

LOW SALINITY OIL RECOVERY ON CLAYEY SANDSTONE: EXPERIMENTAL STUDY

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ABSTRACT

It's now largely agreed from laboratory and field tests results that brine composition and ionic strength can have influence on oil recovery [Jadhunandan and Morrow 1991, 1995]. Morrow et al. in the 90's first carried out a large study on the ability of low salinity waterflooding to become an EOR method [Yildiz et al. 1996; Tang and Morrow 1999]. More recently BP, carried out a large program on low salinity injection and proposed additional recovery mechanisms [Mc Guire et al. 2005; Lager et al. 2006]. Although there are evidences of this phenomenon, the physical causes are still uncertain and several possible recovery mechanisms have been put forward in the literature: clay release, wettability alteration, IFT modification, key role of kaolinite etc.

In this paper, we present experiments dealing with tertiary and secondary recovery tests performed on an outcrop sandstone containing 9.2% of clays without kaolinite. Each experiment is performed following a three steps procedure during which pressure drop, pH, brine composition and oil recovery are monitored. First, the core is saturated with a high salinity brine at ambient temperature until equilibrium is reached [pressure drop and pH are constant and ion exchange is achieved]. Second, the initial water saturation is established using a crude oil. Finally, after ageing, high salinity brine waterflooding followed by low salinity brines in the case of tertiary experiment or direct low salinity brines flooding in the case of secondary recovery are performed. Additional oil recovery of about 10% of original oil in place is observed in tertiary experiments when going from 50g/l to 1g/l brine. This oil production occurs after 2 or 3 pore volumes in the form of an oil bank displacement suggesting a more dispersive behavior of the matrix in the presence of oil. It is worth noting that this additional oil recovery has been obtained in a clayey sandstone that does not contain kaolinite. When low salinity brine was injected, both pH and pressure drop increased. Successive dilutions of injection brine from high salinity did not lead to an oil recovery increase until using a 1g/l, showing a salinity concentration threshold, whereas pressure drop and pH were increasing significantly at each salinity dilution. Waterflooding at different temperatures shows that oil recovery in secondary mode increases in increasing displacement temperature. But response of additional oil recovery in tertiary mode is more positive for moderate temperatures.

INTRODUCTION

Although several experiments demonstrate additional oil recovery by low salinity brine injection [Tang and Morrow 1997, 1999], the underlying mechanisms are not precisely identified as of yet. Some progress has been made toward understanding the physical phenomena leading to this additional recovery. The variability of literature results suggests that these recoveries depend on complex crude oil/brine/rock interactions. In the last 10 years, some mechanisms have been put forward to explain the additional oil recovery, but contradictory results exist for each scenario.

Morrow et al. [1999] proposed that additional oil recovery is the consequence of clay/clay interaction weakening in the porous medium [especially kaolinite] when low salinity brine is injected. They consider that the expansion of clay layers leads, in some conditions, to detachment from the rock surface of mixed-wet clay particles that are able to transport adsorbed oil droplets. This mechanism suggests a permeability reduction due to pore constrictions and/or fines production and evolution to a more water wet system. Permeability reduction is in fact, not systematic when additional oil recovery is observed and evolution to more water wetness has to be confirmed.

Lager et al. [2006] considered a mechanism based on Multicomponent Ionic Exchange [MIE] between mineral surface and invading brine. The substitution of compensator cations of the clay initially complexed with polar components of oil by invading cations leads to the removal of these oil components from the clay surface and their replacement by uncomplexed cations. This mechanism suggests that oil production is more efficient when divalent ions are present in the low salinity brine (but in a lower concentration).

Another mechanism was discussed by McGuire et al. [2005], considering similitude with alkaline flooding and based on pH increase in the porous medium. He suggested that when pH becomes basic, the interfacial tension between crude oil and brine decreases due to in-situ generation of surfactants in the matrix. The IFT reduction leads to reservoir more water wetness and improvement of oil recovery. Each mechanism fits some experiments but counter-examples exist for each of them.

Our study was performed on a clayey outcrop sandstone, without kaolinite. It was aimed at confirming the additional oil recovery in secondary and tertiary mode by low salinity injection, and verifying the main assumptions proposed so far. In order to validate the influence of the more relevant physical parameters and study crude oil/ brine/ rock interactions, pH, permeability, ionic composition of effluents data were monitored during one and two-phase flow experiments.

EXPERIMENTS

We describe in this part one-phase and two-phase experiments corresponding to dispersion, secondary and tertiary waterflooding.

Material

Porous medium

Consolidated core samples coming from the same outcrop and referred as DU3 were used for the experiments. The samples were 7cm long and 4cm diameter. The gas permeability ranged between 398 and 825 mD and porosity, calculated by mass difference, was between 16.4 and 21.5%. Mineralogy measurements showed that this sandstone contained 9.2% of clays including 3.2% of chlorite, 4% of muscovite, 2% of illite and interstratified clays. The sandstone did not contain kaolinite. Table 1 and Table 2 provide the mineral and molecular composition of DU3/A and DU3/0 cores drilled from A and 0 blocks respectively. Table 4 summarizes physical properties of all samples used in this study.

