

CRUDE OIL COMPOSITION AND THE STABILITY OF MIXED WETTABILITY IN SANDSTONES

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ABSTRACT

The effect of crude oil composition on wettability alteration at smooth quartz surfaces is compared to alteration of the wetting properties of sandstones. In both types of study, after adsorption at elevated temperature, the mineral surface was washed or flushed with a solvent to leave an adsorbed organic film. Cores prepared in this way have mixed wettability and are referred to as MXW-F cores. Wetting studies were then performed using an aqueous phase and mineral oil as the oleic phase. Previously reported contact angle measurements at smooth quartz surfaces for ten crude oils showed that asphaltic crudes gave stable advancing and receding contact angles whereas for paraffinic oils wetting was unstable. Instability was evidenced by decrease in contact angle hysteresis for repeated measurements of advancing and receding contact angles.

In the present work two of the ten crude oils, one asphaltic (Minnelusa) and one paraffinic (Gulfaks), were used to prepare sandstone MXW-F cores. Changes in wettability were assessed by rate and extent of spontaneous imbibition for recovery of mineral oils of different viscosities. Results for the MXW-F cores treated with Minnelusa oil were consistent with previously reported MXW-F results for a Prudhoe Bay (A95) crude oil of comparable acid and base numbers and asphaltene content. For A95 crude oil, imbibition rates were slightly lower for the first measured imbibition curve than for those measured subsequently. For Minnelusa oil, the first and subsequently measured imbibition curves were in closer agreement. Close reproducibility of sequential spontaneous imbibition measurements provides a measure of stability for induced MXW-F wetting states. Scaling of recovery of mineral oils by the geometric mean viscosity of oil and water provided further indication of reproducible wetting states for the two asphaltic crude oils. For the Gulfaks oil, erratic variation in imbibition behavior of MXW-F cores treated with Gulfaks oil indicated that established wetting states were not reproducible.

Keywords: mixed-wettability, asphaltic, paraffinic, crude oil, stable/unstable wetting, viscosity.

INTRODUCTION

It is widely accepted that the wettability of reservoir rocks controls the oil/water distribution and can have a major impact on oil recovery, especially in fractured reservoirs. Crude oil composition is a controlling factor in the wettability of reservoir rock (Akhlaq et al., 1996). Al-Maamari and Buckley (2000) raised the question of the

stability of wettability of reservoirs changes during the course of oil production. If so, how and why does it change? The stability of wettability states generated under laboratory conditions is also a key question with respect to wettability research. The complexity of wetting states that result from crude oil/brine interactions with mineral surfaces is well recognized. Wettability states induced will be described as reproducible if the results given by duplicate experiments (i.e. separate cores from a homogeneous rock) are essentially the same. Wettability states that give reproducible results for repeated cycles of measurement will be described as stable.

The effect of crude oil composition on wettability was also studied on smooth surfaces (Buckley et al., 1997; Liu and Buckley, 2000). Xie et al. (2002) investigated the wetting behavior of quartz surfaces after aging in 10 different crude oils at elevated temperature. The plates were lightly rinsed with toluene to leave an adsorbed organic film on the mineral surface. Contact angles of treated plate/brine/decane were measured by dynamic Wilhelmy plate technique. Contact angle measurements for the 10 crude oils showed that asphaltic crudes gave stable wetting as judged from cyclic measurements of advancing and receding contact angles. The wetting states obtained by adsorption from paraffinic oils were unstable as evidenced by systematic decrease in contact angle hysteresis. The acid and base numbers and asphaltene contents of the related oils are included in Fig. 1a (Xie, et al., 2002). Examples of reproducible contact angle data for three crude oils of relevance to the present work are presented in Figs. 1b, 1c, and 1d. The contact angle data shown in Figs. 1b and 1c indicate stable wetting states. The results in Fig. 1d indicate a series of unstable wetting states.

The stability of wetting states obtained after alteration of strongly water-wet sandstone cores by an asphaltic crude oil from Prudhoe Bay, A95, was investigated by Tong et al. (2002). Reproducible mixed-wetting was achieved by aging the Berea sandstone cores, at initial water saturation, S_{wi} , in A95 crude oil at elevated temperature, removal of crude oil by flushing with decalin and then replacing the decalin by mineral oil. The objective of displacing the crude oil with decalin is to remove the bulk oil but to leave in place an organic film of the adsorbed polar components on the rock surface. This procedure is used to avoid precipitation of asphaltenes and associated effects that might result from direct displacement of crude oil by mineral oil (Morrow, et al., 1986; Graue, 1999; Yang, et al., 1999; Tong, et al., 2002). Mixed wettability cores prepared in this way are referred to as having MXW-F wettability.

