Paper I

Alteration of wettability and wettability heterogeneity

A. Graue[*], E. Aspenes, T. Bognø, R.W. Moe, J. Ramsdal

Department of Physics, University of Bergen, Allegaten 55, 5007 Bergen, Norway

Abstract

This paper emphasizes how wettability may be altered for special core analysis purposes and by which processes this occurs. Studies of how the imbibition characteristics change after aging core plugs in crude oil are reported, focusing on imbibition rate and endpoint saturations and on the induction time. Imbibition characteristics after wettability alteration by aging core plugs submerged in crude oil at elevated temperature are compared to the effects from aging procedures, where crude oil is continuously flushed through the core plugs during the aging and after simply leaving the plug at connate water saturation under static, i.e. no flow conditions, shows different significant results. The results show that: (1) continuously introducing fresh crude oil boosts the aging process and (2) a significant impact on the wettability alteration as a function of core length is observed, reflecting the absorption of active components for the wettability alteration process. In an integrated study consisting of experiments and numerical simulation, this paper also demonstrates the importance of close interaction between how to alter the wettability, applications of the technique in core analysis and numerical simulations of the latter. Finally, the significance of some sensitive parameters is demonstrated. © 2002 Elsevier Science B.V. All rights reserved.

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1. Introduction

For improved special core analysis, it is important to establish a wettability condition as close as possible to that found in the reservoir, both for restored state core analysis of reservoir rock and when using outcrop rock.

The main objective of this work was to study the methods for altering the wettability for core analysis purposes. A secondary objective was to determine how the wettability change was produced and the stability and homogeneity of the wettability alteration.

A reproducible method of altering the wettability in outcrop rock has earlier been reported (Graue et al., 1995, 1999b; Eilertsen et al., 1999) and, recently, this technique has successfully been applied in laboratory studies of how oil recovery mechanisms in fractured reservoirs change depending on the wettability conditions (Graue et al., 1999a,c, 2000a,b; Viksund et al., 1996a,b, 1997; Graue and Bognø, 1999). In this paper, we have investigated why and how the wettability after aging in crude oil under certain conditions produces a nonuniform distribution. Included in this study also are the results on the significance of some selected parameters that demonstrate how crucial it is to be consistent in the choice of all experimental constants.

Not knowing that a heterogeneous wettability distribution is present may seriously affect the interpretation of laboratory experiments. An example of a

* Corresponding author. Tel.: +47-555-82-721; fax: +47-555-89-440.

E-mail addresses: arne.graue@fi.uib.no (A. Graue), eirik.aspenes@fi.uib.no (E. Aspenes), thomas.bogno@fi.uib.no (T. Bognø), robert.moe@fi.uib.no (R.W. Moe), johannes.ramsdal@fi.uib.no (J. Ramsdal).

radial variation in wettability during aging of core plugs submerged in crude oil has been reported (Spinler et al., 1999). The phenomenon was observed through the variation in capillary pressure scanning curves, detected by NMR-imaging, across the cross section of the aged core plugs.

In some recent publications, (Graue and Bognø, 1999; Graue et al., 2000a,b) heterogeneous wettability conditions, created by aging in crude oil, had bearing on the interpretation of the oil recovery mechanisms in fractured blocks of chalk at different wettability conditions. Visualization of the effects of various wettability conditions on oil recovery in fractured chalk reservoirs was demonstrated in two-dimensional in situ imaging experiments in large blocks of chalk and by numerical simulation. Recovery mechanisms changed with wettability. Iterative comparison between experimental work and numerical simulations was used to predict oil recovery mechanisms in fractured chalk as a function of wettability. In large-scale nuclear tracer, 2D-imaging experiments waterflooding blocks of chalk were monitored, first for whole blocks and then for fractured blocks. Capillary pressure and relative permeabilities at each given wettability were measured and used as input for the simulations. The results from these studies emphasize the importance of close interaction between the method applied to alter the wettability and the interpretation of the experiments at various wettability conditions.

2. Experimental

To establish larger physical reservoir models in the laboratory, various outcrop rocks have been collected. The objective of scaling up the experiments is to reduce capillary end effects and to investigate, at a larger scale, the simultaneous interaction between the capillary, viscous and gravity forces. The study of alteration of wettability emerged from a need to establish a range of wettability conditions in this outcrop core material. In earlier work (Eilertsen et al., 1999), results on altering the wettability in sandstone and in several chalks were reported. This paper reports on results using the Rørdal chalk from the Portland Cement Factory in Ålborg, Denmark. More than 200 core plugs have been included in this study. In this

paper, a total of 27 chalk core plugs and two chalk blocks have been selected and included in the list of core material in Table 1 to investigate some effects that may occur when altering the wettability. Recent work emphasizes the challenge of establishing uniform wettability distribution in chalk.

2.1. Rock and fluids

The core plugs were all cut with the same orientation from large chunks of Rørdal chalk. Porosity was determined from weight measurements and the permeabilities were measured (see Table 1). Further information on the chalk is found in Graue et al. (1999b). The chalk blocks, CHP-8 and CHP-14, with dimensions of $20 \times 10 \times 5$ cm, were cut from large pieces of the outcrop chalk and epoxy-coated. Local air permeability, measured at each intersection of a 1×1 cm grid on both sides of the blocks using a minipermeameter, indicated homogeneous blocks on a centimeter scale. Complete data on the chalk blocks are found in Table 2 of Graue and Bognø (1999) and in Graue et al. (2000a).

The core plugs and the blocks were vacuum-evacuated and saturated with brine containing 5 wt.% NaCl + 3.8 wt.% $CaCl_2$. $CaCl_2$ was added to the brine to minimize dissolution of the chalk. Sodium azide, 0.01 wt.%, was added to prevent bacterial growth. The density and viscosity of the brine were 1.05 g/cm^3 and 1.09 cP at 20 °C, respectively. The brine was filtered through a 0.45-μm paper filter membrane. The salts used in the brine were NaCl and $CaCl_2$, both with a purity of 99.5% and obtained from Phil. Sodium azide also had a purity of 99.5%. The materials were used as received. The physical properties of the fluids are summarized in Table 2.

2.2. Initial S_w

Immobile water saturations were established by oilflooding. In order to obtain a uniform, low initial water saturation, S_{wi}, oil was injected alternately at both ends of the block. The core plugs to be used at strongly water-wet conditions were oilflooded using decane. Oilfloods of the block and the core plugs to be used at less water-wet conditions were drained using a stock tank crude oil from a North Sea chalk reservoir at 90 °C in a heated pressure vessel. Two cores were