Brines

A synthetic high salinity brine [50g/l], referred as brine #H was used for all the samples as the connate brine. This brine was composed of 90%_w NaCl and 10%_w CaCl₂. Brine #1 [1g/l with 95%_w NaCl and 5%_w CaCl₂] was the most used low salinity brine. Brine #10l, brine #5l, brine #2.5l and brine #0.1l had salt concentrations which were 10, 5, 2.5 and 0.1 times respectively the concentration of brine #1. Two other low salinity brines

were also used as invading brines: Brine #b [1g/l only NaCl], and brine #m [1g/l with 95%_w NaCl, 3.75%_w MgCl₂, and 1.25%_w CaCl₂] [Table 3].

Crude oil

The same crude oil, designated as Oil A, was used for all the tests. Its viscosity and density were 5.42 cP and 835.4 kg/m³ at T = 35°C respectively. The Total Acid Number [TAN] and Total Basic Number [TBN] were 0.17 and 0.95 mg KOH/g of oil respectively. This oil contained 2.3%_w of pentane asphaltenes. The SARA analyses give a crude oil composed of 58,2% of saturated, 31,4% of aromatic and 10,4% of polar components.

Experimental setups:

One phase flow

The experimental setup, presented in Figure 1, was composed of a volumetric pump connected to the core. A back pressure gauge was used to maintain a line pressure of 4 bar. Pressure drop, pH and density were measured on line while ionic concentration was determined from the effluents collection. The coreholder could be placed in an oven for high temperature experiments.

Two phase, secondary and tertiary waterflooding

The experimental setups, presented in Figure 2a and 2b, was composed of a volumetric Isco[®] pump connected to a stock tank containing the crude oil when establishing the S_{wi} or directly to the core for the secondary or tertiary waterflooding tests. Pressure drop and fluid recovery were measured continuously.

Experimental Procedure:

1. One-phase experiments:

Brine saturation at ambient temperature:

The dry core was first saturated under vacuum, then flooded by brine #H. During the brine injection, the pressure drop and the pH of the effluents were monitored. Effluents were collected for ion analysis [ionic chromatography]. The equilibrium was assumed to be reached when effluents pH and ionic composition remain constant [between 10 and 20 pore volumes]. After saturation was achieved, the permeability of the core to brine #H was measured.

Dispersion test at ambient temperature:

This step was carried out for the DU3/0/1 core at ambient temperature. Brine #l was injected in the core at a constant flowrate [2ml/h] after equilibrium with brine #H. The effluent density was continuously monitored using on-line densimeter. pH and pressure drop was also monitored. Effluent fractions were collected to study ionic exchange during the dispersion stage. The dispersion coefficient and permeability to brine #l were then measured.

Dispersion tests at high temperature [60°C, 90°C]:

The objective of these experiments was to study the influence of temperature on single-phase flow dispersion in the porous medium. The dispersion temperature was 60°C for DU3/A/5 sample and 90°C for DU3/D/7. After saturation at ambient temperature with brine H, the cores were maintained at high temperature, while continuing to flood with brine #H. Once equilibrium was reached at high temperature, dispersion test with brine #l followed by brine #0.11 was performed. Effluent density, pH and pressure drop were monitored and effluents collected.

2. Two-phase experiments:

Initial water saturation and Aging:

The initial water saturation [S_{wi}] was established at ambient temperature for samples used in tertiary and secondary waterflooding before wettability change by aging process. Different protocols were used to attain low values of S_{wi} . In some cases, the use of paraffinic oil with higher viscosity than oil A was necessary. The direction of the oil flood was regularly reversed to prevent water saturation gradients. Permeability to crude oil at S_{wi} was also measured. The cores were then aged for 15 days at required temperature [60° or 90°C] and back-pressure [4 bar] in the Hassler coreholders. After the aging period, the dead crude oil in contact was replaced by fresh crude oil. The oil A permeability of the core was then re-measured.

Secondary and Tertiary oil recovery:

In the case of the tertiary experiment, after the aging process was completed, brine #H was flooded through the samples at constant flowrate (5ml/h). This was followed by flooding with low salinity brines. In the case of secondary recovery experiments, brine #1 was directly injected followed, in some cases, by brine 0.1#1. During secondary and tertiary recovery experiments, oil production was monitored as well as time evolution of other physical parameters [pressure drop, breakthrough time, pH and effluents ionic composition].

Two-phase dispersion at S_{or} :

For DU3/A/4 sample, two dispersion experiments were performed at ambient temperature after low salinity brine injection. First, the core at S_{or} was saturated with brine #1 then flooded with brine #H at 5ml/h. Effluent density was recorded by online densimeter. After dispersion at S_{or} , the core was cleaned using cycles of toluene and isopropanol floods followed by gas drying. One-phase flow dispersion was then performed. The comparison of one and two-phase dispersions gave some indication on the way the residual oil was distributed in the pore network and its role on the concentration dispersion.