In the present work, the reproducibility and stability of MXW-F wetting induced in sandstone by an asphaltic crude oil (Minnelusa) and a paraffinic crude oil (Gullfaks) are compared with the results for A95, an asphaltic oil of comparable base numbers, asphaltene content, and viscosity to that of Minnelusa crude oil. Factors investigated included variation in the viscosity of the mineral oil and the effect of further aging of cores with crude oil.

EXPERIMENTAL

Material

Cores

Cores cut from Berea sandstone blocks were nominally 3.8 cm in diameter and 7.6 cm in length. The air permeabilities of the cores ranged from 80 to 106 md, and the porosities were all within $18.3 \pm 0.3\%$. Core properties are listed in Tables 1 and 2.

Crude oil

Two crude oils were used in this study. One was an asphaltic crude oil from the Gibbs field (Minnelusa formation) of Wyoming, designated as Minnelusa oil. The other was from the North Sea Gullfaks field, designated as Gullfaks oil. Selected properties of these oils and that of A95 (Tong et al., 2002) are listed in Table 3.

Mineral oil

Mineral oils with different viscosities were prepared by mixing Soltrol 220 mineral oil (3.8 cp) and white mineral oil (180.0 cp) in ratios selected to give intermediate viscosities as required. The mineral oils were cleaned by exposure to silica gel and alumina. Viscosities of the mixtures are given in Tables 1 and 2.

Procedure

Establishing initial water saturation prior to aging

Initial water saturations were established with synthetic reservoir brine or sea water (Table 4). NaN_3 (0.10 g/L) was added as a biocide in the brine. The core samples were first saturated with brine and soaked for at least 10 days to attain ionic equilibrium. S_{wi} of about 25% was established by displacing reservoir brine with Minnelusa crude oil at 45°C or Gullfaks crude oil at 40°C at 0.2 ml/min to 5.0 ml/min (about 0.72 to 18.75 PV/hr) (see Tables 1 and 2).

Aging and Replacement of Crude Oil with Mineral Oil

The cores containing crude oil at S_{wi} were submerged in crude oil in sealed pressure vessels for 10 days at 75°C (T_a). After aging, the crude oil was displaced by 5 PV of decalin at 3 ft/day (about 0.72 PV/hr). The displacement temperature was 50°C for Minnelusa treated cores and 45°C for Gullfaks treated cores. Decalin was then displaced with 5 PV of mineral oil of selected viscosity at ambient temperature.

1st and Subsequent Spontaneous Imbibition

The core samples containing initial water saturation and mineral oil of the selected viscosity were set in glass imbibition cells filled with synthetic brine. Oil volume produced by imbibition of brine, expressed as percentage of original oil in place (%OOIP), versus time was recorded. All of the imbibition tests were performed at ambient temperature.

For sequential imbibition tests, brine was displaced by mineral oil at room temperature to re-establish initial water saturation. The displacement rate was 0.1 to 6.0 ml/min (about 0.36 to 22.5 PV/hr) according to the viscosity of the mineral oil and the S_{wi} . The water production was monitored during the displacement until the original water saturation had been reestablished. For some low viscosity mineral oils (less than 80 cp, see Tables 1 and 2), the desired initial water saturation was achieved by flushing the core sample with 170 cp white mineral oil followed by flushing with mineral oil of the desired viscosity for the imbibition test. Up to four sequential imbibition tests were run for each core. After the 3rd imbibition, two cores were displaced first with 5 PV of decalin and then with 5 to 12 PV of Minnelusa crude oil, followed by aging in Minnelusa oil for 10 days at 75°C. A 4th imbibition test was then run after decalin/mineral oil displacement.

RESULTS AND DISCUSSION

Wettability of MXW-F cores was assessed by comparing results scaled by the following group (Ma, et al., 1997):

$$t_D = t \sqrt{\frac{k}{\phi}} \frac{\sigma}{\sqrt{\mu_o \mu_w}} \frac{1}{L_c^2} \quad (1)$$

where t_D is dimensionless time, t is time, k is permeability, ϕ is porosity, σ is the interfacial tension, μ_o and μ_w are the oil and brine viscosities. L_c is a characteristic length that compensates for sample size, shape and boundary conditions.

Core Samples Aged with Minnelusa Crude Oil

1st Spontaneous Imbibition

For Minnelusa MXW-F cores, mineral oil with viscosities of 3.8, 41.4, 83.8 and 172.6 cp were used in the imbibition tests. The 1st imbibition curves for recovery vs time are shown in Fig. 2a. The imbibition rate curves were correlated satisfactorily by Eq.1 as shown in Fig. 2b. This indicates that, as found for A95 MXW-F cores, the viscosity of the mineral oil did not affect the wettability. The average of the correlated 1st imbibition data for MXW-F (A95) cores (Tong, et al., 2002) falls close to the Minnelusa MXW-F data set. This correspondence is probably related to the similarity in chemical characteristics of the two oils (see Fig.1a). The higher advancing contact angles for Minnelusa vs A95 (cf Figs.1b and 1c) measured on smooth quartz are only weakly reflected by the imbibition results.