Table 1
Summary of core data

Core no.	ϕ [%]	PV [ml]	k [md]	I_w	Aging time	Remarks
CPJR 1	47.2	75.7	4.1	0.6	14 days	aged while submerged in crude oil
CPJR 2	47.4	75.1	3.4	0.1	14 days	aged while submerged in crude oil
CPJR 3	46.9	73.0	4.0	0.4	14 days	both core plugs aged while submerged
CPJR 4	47.2	76.9	3.7	0.5	14 days	in crude oil 3 days at 90 °C + 2 days at 20 °C + 11 days at 90 °C
CPJR 7	47.0	73.7	3.8	1	3 days	oil from new oil barrel
CPJR 13	46.7	71.0	4.5	1	3 days	oil from new oil barrel
CPJR 14	47.0	71.4	3.4	1.0	3 days	oil from new oil barrel
CPJR 15	47.7	73.2	5.0	1	3 days	oil from new oil barrel
SP 34	48.0	77.6	5.3	0.6	3 days	oil from old oil barrel
SP 35	47.9	77.8	4.6	0.7	3 days	oil from old oil barrel
SP 1	46.8	74.7	4.2	0.4	1 day	$S_{wi} = 18.0\%$
SP 3	47.6	76.1	6.8	0.4	1 day	$S_{wi} = 20.5\%$
SP 27	47.2	76.4	4.0	0.7	1 day	$S_{wi} = 24.7\%$
SP 36	46.9	73.0	3.7	0.7	1 day	$S_{wi} = 24.6\%$
CPA-1.1	48.0	74.7	3.4	0.6	72 h	aged while submerged in crude oil
CPA-1.2	46.2	74.0	3.5	0.6	72 h	aged while submerged in crude oil
CPA-1.3	47.1	74.8	3.7	0.5	73 h	aged in core holder with unidirectional flow of crude oil
CPA-1.4	47.9	77.3	4.2	0.4	72 h	aged in core holder with unidirectional flow of crude oil
CPA-1.5	45.8	65.6	4.4	1.0	0 day	not aged
CPA-1.6	46.7	61.2	4.1	1.0	0 day	not aged
CPA-1.7	46.4	57.6	5.3	1.0	72 h	aged in core holder with no flow
CPA-1.8	47.3	51.5	4.5	1.0	72 h	aged in core holder with no flow
CPA-3.1	45.6	138	4.1	0.3	72 h	aged in core holder with multidirectional flow of crude oil
CPA-4.1	47.3	36.3	3.4	0.4	72 h	aged in core holder with unidirectional flow of crude oil
CPA-4.2	45.3	32.9	3.5	0.7	72 h	aged in core holder with unidirectional flow of crude oil
CPA-4.3	46.3	75.3	3.9	0.9	72 h	aged in core holder with unidirectional flow of crude oil
CPA-4.4	47.0	34.8	3.0	1.0	72 h	aged in core holder with unidirectional flow of crude oil
CPA-4.5	46.5	35.8	4.8	1.0	72 h	aged in core holder with unidirectional flow of crude oil
CHP-8	47.6	516	2.3	0.8	83 days	aged in core holder with no flow
CHP-14	46.2	450	2.2	0.6	2 days	aged in core holder with unidirectional flow of crude oil

Table 2
Fluid properties

Fluid	Density [g/cm^3]	Viscosity at 20 °C [cP]	Viscosity at 90 °C [cP]	Composition
Brine	1.05	1.09		5 wt.% NaCl + 3.8 wt.% CaCl$_2$
n-Decane	0.73	0.92		
Decahydronaphtalene	0.896			
Crude oil	0.849	14.3	2.7	

oilflooded to low S_{wi} with a viscous mineral oil that subsequently was flushed out by decahydronaphthalene (decalin) followed by crude oil. The pressure vessel used for the oilfloods was capable of holding up to 1000 kPa of confining pressure so that a maximum pressure drop of 900 kPa could be applied. This resulted in a lower limit of 25% S_{wi} of the blocks and this was taken as a baseline S_{wi}.

2.3. Alteration of wettability

Wettability was altered by aging in crude oil at elevated temperature for various lengths of time. Because of possible wax problems with the selected North Sea crude oil, the aging temperature was maintained at 90 °C as long as crude oil was in the rock. Crude oil composition was measured to be 0.90 wt.% asphaltenes, 53 wt.% saturated hydrocarbons, 35 wt.% aromatics and 12 wt.% nitrogen–sulphur–oxygen (NSO)-containing components. The acid number was measured at 0.09 and the base number at 1.79 (Buckley, Private communication). The following procedure for preparing the crude oil was used: (1) the barrel containing crude oil was shaken and the crude oil was tapped from the center of the barrel; (2) the crude oil was heated to 90 °C in a closed accumulator cell and flushed through a chalk core filter (this core was at immobile water saturation) in-line with the test core plug.

After aging, the crude oil was flushed out with decahydronaphthalene (decalin) which in turn was flushed out by n-decane at 90 °C. Decalin was used as a buffer between the mineral oils and the crude oil to avoid asphaltene precipitation. Decane and brine were used during the waterfloods of the plugs and the block.

Duplicate sets of core plugs drilled from the same piece of chalk received the same treatment for wettability alteration. Sets of core plugs with similar mineralogy and pore geometry but with different wettabilities were prepared using several different aging techniques. The wettability of core plugs submerged in crude oil in a closed beaker stored at 90 °C was compared to that obtained by aging core plugs, mounted in a core holder, with and without continuous flow of fresh crude oil through the plug during the aging process. Aging with crude oil flushed in only one direction was compared with alternating direction

of flooding. Finally, the effect of continued aging after the plug had cooled to room temperature, e.g. because of power failure, was also determined.

2.4. Wettability testing

After the aging was completed, wettability was measured by the Amott–Harvey test. The definition and formula for the Amott index to water can be found in Amott (1959). Wettability was also evaluated by considering the imbibition characteristics reflected by the imbibition rate, the imbibition water saturation endpoint and the induction time for onset of imbibition.

2.5. Imaging waterfloods in chalk

For all imaging purposes, a nuclear-tracer imaging technique was used (Bailey et al., 1981; Lien et al., 1988; Graue et al., 1990; Graue, 1994). Nuclear tracer, 2D-imaging experiments monitored the waterflooding of the chalk blocks: first whole, then fractured (Graue and Bognø, 1999; Graue et al., 2000a). 2D-brine saturations were determined by measuring gamma-ray emission from ^{22}Na and ^{60}Co, separately dissolved in brines used alternately as injection water and in situ water (Graue et al., 2000a). The block was mounted in a vertical position while flooded and a saturation map was produced at each specified point in time. Brine saturation development in core plugs was also measured using the nuclear-tracer technique.

3. Results and discussion

Fig. 1 shows the effect on spontaneous brine imbibition characteristics for core plugs containing brine and decane that had been submerged in crude oil at 90 °C for various lengths of time. The figure shows that the imbibition rate and the imbibition endpoint for the water saturation decrease as the aging time increases. The induction time, i.e. the time before imbibition starts, increases as the aging time increases. The measured Amott indices for these core plugs show a decrease towards less water-wet conditions with increasing aging time. Although this aging technique produced reproducible and stable wettability conditions (Figs. 2 and 3 in Graue et al., 1999b), it has recently been shown that for long aging times
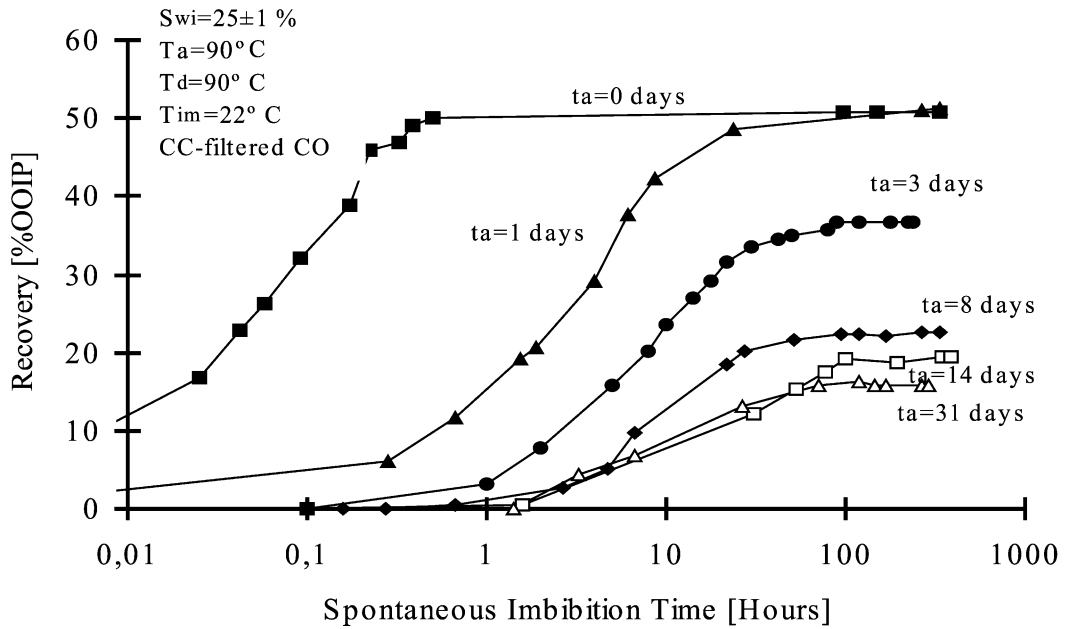
Fig. 1. Effect of aging time on spontaneous brine imbibition characteristics and oil recovery.

