A SYSTEMATIC INVESTIGATION OF WETTING STABILITY IN AGED CHALK

M. Steinsbø, A. Graue and M.A. Fernø
Department of Physics and Technology, University of Bergen, NORWAY

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ABSTRACT
Waterflood performance of fractured chalk reservoirs are strongly influenced by wettability, and spontaneous imbibition of water from fractures to matrix is a key mechanism for oil production. To determine the enhanced oil recovery (EOR) potential for induced wettability change, for instance during a surfactant injection, stable and uniform wetting preferences representing reservoir conditions must be established during core analysis to avoid laboratory artifacts. A systematic investigation of wettability stability in laboratory aged core plugs was performed by repeated cycles of spontaneous and forced imbibitions. Altered wetting states, ranging from strongly water-wet to moderately water-wet conditions were established by aging outcrop Rørdal chalk samples in crude oil at 80°C for 2 to 6 days at a range of initial water saturations during aging. The Amott-Harvey water index (I_w) was found stable over two cycles of spontaneous imbibition tests, with an average change of $I_w \pm 0.04$ between the first and second cycle. The oil recovery rate in partly waterflooded regions and transition zones was evaluated by varying the initial water saturation.

INTRODUCTION
Residual oil saturation in fractured chalk reservoirs, after primary and secondary water or gas injections, is often high because large areas may be unswept due to fingering, or bypassed due to fluid flow preference along fractures, and gravity segregation. It is well known that the wettability preference of a reservoir is a major factor for oil recovery because it controls the location of fluids in the pore space and the interaction of the driving forces and thus determining the ultimate recovery [1]. The wettability of a rock surface is determined by the presence of water and/or oil layer [2]. To alter the wettability preference during laboratory core analysis, in particular when working with outcrop analogue rocks, an aging process is often used to allow polar components from the crude oil to be absorbed on the rock surface. The established wetting preference depends on aging time, initial water saturation during aging, temperature and the crude oil/rock/brine system, and may reflect different reservoir wetting conditions ranging from strongly water-wet to near neutral-wet.

A dynamic aging method, capable of establishing uniform wetting conditions by continuously flowing crude oil through core samples [3, 4], was used to establish different wetting preferences, representing typical wetting conditions for partly waterflooded areas, transition zones or residual oil zones (ROZ). 12 originally strongly water-wet outcrop chalk core plugs (6 pairs of 2 similar core plugs) resembling a North Sea reservoir were aged with North Sea crude oil at 80°C at six different initial conditions varying both the aging time and the initial water saturation; *short* (2 days) and *long* (6 days) aging, and, at three different initial water saturations; *low*, *medium* and *high*. The initial water saturation ranged from S_w=0.23 to S_w=0.35 and the measured Amott-

Harvey water index [5] ranged from I_w=0.39 to I_w=0.80. Repeated drainage, spontaneous and forced imbibition tests were performed at each core plug at varying initial water saturations ranging from S_w=0.23 to S_w=0.38.

EXPERIMENTAL

Rock and fluids

Rørdal chalk [6, 7] core plugs were drilled from larger slabs of outcrop rocks obtained from the Portland cement factory at Ålborg, Denmark. The rock formation is of Maastrichtian age and consists mainly of coccolith deposits, and the composition is calcite (99%) with some quartz (1%). The rock is homogeneous, porosity and permeability range from 45-47% and 3-8mD, respectively. The synthetic brine composition was: 5% NaCl, 5% $CaCl_2$, 0.05% NaN_3 and the density and viscosity were: ρ=1.05g/cm^3, μ=1.09cP. A North Sea crude oil and two mineral oils, decahydronaphthalene and n-Decane were used in the experiments; the fluid properties are listed in **Table 1**. A summary of crude oil chemical analysis including SARA components, API, refractive index, and acid and base numbers is tabulated in **Table 2**.