Subsequent Spontaneous Imbibition

The scaled results for 2nd spontaneous imbibition by MXW-F (Minnelusa) cores are generally close to and slightly higher than the average of the 1st imbibition curves (dashed line in Fig. 3a). For core 5B3, of imbibition rate fell below that measured for 1st imbibition; this does not fit the usual pattern of behavior for 1st and 2nd imbibition. Scaled rate of recovery for 172.6 cp oil (see core 5B4) was somewhat higher than the average given by the semi-empirical scaling group (Eq.1). 3rd imbibition results, except

for core 5B5, also fell very close to the averages for the 1st and 2nd cycles (Fig.3b). Results for 4th imbibition tests for Core 5B7 were also very close to the average for the previous measurements (Fig.3b).

Two examples of sequential imbibition measurements, drawn from the results presented in Figs. 2 and 3, are shown in Fig.4. Core 5B11 exhibited stable wetting with close correlation of the 1st, 2nd and 3rd imbibition recovery curves, even when the mineral oil viscosity was changed from 172.6 cp for the 1st cycle to 3.8 cp for the following 2nd, 3rd cycles (Fig.4a). Stable wetting was also indicated by reproducibility of recovery of 40 cp mineral oil from core 5B5 (Fig.4b). Thus, in general, the sequential imbibition results were in close agreement showing that the wettability of the MXW-F Minnelusa cores was stable. (The A95 MXW-F cores showed shift between 1st and 2nd imbibition but were subsequently stable.)

Reaging with Minnelusa Crude Oil

After the third imbibition test on these cores, S_{wi} was re-established with 170 cp mineral oil. The mineral oil was then displaced from the cores by flooding with 5 PV of decalin followed by 5 PV of Minnelusa crude oil. The cores were then re-aged in the Minnelusa oil for 10 days at 75°C. After aging, the crude oil was displaced by 5 PV decalin which was in turn displaced by 3.8 cp mineral oil for 5B11 and 40 cp mineral oil for core 5B5. In both cases imbibition rates decreased slightly as a result of further aging (Figs. 4a and 4b), indicating less water-wetness and further adsorption of polar components at the rock pore surfaces.

Core Samples Aged with Gullfaks Crude Oil

Reproducibility of 1st Spontaneous Imbibition

Initial water saturations established with Gullfaks crude oil were all close to 26%. In all cases the rate and extent of recovery by imbibition was higher than for either Minnelusa or A95 oil. However, for MXW-F (Gullfaks) cores problems were experienced with reproducibility of 1st imbibition curves for duplicate core plugs. Scaled results for 3 duplicate tests with 3.8 cp mineral oil, 4 with 19.8 cp mineral oil and 1 with 9.3 cp mineral oil are presented in Fig.5a. Recoveries after about 27 days of imbibition were comparable, but 3 of the recovery curves are well separated from the other 5 with respect to rate. Scaled results for duplicate core plugs using 38.7 and 83.8 cp mineral oil and 1 curve for 172.6 cp mineral oil are presented in Fig. 5b. The MXW-F (Gullfaks) cores that gave similar slow rates of imbibition will be referred to as Group 1 (5B13, 5B15 and 5B18 three cores, see Fig.5a) and the remainder as Group 2 (9 cores, see Figs. 5a and 5b). Examination of rock properties, and the batches and dates of core preparation and testing gave no indication as to why the results fell into 2 groups. For the 9 plugs which exhibit higher recovery rates the scaled results are shown in Figs. 5a and 5b, recoveries at long times are comparable, but there is still considerable scatter in the rates of imbibition.

Stability of MXW-F (Gulfaks) wetting

The stability of wetting was tested for two cores from each of Groups 1 and 2 through measurement of three sequential spontaneous imbibition curves. Scaled results for the two Group 1 cores (5B13, 5B18) are shown in Figs. 6a and 6b. Results for recovery of 3.8 cp and 19.8 cp mineral oil were very similar in form. For the 2nd and 3rd cycle imbibition, rates of recovery were higher, but final recoveries were lower. Both cores exhibited the same rate of recovery at early time but decreased recovery for the 3rd imbibition.

Imbibition curves for the two Group 2 cores 5B16 and 5B29 are plotted in Figs. 6c and 6d. Rates of imbibition increased for the 2nd and 3rd imbibition but there was essentially no change in final recovery. The 2nd and 3rd imbibition were closely reproduced for Core 5B29, indicating attainment of a stable wetting state. Overall, although the MXW-F (Gulfaks) wetting states were not reproducible (i.e. varied between duplicate cores). Particular achieved wetting states, as indicated by imbibition measurements, showed partial reproducibility between 2nd and 3rd imbibition (Figs. 6a-d).