(Spinler et al., 1999), heterogeneous wettability distribution within the core plug may be encountered. The nature of this nonuniformity of the wettability, being symmetrical with respect to the radii of the core plug, leads to speculations that this may be due to the effects from diffusion of the surrounding crude oil.
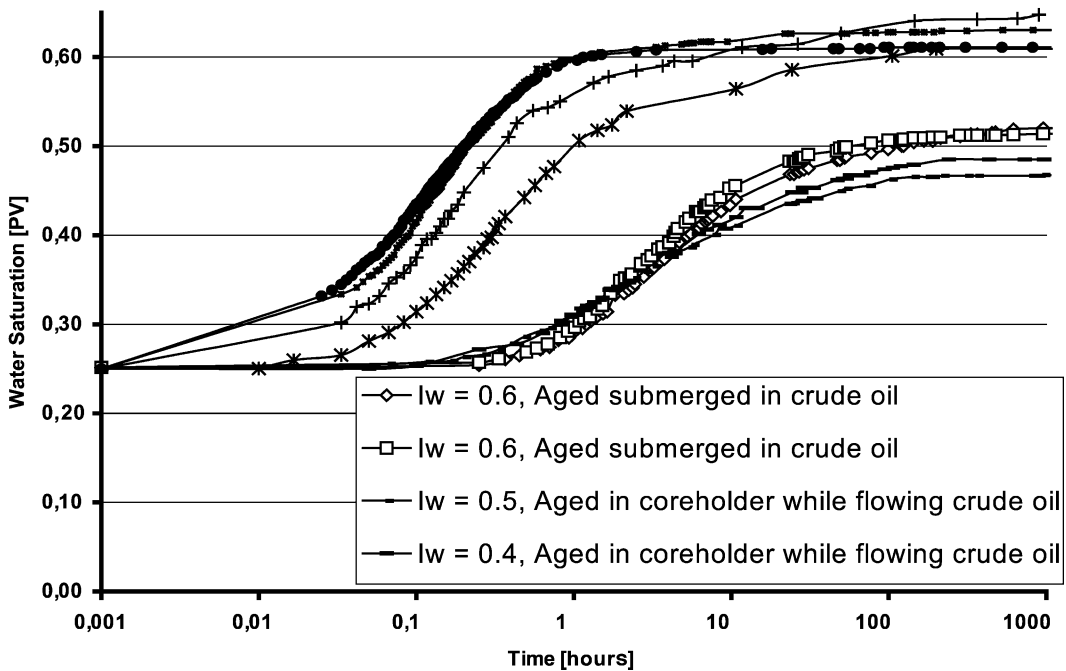

Fig. 2. Imbibition characteristics for duplicate sets of core plugs aged for 72 h using three different aging techniques.

Less efficient aging was also observed in epoxy-coated blocks of chalk compared to core plugs aged the same length of time submerged in crude oil. Thus, in Fig. 2, the imbibition characteristics for duplicate sets of core plugs aged for 72 h using three different aging techniques are compared. Aging core plugs submerged in crude oil in a closed jar stored at 90 °C were compared to aging core plugs mounted in a core holder, with and without the continuous flow of fresh crude oil through the plug during the aging process. Baseline imbibition characteristics for two strongly water-wet plugs are included for comparison. The figure shows that the most efficient aging as judged by alteration in wetting occurred when fresh crude oil was flushed through the plugs during the aging process. The static aging in a core holder, where only the initial amount of oil in place was available as the active component for the aging process, exhibited very poor wettability alteration capabilities. This indicates that the diffusion of fresh crude oil at 90 °C may play an important role for changing the wettability in the plugs submerged in crude oil by providing additional "active" components for the aging process.

The increased efficiency for altering wettability observed when continuously flushing the core plug with crude oil during aging indicated that increased concentration of some "active" components accelerated the aging process. Concern about potential absorption of these components as function of core length resulted in an experiment schematically illustrated in Fig. 3. Five core plugs were stacked in a core holder and continuously flushed in one direction during 72 h of aging. The initial water saturation was 25% PV and its distribution, measured with a nuclear-tracer technique, is shown in the figure. After aging and the subsequent flooding with decalin and decane, the imbibition characteristics of each core plug, saturated with brine and decane, is shown in Fig. 4. From this figure, it is clear that the aging process becomes less efficient with increasing core length. It is interesting to notice that the last core plug, position 19.1–22.8 cm, is less water-wet than the second last plug. This is probably because the crude oil direction was reversed for a short period of time at the end of the drainage process to smooth the capillary end effect that is always observed after completion of
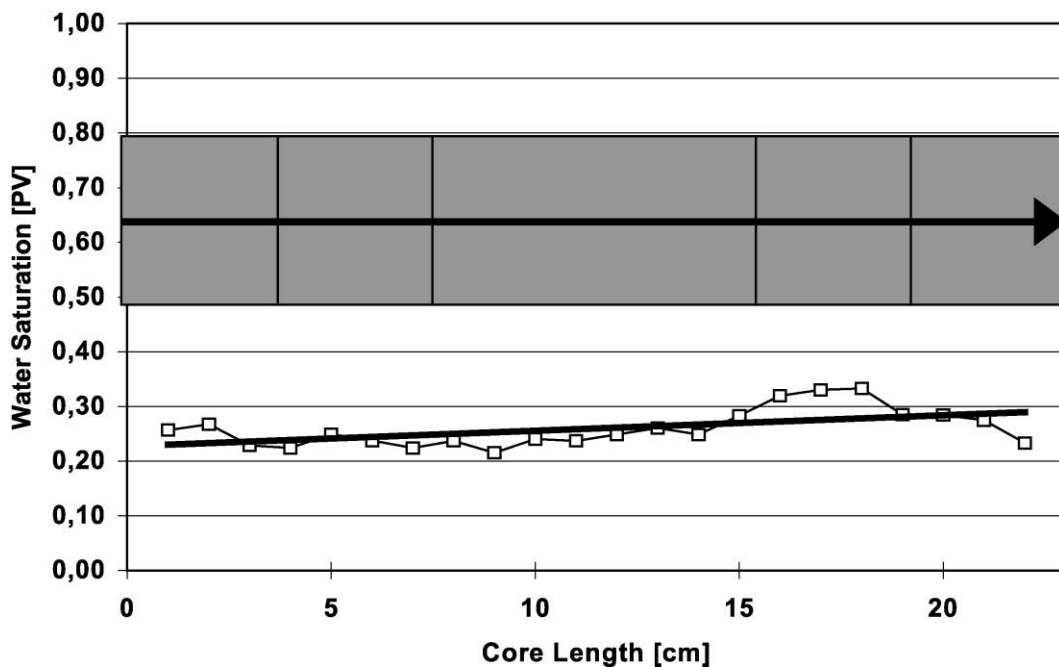