Establishing different wettability conditions

The core plugs were cleaned and dried at 80°C and thereafter saturated with synthetic brine under vacuum. Porosity was determined from weight measurements. Permeability to brine was calculated using Darcy's law. **Table 3** lists basic properties and permeability for the core plugs. Prior to the aging, the core plugs were drained with crude oil at 80°C to different initial water saturations S_w1. Four core plugs (M3, M4, M5, M6) were aged at irreducible water saturation S_{wi}~0.23 (*low* S_w), four core plugs (M7, M8, M11, M12) were aged at S_w~0.29 (*medium* S_w) and four core plugs (M9, M10, M15, M16) were aged at S_w~0.35 (*high* S_w). Six core plugs (M3, M4, M7, M8, M9, M10) were aged 6 days (*long* aging) and six core plugs (M5, M6, M11, M12, M15, M16) were aged 2 days (*short* aging). Core plugs aged at irreducible water saturation S_{wi} were oilflooded with crude oil with a constant differential pressure (2bar/cm) from both directions at 80°C. Core plugs aged at medium and high initial water saturation were oilflooded at lower differential pressure. **Figure 1** shows the experimental set-up for the draining and aging. After primary drainage, the flow of crude oil was reduced to constant injection rate 3ml/hr, continuously injecting crude oil through the core plugs during an aging period of 2 or 6 days. The direction of flow was reversed midway. After aging the crude oil was displaced from the core plugs by injecting 5PV decahydronaphthalene followed by 5PV n-Decane to avoid asphaltene precipitation, to stop the aging and to establish more reproducible experimental conditions by using the mineral oil n-Decane as the oil phase throughout the experiments.

Wettability Measurement

After aging, the core plugs at initial water saturation S_w1, saturated with brine and n-Decane were cooled to room temperature and then placed in graduated imbibition cells for spontaneous imbibition cycle number one (c#1). Produced oil as a function of time was measured by the volumetric method. Before each measurement, the imbibition cell was gently swirled to expel the oil drops adhered to the cell and core plug surface. When no more oil was spontaneously produced from the core plugs at $S_{wsp}1$, the core plugs were waterflooded with a differential pressure of 2bar/cm in a core holder with 10bar confinement pressure. End-point water saturations after both spontaneous and forced imbibition ($S_{or,WF}$) were used to calculate the Amott-Harvey water index, I_w. After waterflooding, the core plugs aged at S_{wi} were oilflooded with n-Decane at low pressure to water saturation S_w2>S_{wi}. Irreducible water saturation for the core plugs aged at $S_w1>S_{wi}$ was established after secondary drainage (2bar/cm) to $S_w2=S_{wi}$. Second cycle of spontaneous imbibition (c#2) was started at S_w2 and stopped when no more water spontaneously imbibed at $S_{wsp}2$. An overview of water saturations, calculated Amott-Harvey water indices for the two cycles and recoveries may be found in **Table 4**.

RESULTS

Spontaneous imbibition rate

As expected, a systematic increase in spontaneous imbibition rate was observed for more water-wet core plugs (aged *short* time at *high* initial water saturation) compared to less water-wet core plugs (aged *long* time at *low* initial water saturation). **Figure 2** shows development in water saturation and imbibition rate vs. time for core plugs aged 6 days at low ($M4^{L6}$), medium ($M8^{M6}$) and high ($M9^{H6}$) initial water saturation. The second imbibition cycle (not shown) exhibited slightly, but consistent, higher imbibition rates. This effect has also been observed in previous experiments and are discussed elsewhere[8].

End-point water saturation after spontaneous imbibition S_{wsp}

The end-point water saturation after spontaneous imbibition (S_{wsp}) increased with 1) higher initial water saturation during aging, and, 2) decreased aging time. For core plugs aged with different initial water saturations, there was a systematic increase in average S_{wsp} with increased initial water saturation during aging, for both 2 and 6 days: aged at low $S_w=S_{wi}$: 0.49 (2 days) and 0.46 (6 days); aged at medium S_w: 0.55 (2 days) and 0.50 (6 days); aged at high S_w: 0.60 (2 days) and 0.56 (6 days). In repeated tests, using the same core plug, variations in the initial water saturation did not change the end-point S_{wsp} (±0.02) between c#1 and c#2, i.e. the established wettability preference was stable in terms of recovery by spontaneous imbibition. **Figure 3** shows the development in water saturation vs. dimensionless time t_D [9] for two cycles of spontaneous imbibition for core plugs aged 6 days at low, medium or high initial water saturation during aging.

Amott-Harvey Water Index I_w and Residual oil saturation S_{or}

For core plugs aged 6 days the average I_w increased from 0.40 (aged at low $S_w=S_{wi}$) to 0.41 (aged at medium S_w) to 0.51 (aged at high S_w). For core plugs aged 2 days average I_w varied from 0.47 (aged at low $S_w=S_{wi}$) to 0.65 (aged at medium S_w) to 0.63 (aged at high S_w). The change in I_w between first and secondary spontaneous imbibition ranged from 0.00-0.10 and the average change was 0.04. Residual oil saturation S_{or} after forced imbibition are lower for neutral-wet core plugs compared to strongly water-wet core plugs [10]. **Figure 4** shows $S_{or,WF}$ vs. I_w for the 12 core plugs after first cycle of spontaneous and forced imbibition.