Comparison of Spontaneous Imbibition with Contact Angles

As for contact angle behavior at smooth quartz surfaces (Fig.1), the MXW-F (Gulfaks) wetting states are clearly more water-wet than those induced by the two asphaltic crude oils with relatively high base numbers. Reproducibility of sequentially measured contact angles was observed for the asphaltic oils (Figs. 1b and 1c). Minnelusa oil gave the most stable wetting behavior as indicated by sequential imbibition measurements (Figs.2, 3, and 4) and A95 oil results indicated stable wetting after completion of the 1st imbibition (Tong, et al., 2002). During the course of imbibition, a slight amount of dark material accumulated at the mineral oil/brine interface in the imbibition cells for A95 oil but not for Minnelusa. Also, the mode of precipitation of solids in response to addition of alkane for A95 (Wang, 2000) was different to that for the Minnelusa oil (Wang, 2002).

Compared to results for the cores treated with asphaltic crude oils, less change in wetting was induced by Gulfaks crude oil. However, the sequential decrease in advancing contact angles (see Fig. 1d) was not matched by a clearly sequential increase in water wetness for consecutive spontaneous imbibition tests. In addition to the difference in brine composition, there are many other reasons why contact angle measurements at smooth surfaces are not likely to provide reliable prediction of core displacement behavior. These include differences in mineralogy and pore morphology and the response of adsorbed organic films to movement of oil-water interfaces and the differences in time (less than one day for contact angle measurements) versus many months for sequential imbibition tests.

CONCLUSIONS

- 1) In interpretation of the imbibition behavior of MXW-F cores, distinction needs to be made between reproducibility of wetting for duplicate core plugs, and the stability of wetting for consecutive imbibition tests.
- 2) Reproducible and stable wetting was exhibited by films deposited from Minnelusa oil. Crude oils which provide stable films are particularly useful for oil recovery research.
- 3) Imbibition results for recovery of mineral oils of different viscosity from MXW (Minnelusa cores) were scaled by the geometric mean of the oil and water viscosities.
- 4) A paraffinic oil (Gulfaks) which gave reproducible but unstable contact angle behavior, gave poorly reproducible MXW-F wetting, but any particular achieved wettability was to some degree stable.
- 5) From imbibition measurements, the paraffinic oil (Gulfaks) caused less change in wetting than the asphaltic Minnelusa crude oil and a previously studied asphaltic oil. This observation is qualitatively consistent with advancing contact angle measurements on smooth quartz after deposition of organic films from these oils. However, such contact angle measurements are not reliable for prediction of imbibition rates.

NOMENCLATURE

k	gas permeability, md	T_a	aging temperature
L_c	characteristic length, cm	T_m	imbibition test temperature
S_{wi}	initial water saturation, %	T_f	decalin flush temperature
t	imbibition time, min	ϕ	porosity, %
t_D	dimensionless imbibition time	σ	oil-water interfacial tension, dynes/cm.
		μ_w	water viscosity, cp,
		μ_o	oil viscosity, cp.

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Core#	K_g , md	ϕ , %	L_c , cm	μ_o , cp	ρ_o , g/ml	S_{wi} , %	2nd cycle*	3rd cycle*	4th cycle*
5B3	97.9	18.0	1.26	3.8	0.7819	25.0	3.8 cp, 23.3 PV	170 cp, 2.5 PV	—
5B32	94.3	18.5	1.26	3.8	0.7819	25.7	—	—	—
5B5	105.6	18.4	1.27	40.0	0.8456	24.5	40.0 cp, 5.5 PV	40.0 cp, 5.1 PV	170 cp, 2 PV
5B1	87.0	17.7	1.26	83.8	0.8168	24.9	84 cp, 7.5 PV	84 cp, 7.0 PV	84 cp, 6 PV
5B7	104.9	18.3	1.26	83.8	0.8168	24.6	84 cp, 5.7 PV	84 cp, 6.6 PV	84 cp, 5.7 PV
5B4	102.6	18.4	1.27	172.6	0.8745	24.9	173 cp, 6.9 PV	173 cp, 6.6 PV	173 cp, 5.5 PV
5B11	79.1	17.6	1.27	172.6	0.8745	24.8	173 cp, 4.4 PV	173 cp, 4.0 PV	173 cp, 4.0 PV

*The viscosity and pore volume of the mineral oil used to establish S_{wi} .