Fig. 3. Schematic experimental set-up for five stacked core plugs and the initial water saturation profile for the cores when aged during unidirectional flow of crude oil.
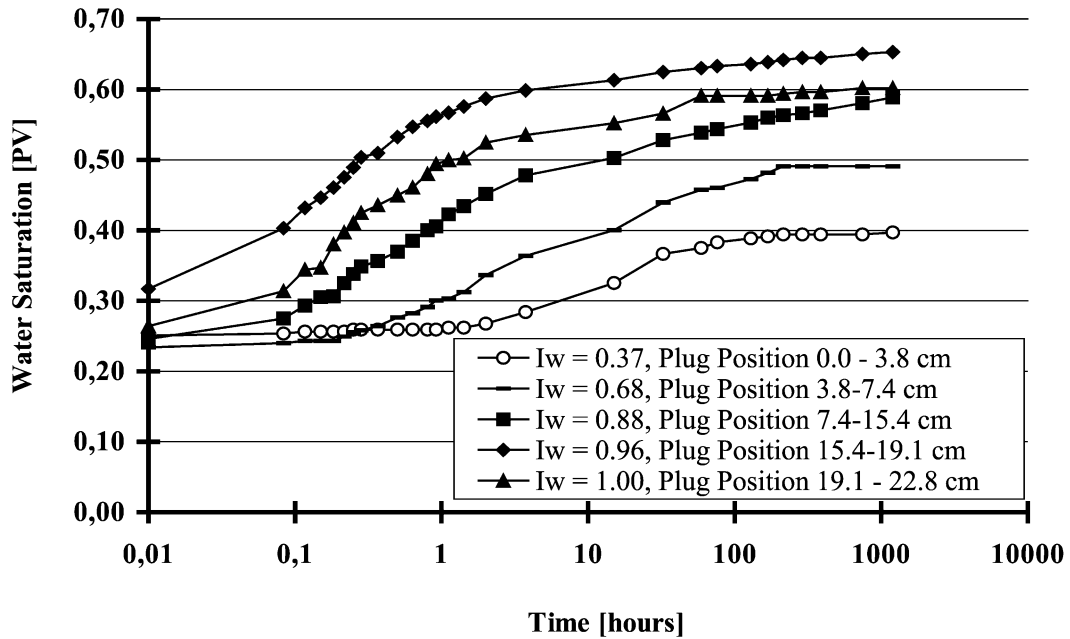
Fig. 4. Individual imbibition characteristics of the five stacked core plugs after 72 h of aging during unidirectional flow of crude oil.

the primary drainage. Fig. 5 shows that the in situ water saturations before imbibition, after spontaneous brine imbibition and after the subsequent waterflood (to obtain the Amott index) are completed. The solid line in the figure indicates the distribution of the Amott index to water as calculated by material
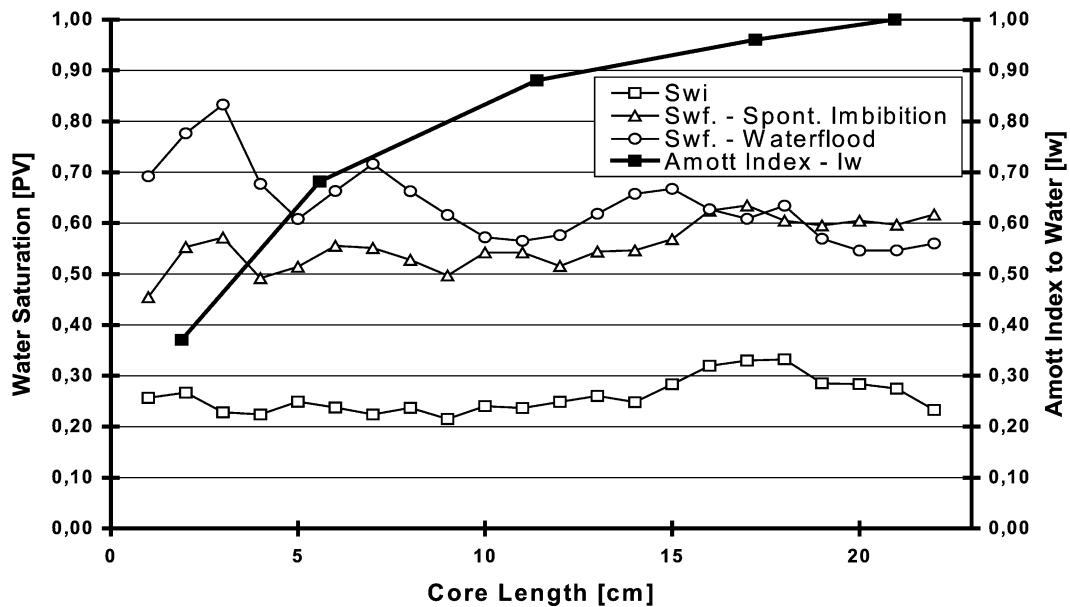