Results

One-phase flooding with brines

Dispersion experiment at 20°C on DU3/0/1 sample showed a very homogenous sample with a dispersion coefficient of $1.5E^{-8}$ m²/s at 2ml/h. Experimental results for high temperature dispersions were presented in Figure 3. In both cases, pore volume by saturation and derived from effluent curves were in excellent agreement. During the first stage of the experiment corresponding to brine #H saturation at room temperature, the pH increased from 7.0 to 8.1 for DU3/A/5 and 6.7 to 7.6 for DU3/D/7. During this saturation, the pressure drop remained constant for both samples. When the system was held at high temperature, pressure drop increased whereas pH had curious variations composed of irregular increases and decreases. Switching brine #H to brine #1 led to a large pH increase from 7.3 to 9.6 for DU3/A/5 and 7.4 to 8.7 for DU3/D/7 while pressure drop remained constant. Pressure drop started increasing when injection brine was changed with brine #0.11, same as pH that continues increasing until 10 for DU3/A/5 and 9.6 for DU3/D/7.

Effect of brine concentration and composition

Three experiments, performed at $T = 35^{\circ}\text{C}$ confirmed additional oil recovery when low salinity brine was injected in tertiary mode. In Figures 4a and 4b, oil recovery, pH and pressure drop are plotted during the secondary and tertiary waterfloodings. We clearly observe an additional recovery of +11% ooip when injecting brine #b [composed only

of NaCl] after H brine. This gain of recovery was obtained even if no divalent ion was present in the invading brine. pH and pressure drop also increased as soon as brine #b was injected.

Figures 5a and 5b present similar results obtained with brine #1 [composed of 95% NaCl and 5% CaCl₂] on tertiary waterflooding. Additional oil recovery of +10.5% ooip is observed. The pH and pressure drop also increased as soon as brine #1 was injected. Switching from brine #1 to brine #m [same concentration but containing MgCl₂] didn't permit to improve recovery but decreasing brine salinity by injecting brine #0.11 led to a gain of 4.2%. At the end of the tertiary water flood, injection of brine #H was not followed by any oil production but confirmed pH and pressure drop dependence to the invading brine concentration. During this late brine #H injection, pH decreased and reached its initial values whereas pressure drop didn't decrease to its initial values.

Figures 6a and 6b present a different experimental procedure. In this case we injected brine #H followed by stepwise injections of brine #10l, brine #5l, brine #2.5l and brine #1 injections. No additional oil recovery was observed until brine #1 was injected where a gain of +9% ooip was observed. It's to note that pH and pressure drop increased at each brine injection step even if no additional oil recovery was obtained. This result suggests that there is not a direct relationship between pressure drop or pH increase and additional oil recovery and/or that a threshold pH must be overcome. It also suggests a threshold salt concentration value, between 2.5 and 1g/l, above which no additional recovery is possible.

Results of secondary recovery experiment with injection of brine #1 followed by brine #0.11 on DU3/A/6 are presented on Figures 7a, 7b and Figure 8. For a given concentration, oil recoveries in secondary and tertiary mode were very close. We also noticed a gain of production of +6.8% ooip with brine #0.11. The pH and pressure drop showed the same behavior as in tertiary mode.

Additional oil recovery was still observed whereas DU3 sandstone does not contain any kaolinite. The mechanism for additional oil recovery doesn't need the presence of kaolinite, even if the presence of clays seems necessary. One phase experiments showed that ionic exchanges take place in the first three pore volumes when low salinity brine was injected, which is the same as the additional oil recovery. This indicates the importance of ionic exchange in the recovery mechanism.

Two-phase dispersion at S_{or}

Figure 9 shows the comparison of the normalized concentrations versus cumulative injection for both the single phase and two-phase dispersion tests. In the single phase test, the injected concentration was produced after 1.7 PV of injection where 3 to 5 PV were required in the two-phase situation. Whereas the single phase concentration curve is symmetrical on DU3/A/4, the two-phase curve is largely skewed, with a long tail. In the former case, the test confirmed the homogeneity of the sample, whereas in the latter case, the high dispersivity suggested that part of oil is trapped in dead zones that can only be reached after injection of several pore volumes of brine. There is an excellent agreement between the S_{or} deduced from effluent curve and S_{or} from volumetrics.

Effect of displacement temperature

Results presented in figures 10a and 10b showed that final recovery and behavior after breakthrough of secondary high salinity water injection were strongly dependent on displacement temperature. The viscosity ratios μ_o/μ_w at 35°C, 60°C, 90°C were 7.5, 6.4 and 5.4 respectively. At $T^\circ = 35^\circ\text{C}$, oil production almost stopped after breakthrough whereas for high displacement temperatures it was followed by a long production tail. Final recovery with brine #H increased in increasing displacement temperature [67.8 %

ooip at $T = 35^{\circ}\text{C}$, 73.6% at $T = 60^{\circ}\text{C}$ and 80.8% at $T = 90^{\circ}\text{C}$]. It suggests that the wettability changes towards more intermediate-wet as temperature is increased.