Core#	K_g , md	ϕ , %	L_c , cm	μ_o , cp	ρ_o , g/ml	S_{wi} , %	2nd cycle*	3rd, cycle*
5B13	92.1	18.2	1.26	3.8	0.7819	25.8	170 cp, 11 PV	170 cp, 7.8 PV
5B21	79.4	17.7	1.26	3.8	0.7819	25.9	—	—
5B28	99.5	18.7	1.27	3.8	0.7819	25.8	—	—
5B20	88.0	18	1.26	9.3	0.8079	25.6	—	—
5B15	89.1	18.2	1.26	17.4	0.8240	25.2	—	—
5B18	79.5	17.7	1.26	19.8	0.8280	25.8	170 cp, 15 PV	170 cp, 9.4 PV
5B23	81.3	17.7	1.26	19.8	0.8280	25.8	—	—
5B25	106.1	18.6	1.27	19.8	0.8280	26.2	—	—
5B16	90.8	18.1	1.26	38.7	0.8420	25.7	170 cp, 1.8 PV	170 cp, 3.3 PV
5B30	109.9	18.8	1.26	38.7	0.8420	25.9	—	—
5B17	88.9	18	1.27	83.8	0.8168	26.0	—	—
5B29	97.7	18.5	1.26	83.8	0.8168	25.9	—	—
5B14	78.7	17.7	1.26	172.6	0.8745	25.8	—	—

* The viscosity and pore volume of the mineral oil used to establish S_{wi} .

Oil	Type	Density, g/ml	μ_o at 22°C, cp	n-C7 asphalt., wt%	Acid #, mg KOH/g oil	Base #, mg KOH/g oil
Minnelusa	asphaltic	0.9062	77.2	9.0	0.17	2.29
A95	asphaltic	0.9086	70.9	8.7	0.24	2.20
Gulfaks	paraffinic	0.8803	18.6	0.4	0.24	1.19

Brine	NaCl (g/L)	KCl (g/L)	CaCl ₂ (g/L)	MgCl ₂ (g/L)	MgSO ₄ (g/L)	Na ₂ SO ₄ (g/L)	NaN ₃ (g/L)	pH	TDS (mg/L)
Minnelusa	29.8	0	2.1	0	0.394	5.903	0.1	6.8	38297
Gulfaks	28	0.935	1.19	5.365	0	0	0.1	6.6	35590