Fig. 5. Water saturation development for the five stacked cores after 72 h of aging with unidirectional flow of crude oil. Initial water saturation profile, water saturation after spontaneous imbibition and after the waterflood are all obtained by the nuclear-tracer technique. Wettability distribution, I_w, obtained from each core plug is indicated by the solid line.
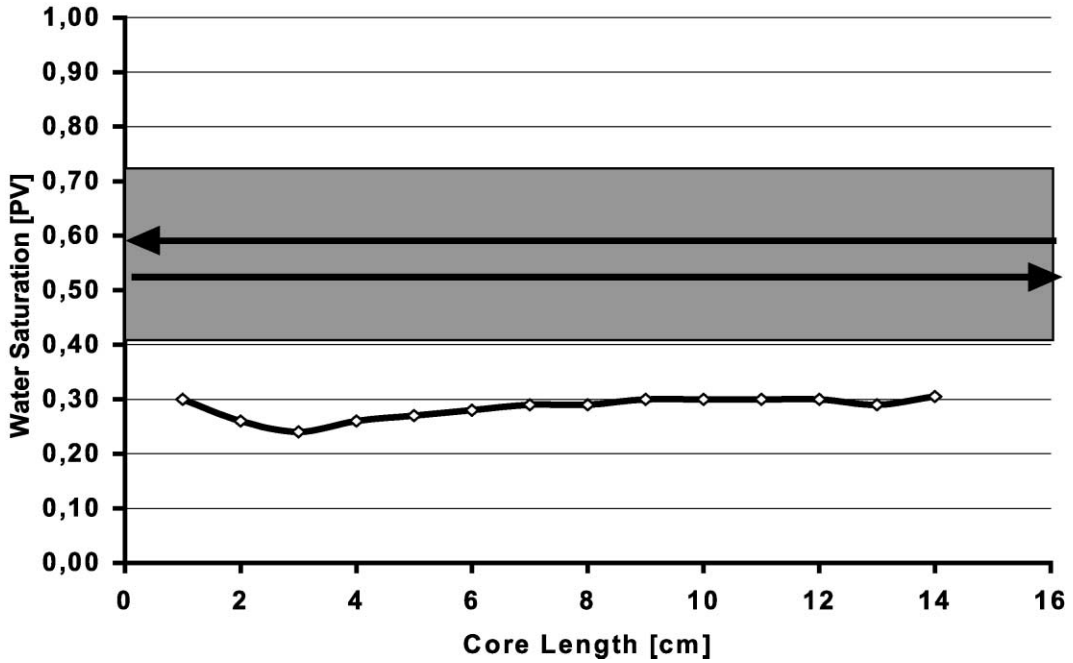
Fig. 6. Schematic experimental set-up for a 15-cm-long core and the initial water saturation profile for the core when aged during multi-directional flow of crude oil.
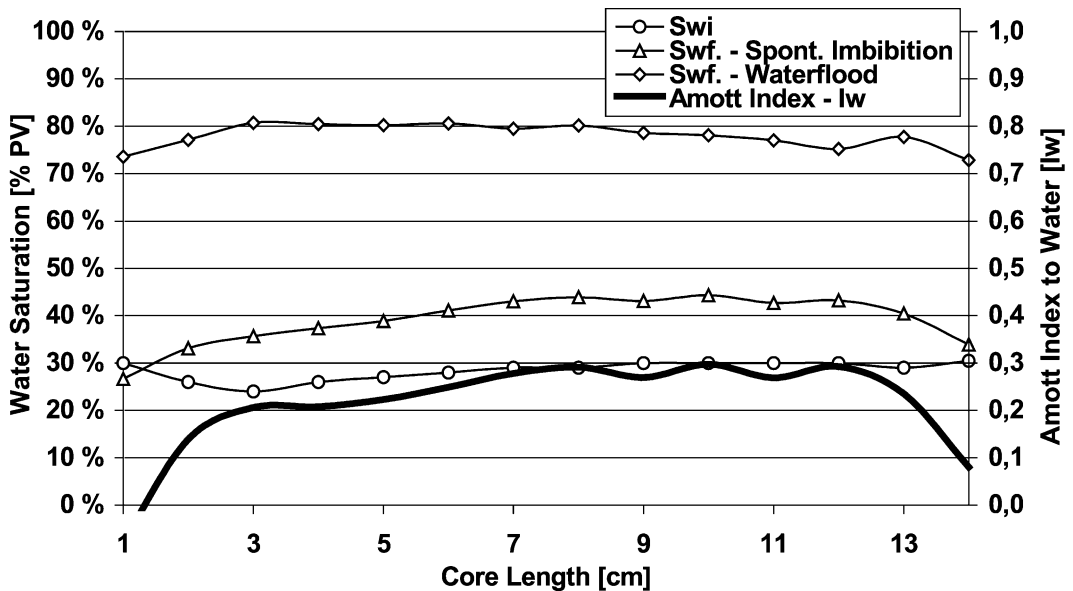

Fig. 7. Water saturation development for the 15-cm-long core after 72 h of aging with multidirectional flow of crude oil. Initial water saturation profile, water saturation after spontaneous imbibition and after the waterflood are all obtained by the nuclear-tracer technique. Wettability distribution, I_w, obtained by the nuclear-tracer technique plug is indicated by the solid line.
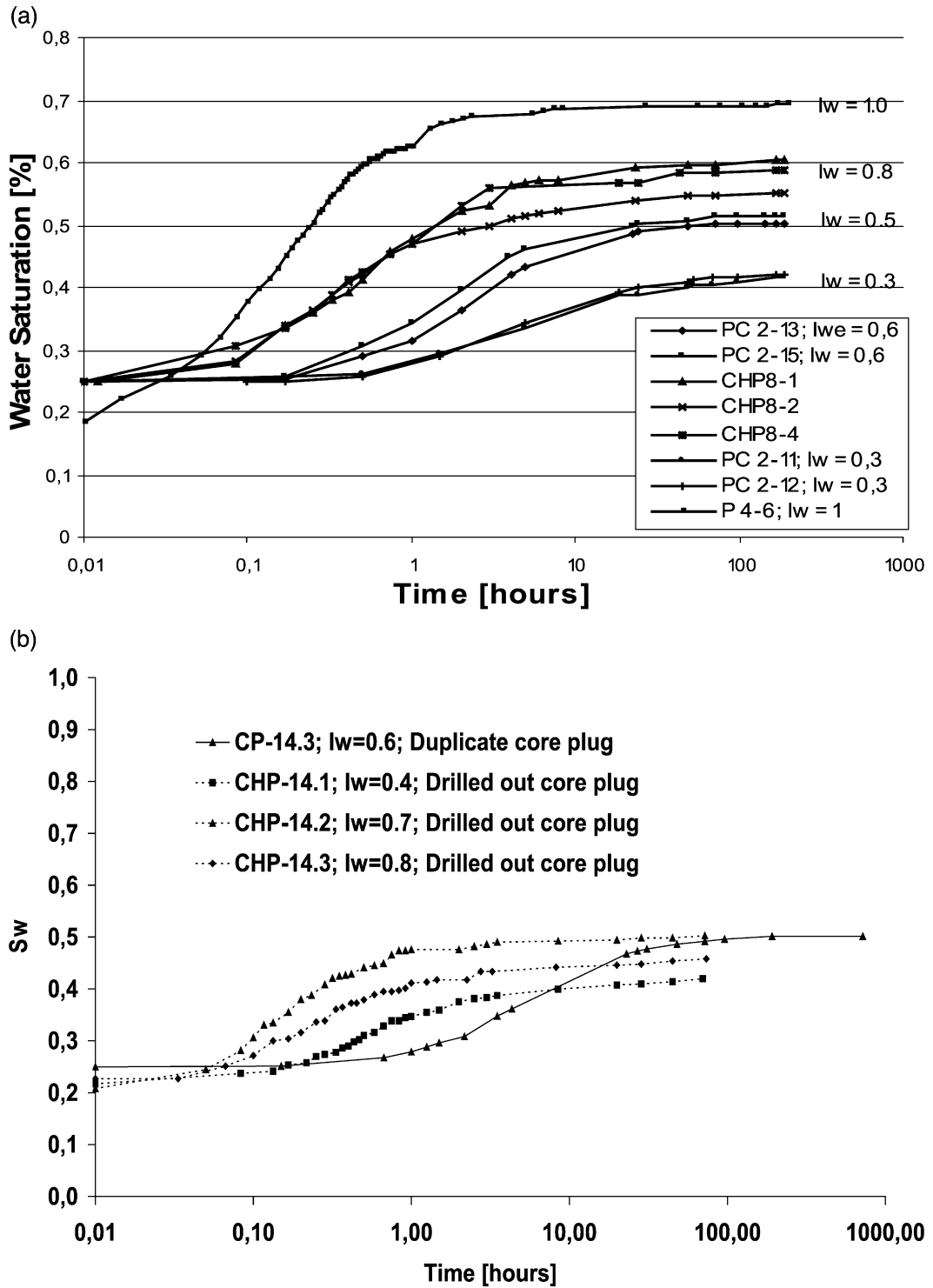
Fig. 8. (a) Imbibition characteristics for the duplicate core plugs and drilled-out plugs from the moderately water-wet block CHP-8. (b) Imbibition characteristics for the duplicate core plug and drilled-out plugs from the moderately water-wet block CHP-14.

balance from each separate imbibition test on each plug.

Fig. 6 shows schematically an experiment designed to test the effect of reversing the oilflood direction during aging in a 15-cm long, 2-in diameter core plug. In this experiment, the direction of continuous oil flow, with a flow rate of 2 pore volumes per day (PV/day), was changed four times during the aging process. The initial water saturation distribution was imaged by the nuclear-tracer imaging technique and showed a uniform distribution. Fig. 7 shows the initial water saturation distribution, the water saturation distribution after the spontaneous imbibition and the final water saturation distribution after the waterflood to obtain the Amott index to water. The solid line in the figure indicates the distribution of the Amott index to water as calculated by the in situ water saturation measurements. The middle section of this core would provide a core plug with homogeneous wettability conditions. If a shorter core plug is extensively flushed in both directions, the results show that a fairly uniform wettability distribution will be obtained. Our experience is, however, that the results of an imbibition test may be improved by cutting away the inlet and outlet face of the plugs and use in situ saturation imaging to obtain the saturation development.