No significant additional oil recovery by low water salinity was observed for high temperatures floods with brine #1 or brine #0.11. However, with the same brine/oil/rock, low salinity waterflood at $T = 35^{\circ}\text{C}$ exhibited a gain of oil production of +11% ooip with brine #1 and of +4.2% ooip with brine #0.11, especially in the first three pore volumes. It shows that the benefit of low salinity brine injection might be temperature dependent.

Discussion

In this study, one-phase and two-phase experiments, during which several physico-chemical parameters were monitored, aimed to further understanding of low salinity waterflooding. We discuss here some relevant points we observed during these flow tests.

pH increase was due to ionic exchange between clay particles and invading brine, and calcite dissolution [slower reaction]. However, pH increases were temperature dependant and reduced when displacement temperature [T_d] was increased. In two-phase flow, pH increase was also obtained when reducing brine concentration even if no additional oil was produced.

Observed pressure drop increases, obtained when reducing the salt concentration, was due to clay layers expansion after modification of electrical forces, as predicted by DLVO theory [Israelachvili, 1985]. Fines release can also happen when brine concentration was reduced even if, in our case, no significant particles were produced in the effluent.

Results obtained on DU3 sandstones confirmed that low salinity brine injection can lead to additional oil recovery in secondary and tertiary mode. However, in our experiments, some conditions in which gain of oil recovery was observed were different from literature examples. Samples used in this study were cut from a clayey sandstone that doesn't contain kaolinite. Results obtained with these cores suggest that even if kaolinite can play an important role in the recovery mechanisms, as proposed by Tang and Morrow [1999], and is more hydrophobic than other clays, additional oil recovery can be observed without the presence of this clay.

Additional oil recovery was also obtained when no divalent ions were present in the invading brine [but was present in the connate]. Divalent cations are known to mediate adsorption of polar components of oil on mineral surfaces during wettability alteration [Buckley 1997; Lager et al. 2006]. The mechanism based on multicomponent ionic exchange [MIE] and removal of oil components from clay surface replaced by uncomplexed cations is not sufficient to explain our results. When low salinity brine, composed only of NaCl is injected, these monovalent ions won't be able to remove divalent ions initially present on mineral surface. Affinity of clay surfaces to cations, in fact, increases when increasing valence and molar weight.

The impact of temperature on oil recovery in secondary mode can be explained by mobility differences between oil and water at high or moderate temperature. Tertiary recovery experiments conducted at $T = 35^{\circ}\text{C}$ and $T = 90^{\circ}\text{C}$ suggested a residual oil saturation that was attained either in increasing temperature or in lowering brine salinity. Experiment at $T = 60^{\circ}\text{C}$ doesn't show the same residual saturation but this sample was half the permeability than the others. The residual oil saturation is then not only dependant on displacement temperature and brine salinity, but also dependant on physical properties of the samples.

CONCLUSIONS

In this paper, we presented a study of influence of low salinity brine injection in tertiary and secondary mode accompanied by single-phase experiments. The crude oil/brine/rock system was preliminarily characterized. Dispersion experiments indicated that DU3 samples were very homogenous. Several observations have been made after this study:

- Additional oil recovery of about 10% was obtained for tertiary recovery experiments at moderate temperatures [$T = 35^{\circ}\text{C}$] when going from 50g/l to 1g/l. A slight additional gain of recovery was also obtained when 0,1g/l brine was injected.
- This recovery occurred even if no divalent ion was present in the low salinity brine.
- Gains of recovery occurred mainly in the first three pore volumes and were accompanied by pH and pressure drop increases. This increase can also occur without additional oil production.
- Successive dilution of injection brine showed a concentration threshold between 2.5 and 1g/l, above which no additional oil recovery was achieved.
- Oil recovery at 1g/l by secondary recovery experiment was close to the recovery at the same concentration by tertiary mode.
- All these additional oil recoveries occurred whereas DU3 sandstone does not contain kaolinite showing that the mechanism of additional oil recovery does not especially need the presence of kaolinite, even if presence of clay seems to be necessary.
- Final oil recovery increased with increasing displacement temperature. But no significant additional oil recovery was obtained when low salinity brine was injected at 60 or 90°C.

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TABLES AND FIGURES

Sample	Quartz	Albite	Microcline	Calcite	Apatite	Anatase	Muscovite	Chlorite	Illite
DU3/A	54.4	34	-	0.5	0.2	0.6	-	2.5	7.7
DU3/0	60.9	28	1.0	0.2	0.1	0.5	4.0	3.2	2.0

Table 1 : Mineralogical composition of the core samples.

Sample	SiO ₂	Al ₂ O ₃	Na ₂ O	K ₂ O	Fe ₂ O ₃	TiO ₂	MgO	CaO	P ₂ O ₅	MnO	SO ₃	BaO	SrO
DU3/A	82.49	9.26	3.76	0.82	1.06	0.6	0.43	0.57	0.08	0.03	0.01	0.01	0.01
DU3/0	83.1	9.18	3.56	0.96	0.96	0.51	0.5	0.26	0.06	0.01	-	-	-

Table 2 : Molecular composition of the core samples.