DISCUSSION

Wettability affects the fluid distributions and together with initial water saturation wettability also influences production rate and recovery for the core samples. The dynamic aging process established stable wetting conditions, in terms of recurring endpoint saturations during two cycles of drainage, spontaneous and forced imbibition in this oil/rock/brine system (saturated with mineral oil and brine), at both varying wettability preferences and varying initial water saturations. Different wettability preference was established by varying the aging time and the initial water saturation during aging in several core plugs. By repeated flooding cycles in the same core plug, with a given wettability, but at varying water saturation, typical reservoir conditions for a large transition zone, residual oil zones or a partly waterflooded area was observed in terms of recovery and production rates. Experimental studies of wettability stability, as presented in this paper, would provide valuable information in reservoir simulations or in considerations of wettability enhancing EOR-efforts, especially for partly waterflooded areas or transition zones.

Other rock materials were also investigated in terms of wettability stability. Identical experimental methods were performed for two other rock materials; Stevns chalk and Niobrara chalk. These rock types did not show consistent results, corroborating earlier experimental results [11], exhibiting inconsistent results on wetting properties with respect to temperature and injected brine composition (sulfate added). The established wettability preference in Rørdal chalk, however, was not affected by sulphate or temperature.

CONCLUSION

- A range of different, reproducible and stable wettability preferences representing different reservoir wetting conditions was established in aged outcrop Rørdal chalk by a dynamic aging process.
    1. The measured I_w decreased systematically with increased aging time and decreased initial water saturation during aging.
    2. End-point water saturations after spontaneous imbibition, at a given wettability, was not affected by varying the initial water saturation.
    3. The established I_w (ranging from 0.39 to 0.80), was stable during several flooding cycles of the core plugs saturated with brine and mineral oil.
- Aged outcrop Rørdal chalk appears to be a good candidate, as rock material, to study the effect of varying wettability preferences and varying initial water saturation on oil recovery, and also, in EOR wettability enhancing experiments. Similar wetting stability tests of Stevns chalk and Niobrara chalk showed less consistent results.

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Table 1. Oil properties

	ρ(20°C) [g/cm3]	ρ(80°C)[g/cm3]	μ(20°C) [cP]	μ(80°C) [cP]
n-Decane	0.73	0.68	0.92	0.40
Decahydronaphthalene	0.89		0.85	
Crude oil	0.85	0.85	14.3	2.7

Table 2. Crude oil analysis.

AN	BN	RI	API	Saturates [%]	Aromates [%]	Resins [%]	Asphaltenes [%]
0.41±0.02	1.4±0.1	1.4834	27±3	61±3	20±1	19±1	0.59±0.03

Table 3. Core plug characteristics.

Core	L[cm]	D[cm]	PV[ml]	Por[%]	K[mD]	$k_{ro,wi}$	$k_{rw,or}$
M3	8.02	5.09	78.8	48.2	3.12	1.03	0.42
M4	8.01	5.07	72.4	44.8	2.28	0.95	0.36
M5	8.03	5.07	74.5	46.0	3.26	0.95	0.35
M6	8.02	5.08	79.6	48.9	3.59	0.91	0.34
M7	7.99	5.07	75.1	46.6	3.49	0.73	0.48
M8	8.01	5.08	76.9	47.3	4.08	0.68	0.37
M11	7.92	5.07	75.8	47.4	3.92	0.53	0.30
M12	8.01	5.08	75.8	46.7	4.27	0.67	0.41
M9	8.02	5.07	76.0	47.0	3.67	0.53	0.23
M10	8.05	5.09	70.7	43.1	5.97	0.63	0.32
M15	8.03	5.09	76.6	46.9	3.39	0.55	0.31
M16	7.99	5.07	75.0	46.4	4.54	0.66	0.27

Table 4. Core plug data for two cycles of spontaneous and forced imbibition and drainage.