To determine the impacts from wettability on oil recovery mechanisms in fractured reservoirs, scaled-up laboratory waterflood imaging experiments with corresponding numerical simulations have been performed. To investigate the simultaneous interaction between capillary forces, viscous forces and gravity and to reduce the impacts from capillary end effects, blocks of outcrop rock were used instead of core plugs. Imaging the local in situ 2D-saturation development of the individual water compositions, the in situ water and the injection water, the mixing of the waters was determined.

Chalk blocks were aged in crude oil to provide moderately water-wet conditions. Epoxy-coated blocks aged at elevated temperature did not show as efficient wettability alteration as the duplicate set of plugs treated similarly. Fig. 8a, obtained from Graue et al. (2000b), shows the imbibition characteristics for the twin plugs treated similarly to the block CHP-8 and for the three plugs drilled from the block. Imbibition characteristics for one strongly water-wet plug

and two plugs at nearly neutral-wet conditions are included in the figure for comparison. Compared to the strongly water-wet baseline, a consistent change in wettability towards less water-wet conditions, longer induction time, lower imbibition rate and lower final water saturation was obtained. However, the twin plugs both exhibit an Amott index to water of 0.5 while all three drilled-out plugs consistently had $I_w = 0.8$. The aging technique used for block CHP-8 was, therefore, modified when block CHP-14 was aged. Crude oil was continuously flushed unidirectionally through the block CHP-14 during aging. Fig. 8b, obtained from Graue et al. (2000b), shows the imbibition characteristics for the twin plugs aged in this manner and for the drilled-out plugs from CHP-14. The inlet end of the block gave a wettability index to water of $I_w = 0.4$. The central part exhibited more water-wet conditions, corresponding to a wettability index to water of $I_w = 0.7$, while the outlet end showed $I_w = 0.8$.

Fig. 9 shows the match of the 1D in situ water saturation development for the moderately water-wet block CHP-14 when waterflooded as a whole block. In this simulation, using the simulator SENSOR (Coats et al., 1995), the experimental capillary pressure curves and the optimized relative permeability curves were used. However, the history match was not good, probably due to the heterogeneous wettability conditions in this block, since only unidirectional flow during aging had been applied. A new simulation was therefore run with three sets of relative permeability and capillary pressure curves, shown in Figs. 10 and 11, reflecting the variation in wettability recorded in the drilled-out plugs. The revised simulation is shown in Fig. 12.

Fig. 13 shows the impact from accidentally cooling down the plugs during aging. Temperature fluctuations by power failure or by other causes may disturb or ruin long-term aging tests. Four core plugs in two duplicate sets were aged for 14 days submerged in crude oil. One set was cooled down after 3 days and was kept at room temperature for 2 days before being reheated and aged for another 11 days. Fig. 13 shows the results on the imbibition characteristics. One of the core plugs aged continuously for 14 days shows very different imbibition characteristics. Such an odd behavior is sometimes observed, but is infrequent, and we believe this is probably due to rock hetero-
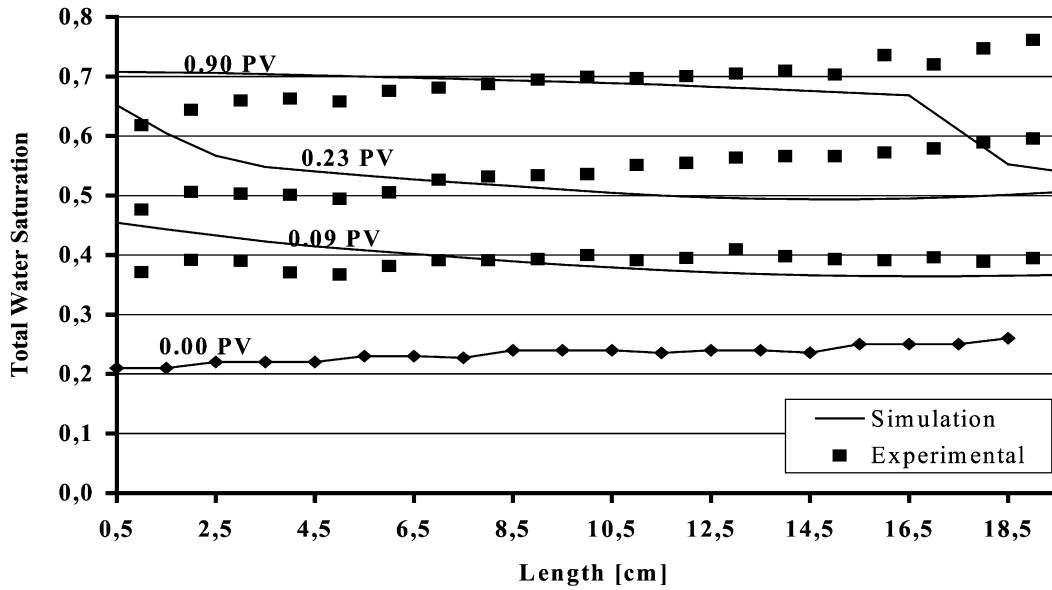
Fig. 9. Experimental and simulated water saturation profiles averaged over cross section of block when waterflooding whole block CHP-14 using the homogeneous wettability conditions in the simulation. Wettability is assumed uniform at $I_w = 0.6$.

geneities. The results from this test do not indicate severe impact from the reduction of temperature during the aging process.

Fig. 14 shows the effect on the aging process of different initial water saturations. From the figure, it is evident that only 5% lower initial water saturation
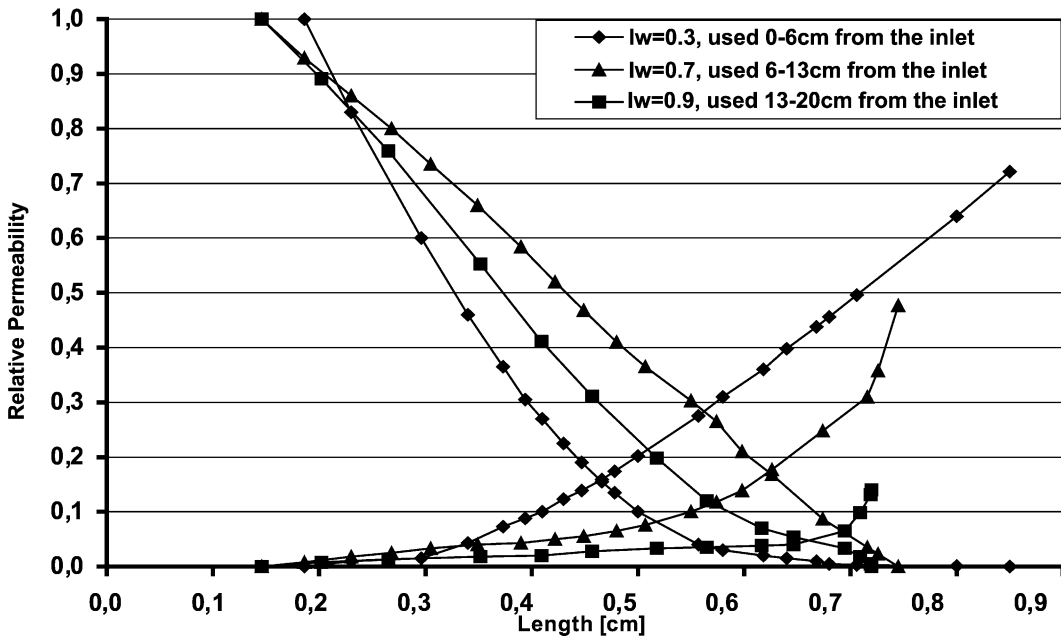