Brines	C(g/l)	NaCl (%w)	CaCl2 (%w)	MgCl2 (%w)
#H	50	90	10	-
#l	1	95	5	-
#10l	10	95	5	-
#5l	5	95	5	-
#2.5l	2.5	95	5	-
#0.1l	0,1	95	5	-
#b	1	100	-	-
#m	1	95	3,75	1,25

Table 3 : Concentration and Composition of brines.

Sample	k_g (mdy)	k_w (brine #H) (mdy)	Φ (%)	VP (ml)
DU3/0/1	704	330	19.3	17
DU3/0/2	825	419	20.5	18.1
DU3/0/3	600	130	17.4	15.3
DU3/0/4	553	232	18.1	16.0
DU3/0/5	681	139	16.4	14.3
DU3/A/3	467.6	54.8	21.5	18.9
DU3/A/4	426.1	109.0	19.2	17.1
DU3/A/5	398.7	75.6	19.9	17.5
DU3/A/6	467.9	81.6	19.3	16.9
DU3/D/7	790.3	---	19.4	17.1
DU3/D/9	796.6	385.3	18.7	16.5

Table 4 : Physical properties of the core samples

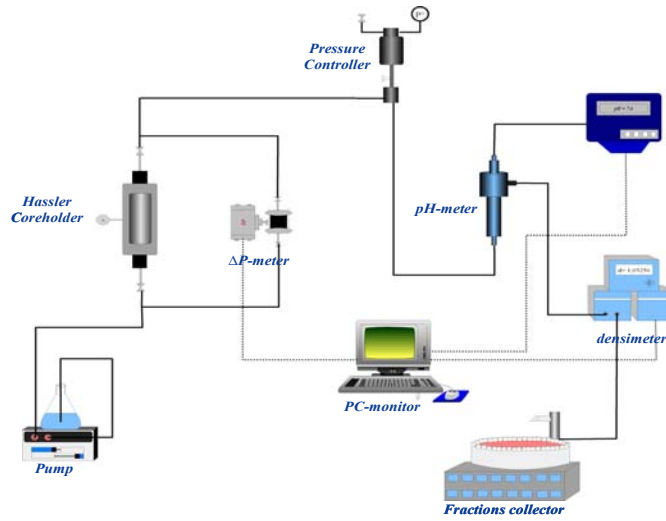


Figure 1: Experimental setup of core saturation and dispersion experiments: for controlled temperature the coreholder is set in an oven.

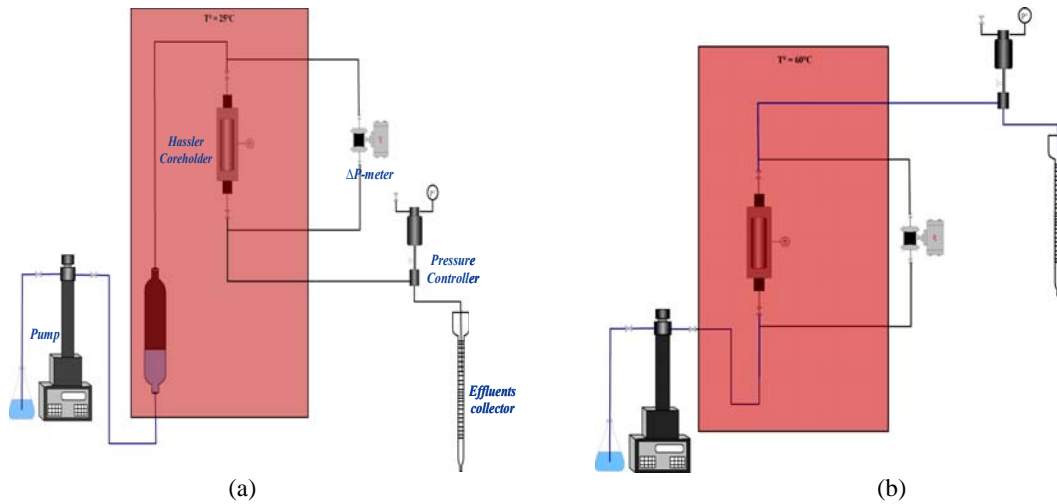
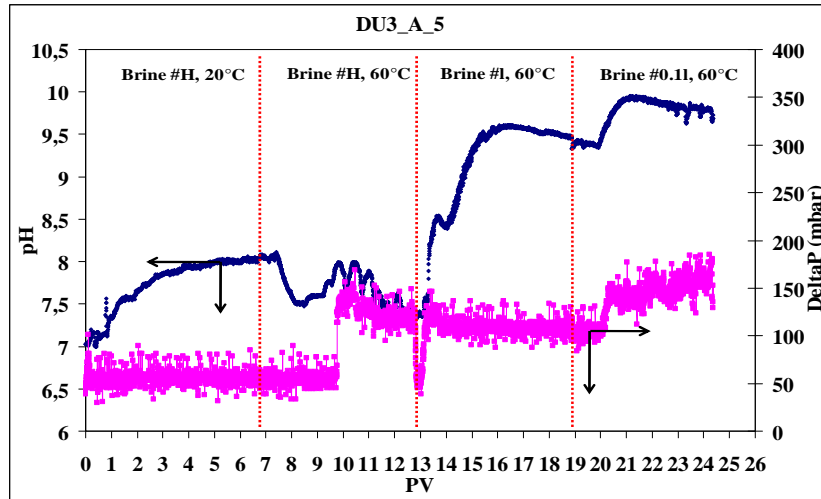
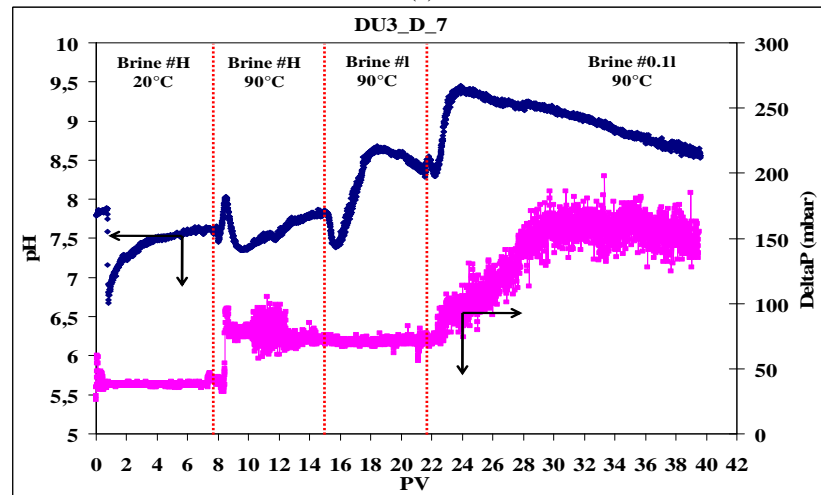


