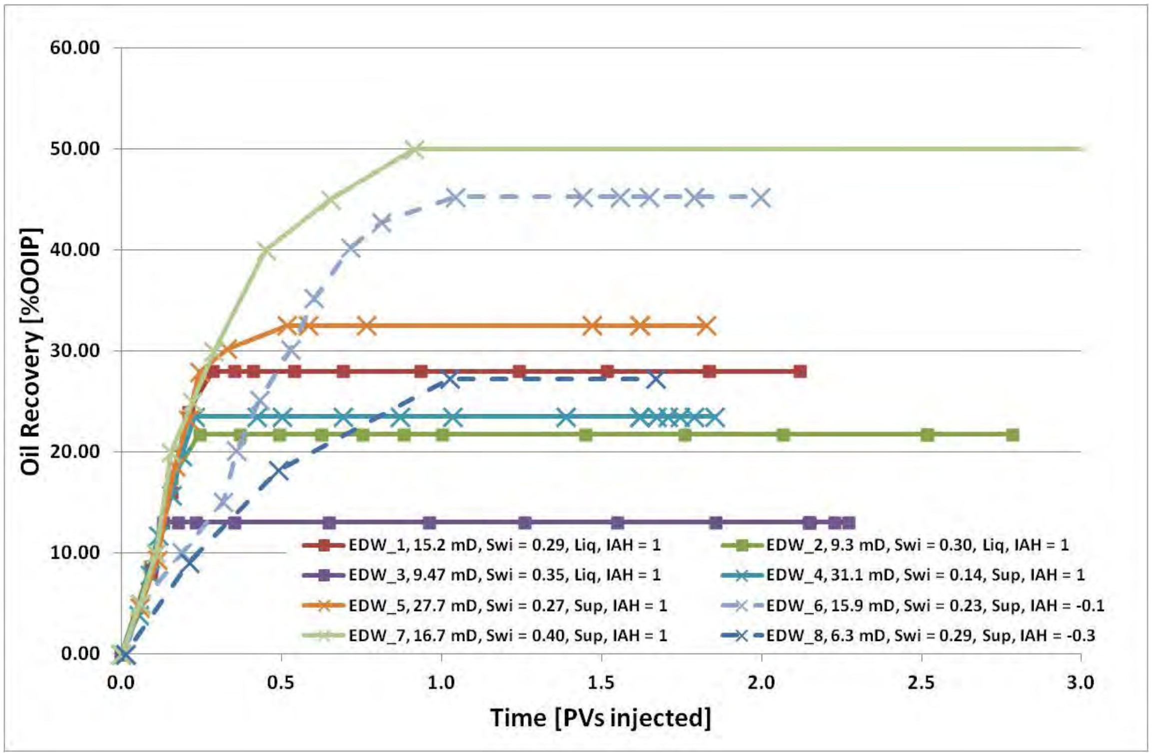

Figure 4. Oil recovery in % of OOIP versus pore volumes injected during waterflooding in 8 Edwards limestone core samples. Note the x-axis extends to 3 PVs injected.

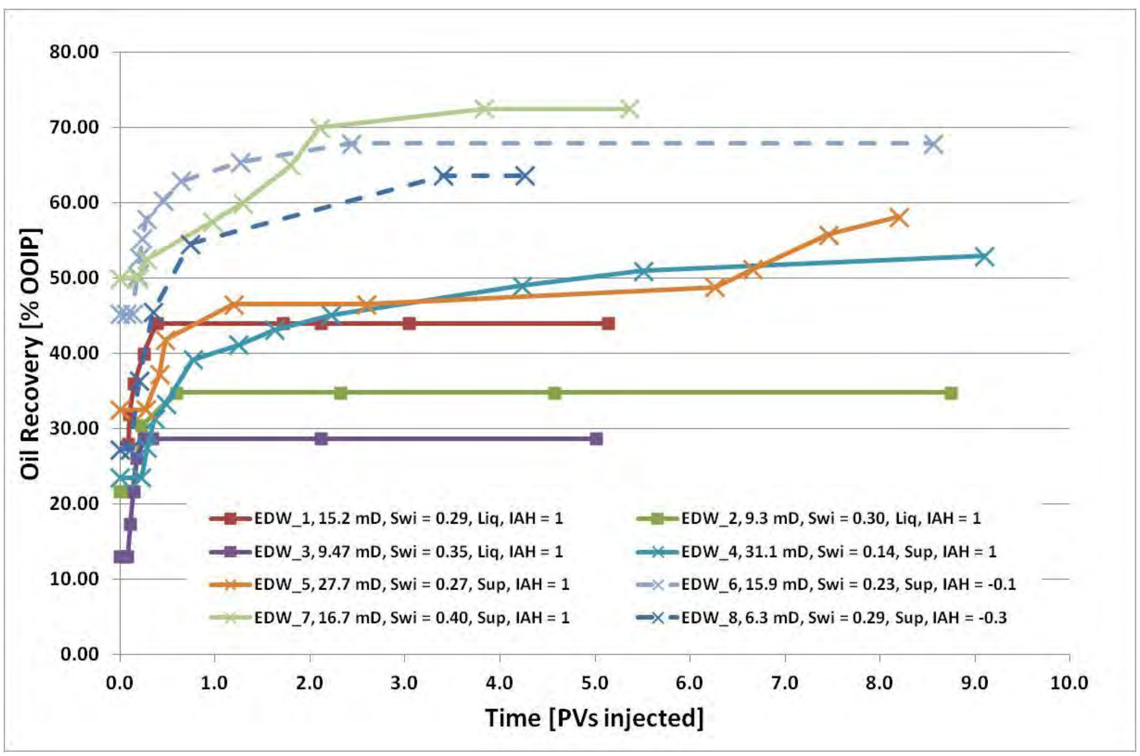

Figure 5. Oil recovery in % of OOIP versus pore volumes injected during CO_2-flood in 8 Edwards limestone cores. Note the x-axis extends to 10 PVs injected.