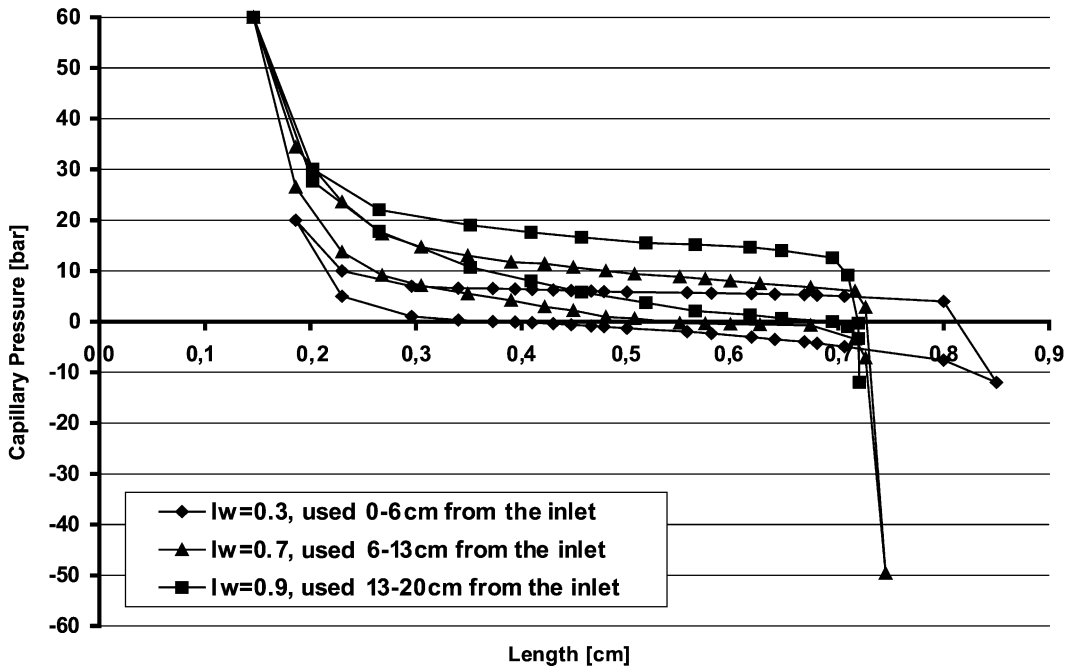Fig. 10. Relative permeability curves at different wettabilities.

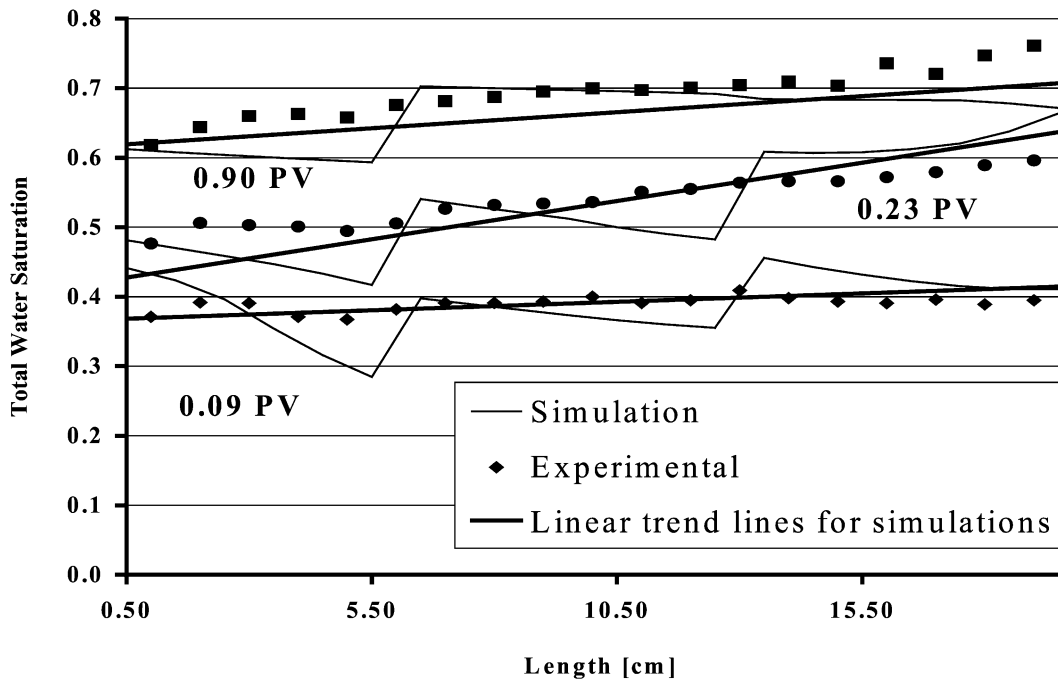Fig. 11. Capillary pressure curves at different wettabilities.

Fig. 12. Experimental and simulated water saturation profiles averaged over cross section of block when waterflooding whole block CHP-14 using the heterogeneous wettability conditions in the simulation.
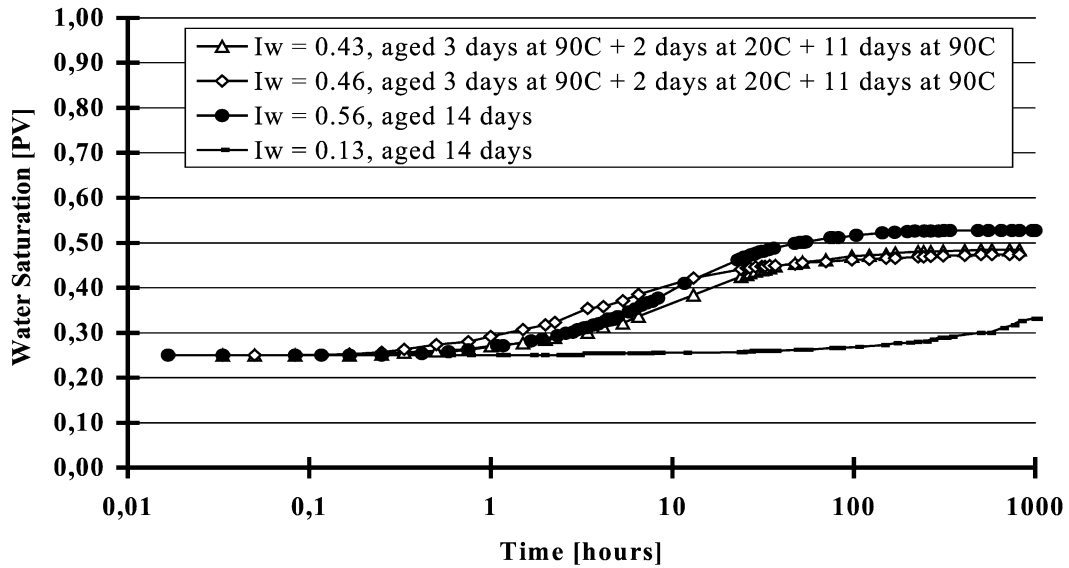
Fig. 13. Imbibition characteristics for two duplicate sets of core plugs aged for 14 days. One set was aged for 14 days submerged in crude oil, the other set cooled to room temperature after 3 days of aging, kept cool for 2 days before reheated and then continued the aging for another 11 days.