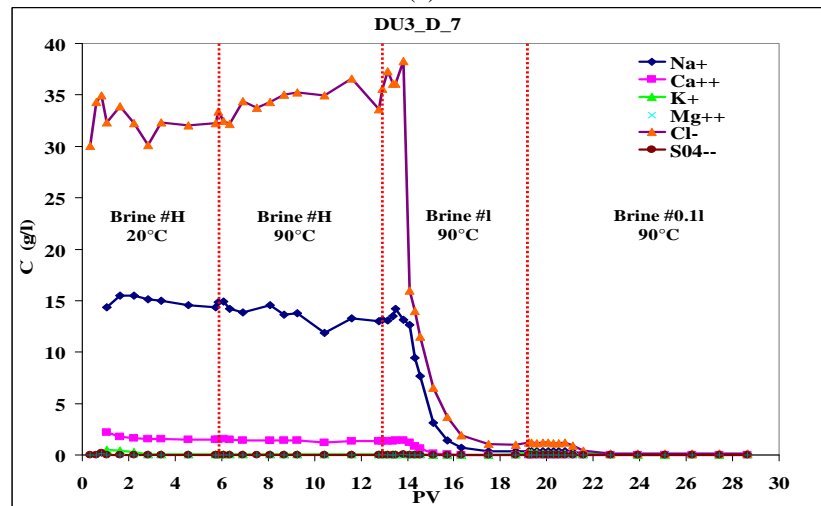
Figure 2: Experimental setup for initial water saturation establishment (a) and secondary or tertiary oil recovery experiments (b).



(a)



(b)



(c)

Figure 3: pH and pressure drop evolution on dispersion experiments at 60°C on DU3/A/5 (a), and 90°C on DU3/D/7 (b). Ionic concentration evolution during dispersion experiment at 90°C on DU3/D/7 (c). Flowrate = 5ml/h.

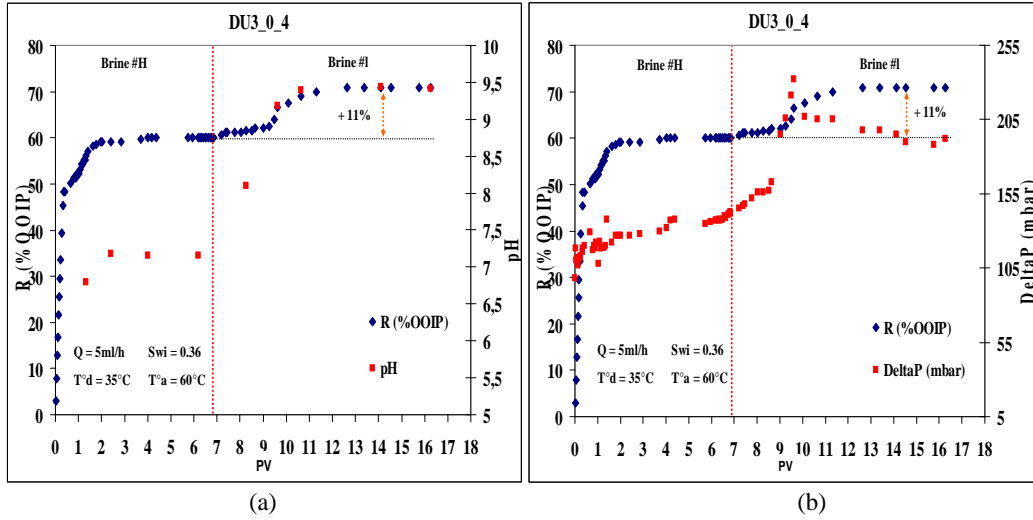


Figure 4: Oil recovery, pH and pressure drop evolution during tertiary recovery experiment on DU3/0/4.