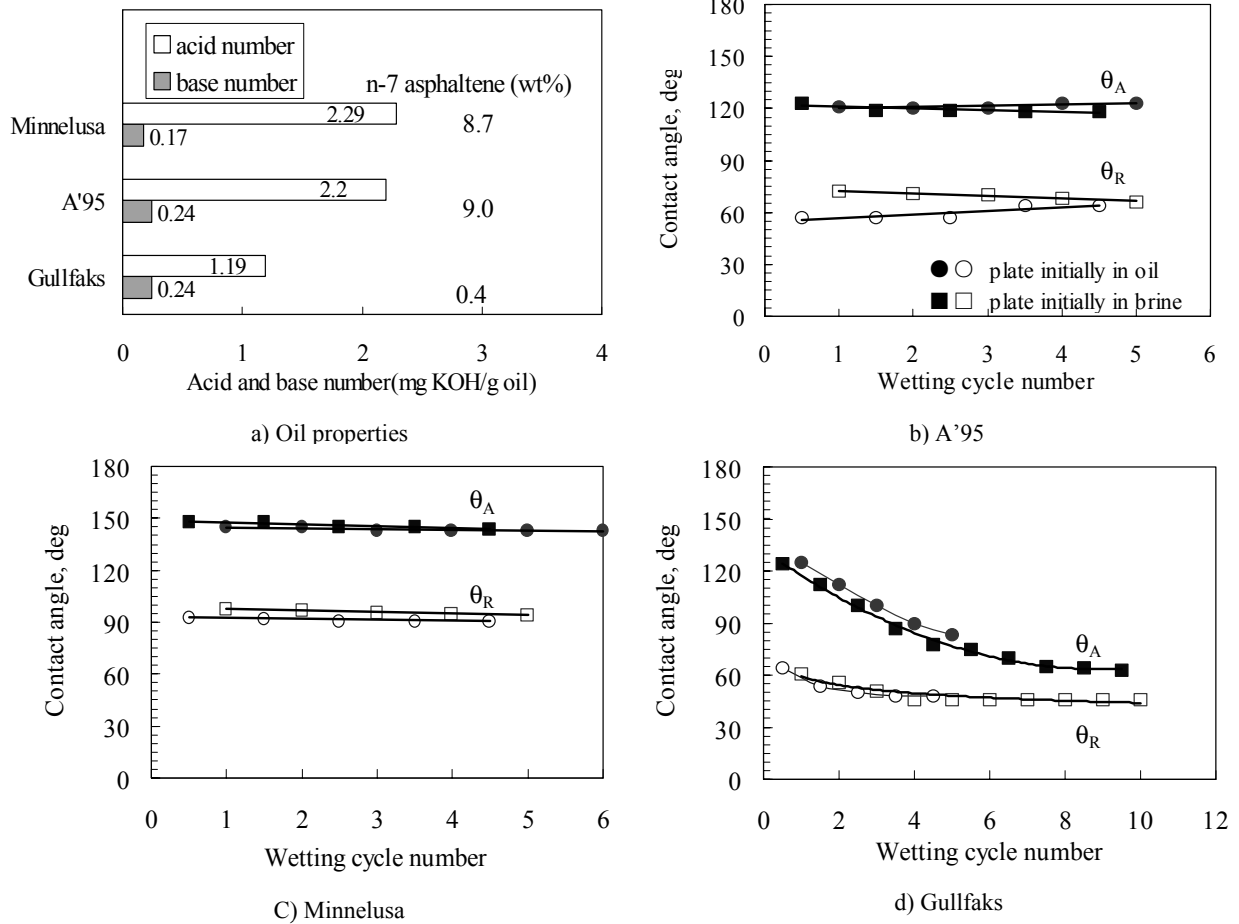


Fig.1 Properties of crude oil and contact angles after deposition of organic films on smooth quartz surfaces (Xie, et al, 2002). Advancing (θ_A) and receding (θ_R) contact angle results shown in Fig. 1b and 1c obtained from asphaltic crude oil are characterized as reproducible and stable. Results in Fig.1d obtained from non-asphaltic crude oil are reproducible but unstable.

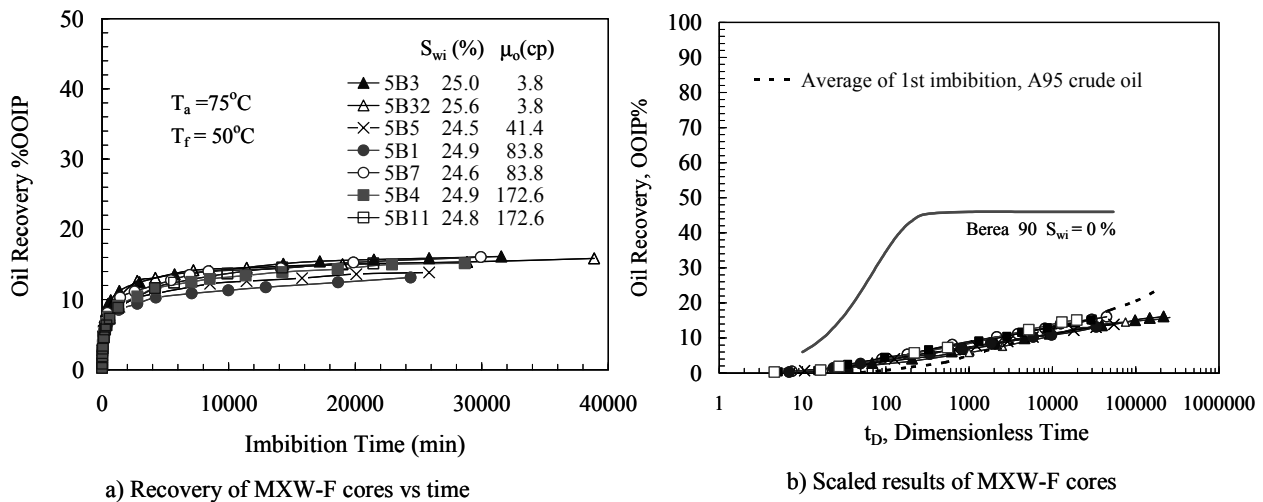


Fig. 2 Primary imbibition recovery and scaled results for MXW-F (Minnelusa) cores