causes a significant more efficient aging process. The lower S_{wi} was obtained by using a viscous mineral oil that was exchanged by decalin and then crude oil. The use of this mineral oil did not seem to reduce the wettability alteration efficiency of the crude oil. A more extensive study is underway to explore the effect
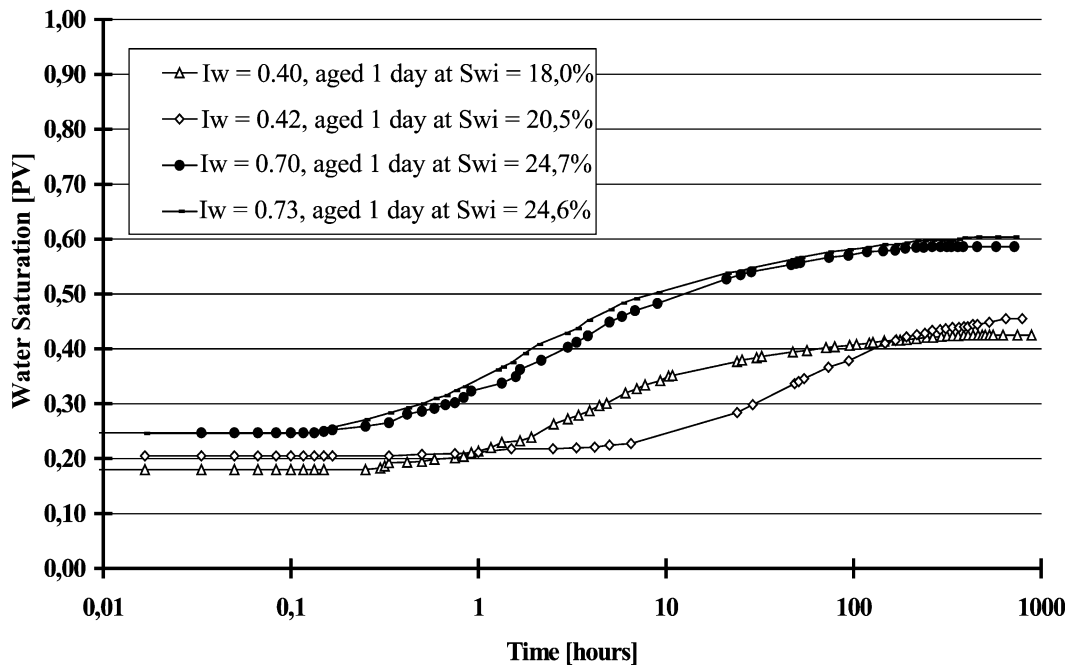

Fig. 14. Effect on the wettability alteration process from initial water saturation reflected by the spontaneous imbibition characteristics.
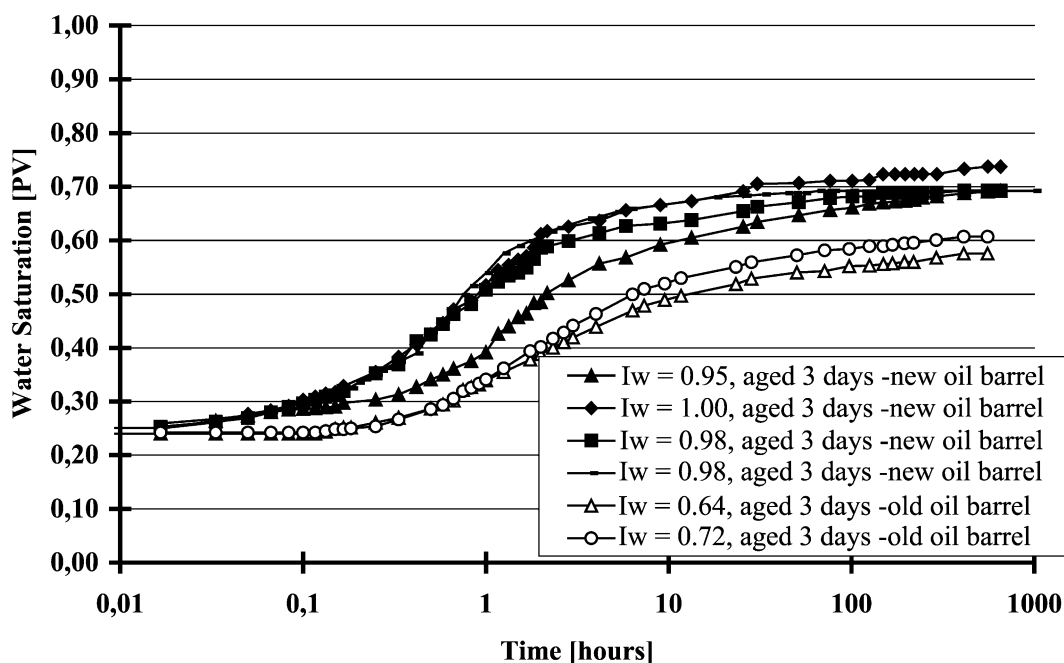
Fig. 15. Effect on the aging process by slightly different oils reflected by the spontaneous imbibition characteristics.

on aging from different initial water saturations. Initial water saturations can affect imbibition characteristics (Viksund et al., 1998).

Fig. 15 shows that the aging process is very sensitive to the crude oil used. Two barrels of crude oil were originally obtained from the same untreated well in a North Sea Chalk reservoir. Oil from one barrel has been used in our experiments over the past 5 years. The other barrel had been kept closed. Recently, we started to use oil from the second barrel. Fig. 15 shows the difference in brine imbibition characteristics after similar aging using oil from the two barrels. The results show that the oil from the second barrel is less efficient in altering the wettability. We speculate that the concentration of heavier components, which is believed to be more important for altering the wettability, is higher in the barrel used over the last 5 years. The lighter ends may have evaporated or have been gravity-segregated and thus tapped from the barrel over the years. However, each oil yields consistent and reproducible wettability alteration.

The results from the reported experiments and from other sensitivity studies of wettability alteration have given us confidence in how to prepare a rock material for special core analysis. This experience also assists in making correct interpretations of our experimental and numerical efforts to determine oil recovery mechanisms at different wettabilities and to improve the understanding of fluid flow in porous media.

4. Conclusions

1. Wettability alteration by submerging core plugs in crude oil and aging for long times may cause heterogeneous wettability conditions.
2. Wettability alteration during aging in crude oil is more efficient if crude oil is flushed through the core material during the aging process.
3. Nonuniform wettability conditions are created if flushing is unidirectional.
4. The most uniform wettability condition is obtained if the core material is flushed with crude oil during aging and the flood direction is reversed several times during the aging process.
5. Wettability alteration by aging in crude oil is more efficient at lower initial water saturations.

6. Wettability alteration by aging in crude oil is very sensitive to the oil composition.
7. Including wettability heterogeneities in numerical simulation of waterfloods in larger physical reservoir models improved the history match and the interpretation of the oil recovery mechanisms.

Nomenclature

I	Amott wettability index
k	permeability [md]
S	saturation
t	time [days]
wt.	weight [%]
OOIP	original oil in place [%]
CC	chalk core
CO	crude oil

Greek

ϕ	porosity [%]

Subscripts

a	aged
d	displacement
f	final
i	initial
im	imbibition
o	oil
w	water

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