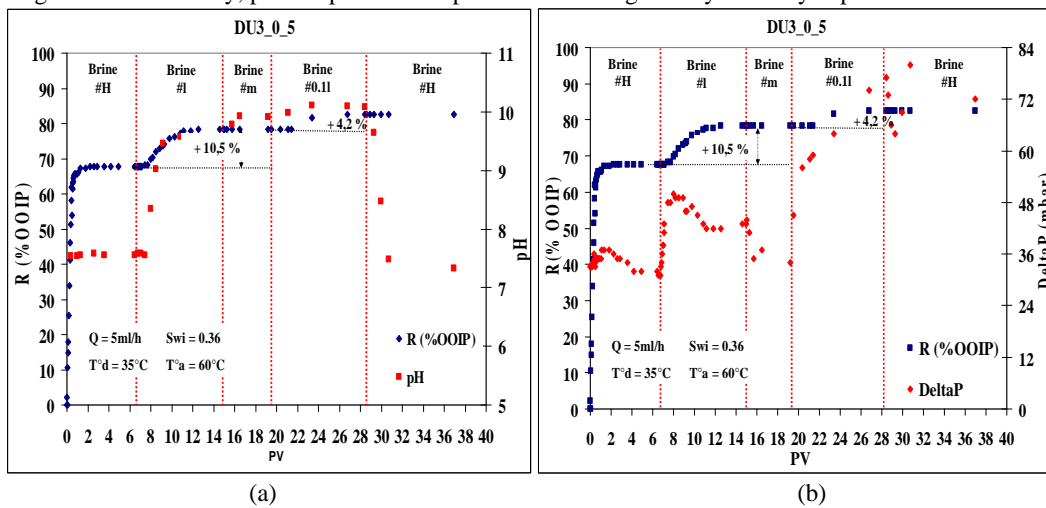


Figure 5: Oil recovery, pH and pressure drop evolution during tertiary recovery experiment on DU3/0/5

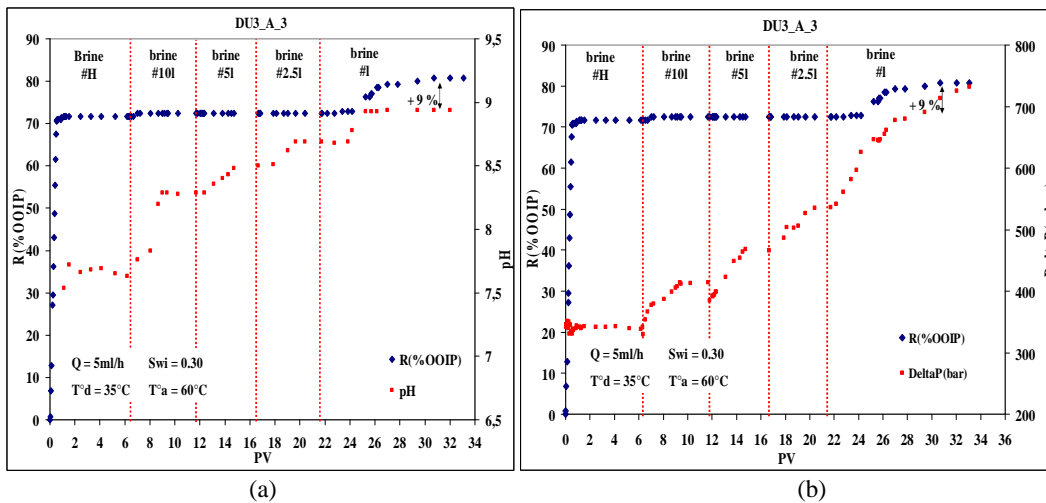


Figure 6: Oil recovery, pH and pressure drop evolution during tertiary recovery experiment with successive dilutions on DU3/A/3.