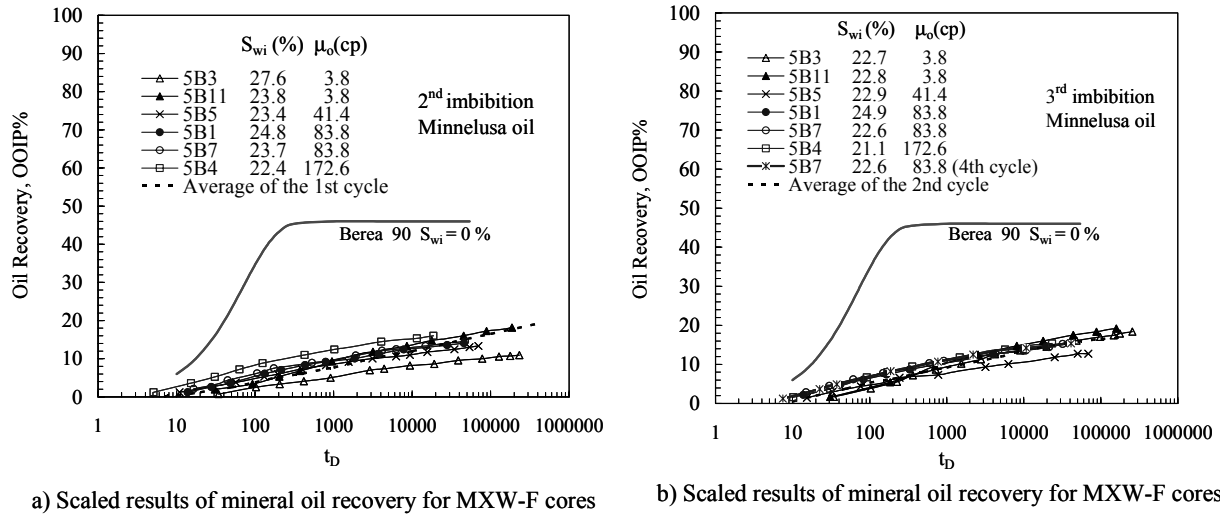


Fig. 3 Scaled subsequent spontaneous imbibition results of MXW-F (Minnelusa) cores.

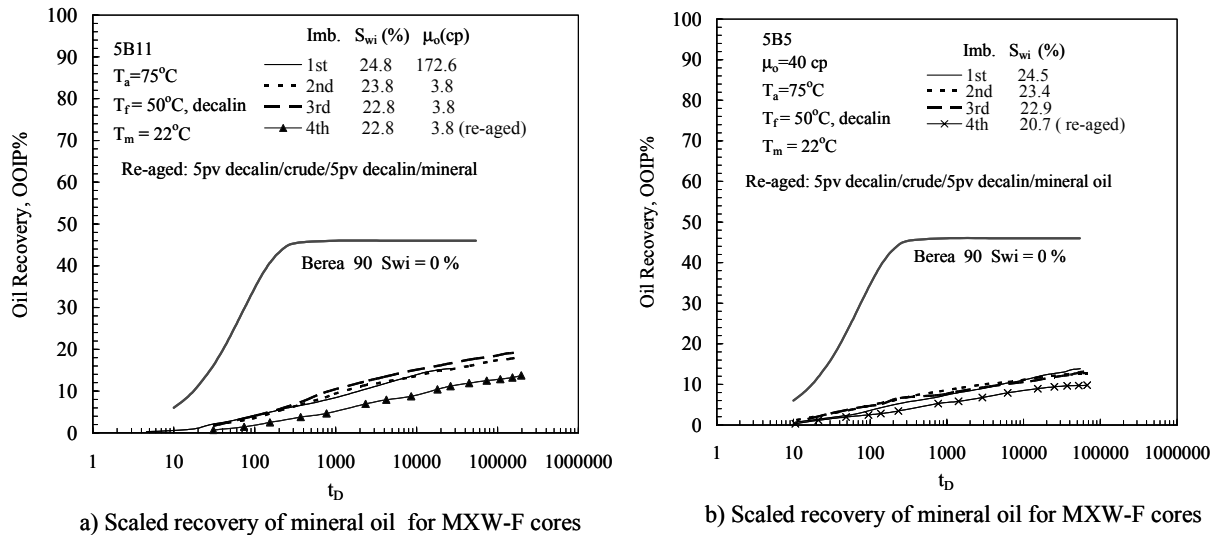


Fig. 4 The effect of Minnelusa oil re-aging on spontaneous imbibition.

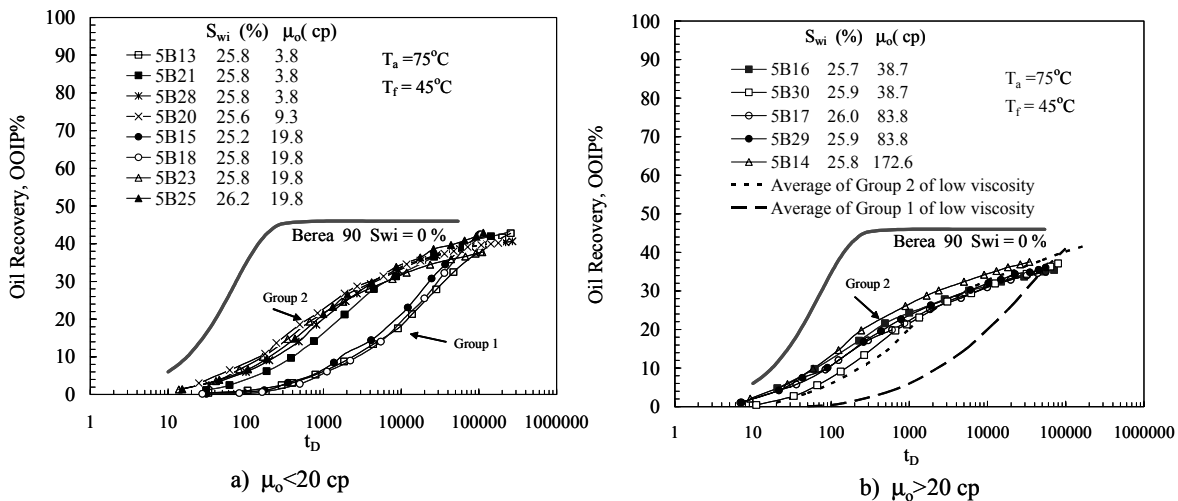


Fig. 5 Scaled 1st imbibition results for MXW-F (Gullfaks) cores.

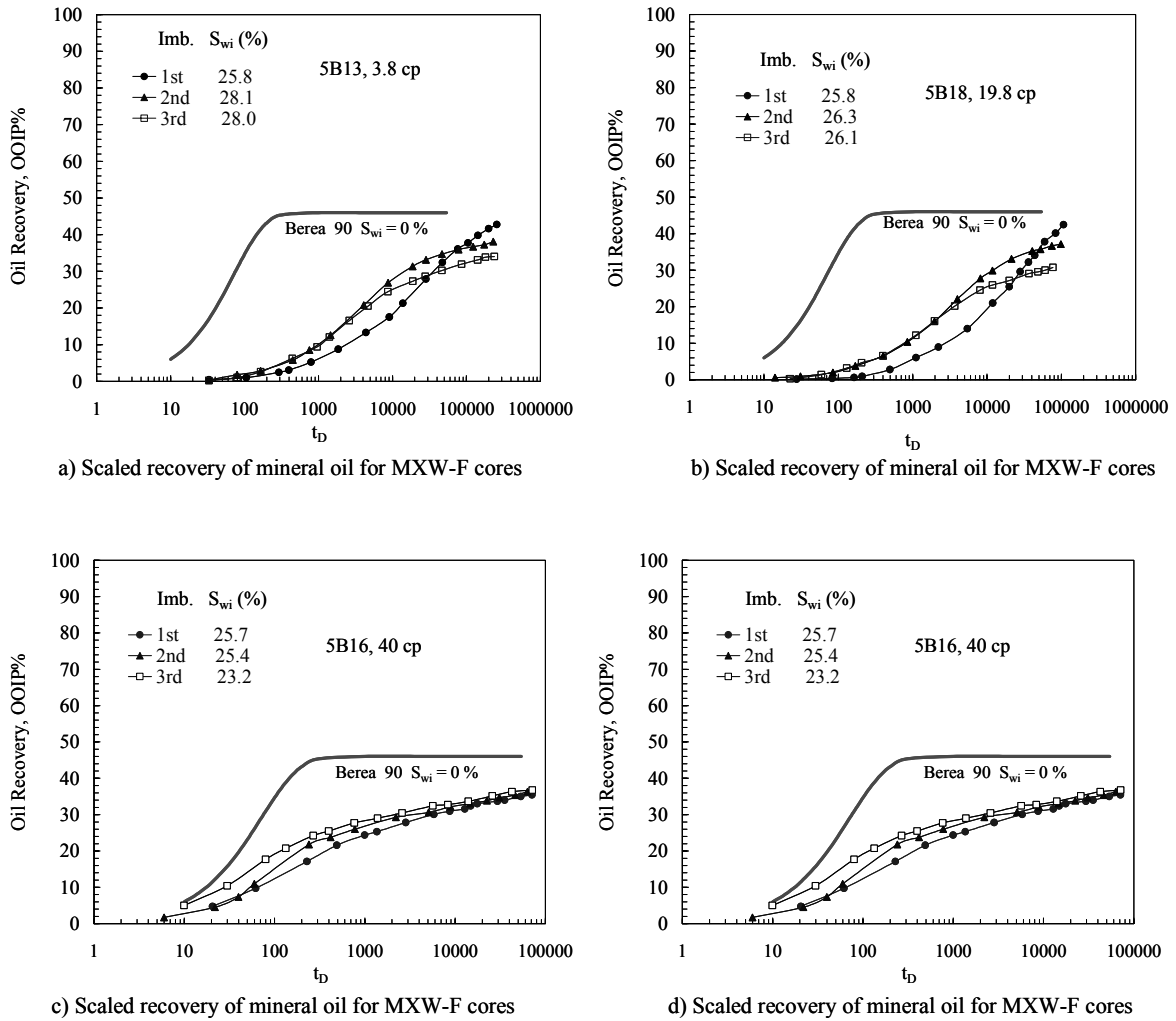


Fig. 6 Sequential spontaneous imbibition tests for MXW-F (Gullfaks) cores.