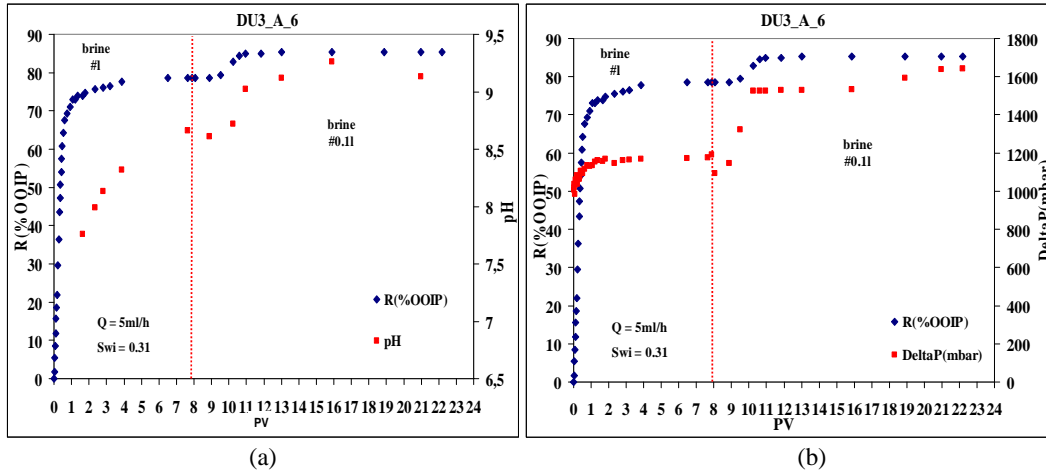


Figure 7: Oil recovery, pH and pressure drop evolution during tertiary recovery experiment on DU3/0/5.

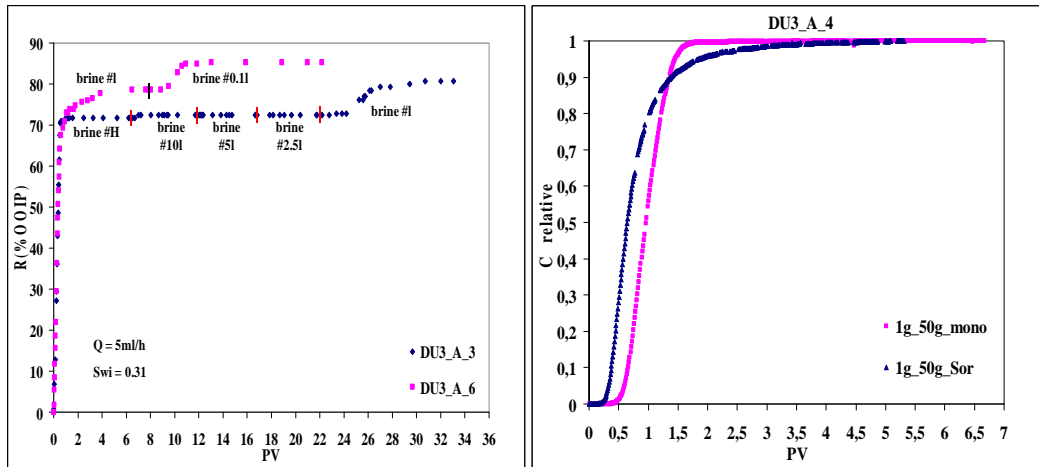


Figure 8: Comparison of secondary and tertiary recovery experiments.

Figure 9: Comparison of Sor and one-phase dispersions. Flowrate = 5ml/h

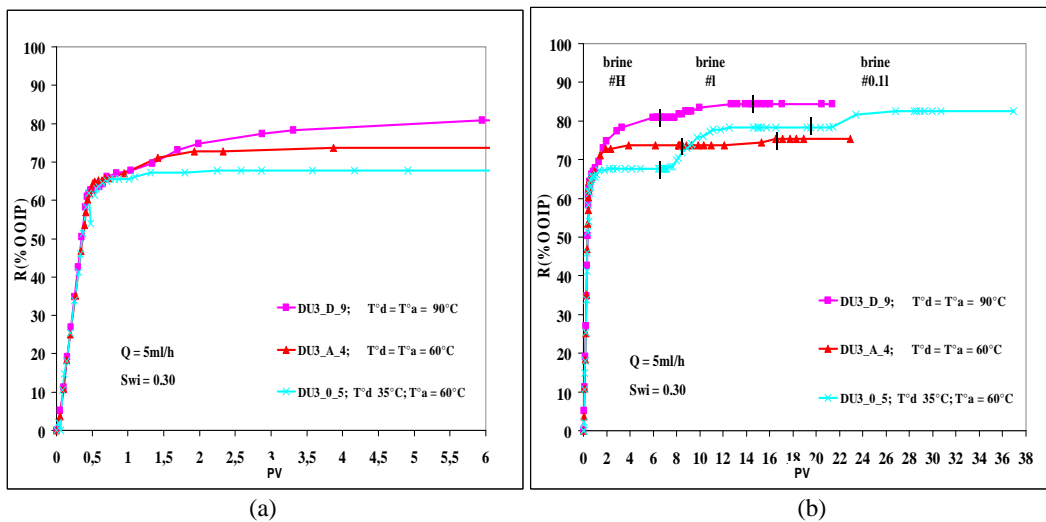


Figure 10: Evolution on oil recovery during tertiary experiments on DU3/0/5, DU3/A/4 and DU3/D/9 samples at different displacement temperatures.