Core	S_{wi}	S_w1	S_w2	$S_{wsp}1$	$S_{wsp}2$	$1\text{-}S_{or,WF}$	I_w1	I_w2	$Rf_{wsp}1$	Rf1
$M3^{L6}$	0.23	0.23	0.34	0.45	0.44	0.80	0.39	0.37	0.29	0.74
$M4^{L6}$	0.23	0.23	0.34	0.46	0.45	0.79	0.42	0.39	0.30	0.73
$M5^{L2}$	0.21	0.21	0.33	0.46	0.42	0.76	0.46	0.38	0.32	0.70
$M6^{L2}$	0.25	0.25	0.34	0.52	0.48	0.82	0.48	0.41	0.36	0.76
$M7^{M6}$	0.28	0.28	0.28	0.47	0.46	0.80	0.35	0.35	0.26	0.73
$M8^{M6}$	0.28	0.30	0.28	0.53	0.53	0.80	0.47	0.47	0.33	0.72
$M11^{M2}$	0.31	0.31	0.31	0.67	0.59	0.76	0.80	0.62	0.52	0.65
$M12^{M2}$	0.23	0.28	0.23	0.55	0.55	0.72	0.65	0.65	0.38	0.62

$M9^{H6}$	0.31	0.36	0.31	0.59	0.57	0.84	0.54	0.50	0.37	0.74
$M10^{H6}$	0.28	0.34	0.28	0.52	0.51	0.78	0.48	0.46	0.27	0.66
$M15^{H2}$	0.32	0.38	0.32	0.62	0.60	0.78	0.66	0.61	0.39	0.64
$M16^{H2}$	0.28	0.33	0.28	0.58	0.53	0.77	0.60	0.50	0.36	0.66

Superscript ^{L,M,H} = aged at low (~0.23), medium (~0.29) or high (~0.34) initial S_w.
Superscript ^{6,2} = aged 6 or 2 days.

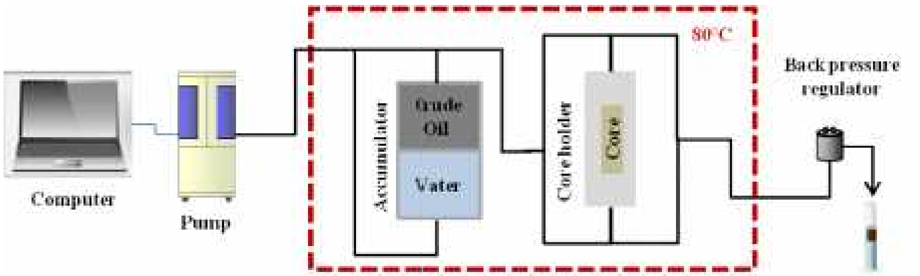

Figure 1. Experimental set-up for drainage and aging. Fluids were injected from a pump (crude oil from an accumulator) through the core plug confined inside a standard core holder with rubber sleeve. 2-way-valves allowed for injection from both directions. Material balance was acquired at ambient conditions.

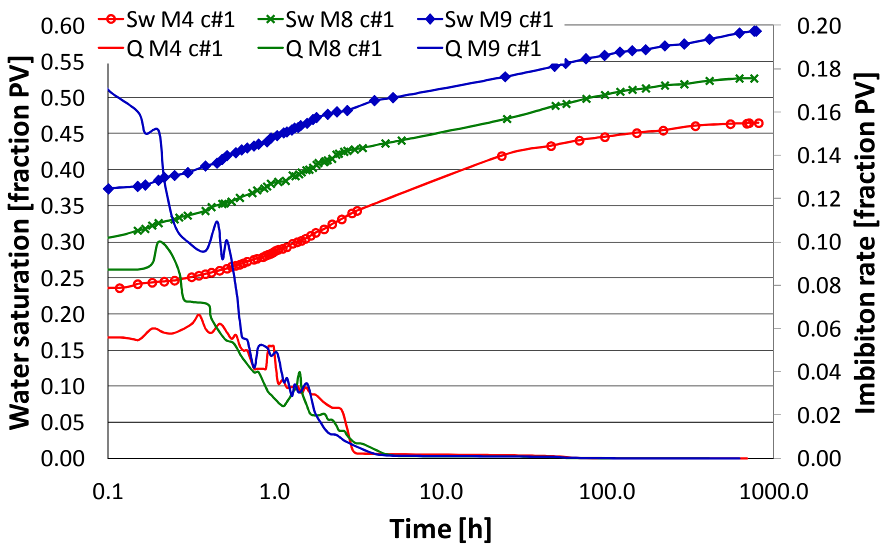

Figure 2. Development in water saturation S_w and imbibition rate Q vs. time for core plugs aged 6 days (c#1) at low ($M4^{L6}$), medium ($M8^{M6}$) and high ($M9^{H6}$) initial S_w.

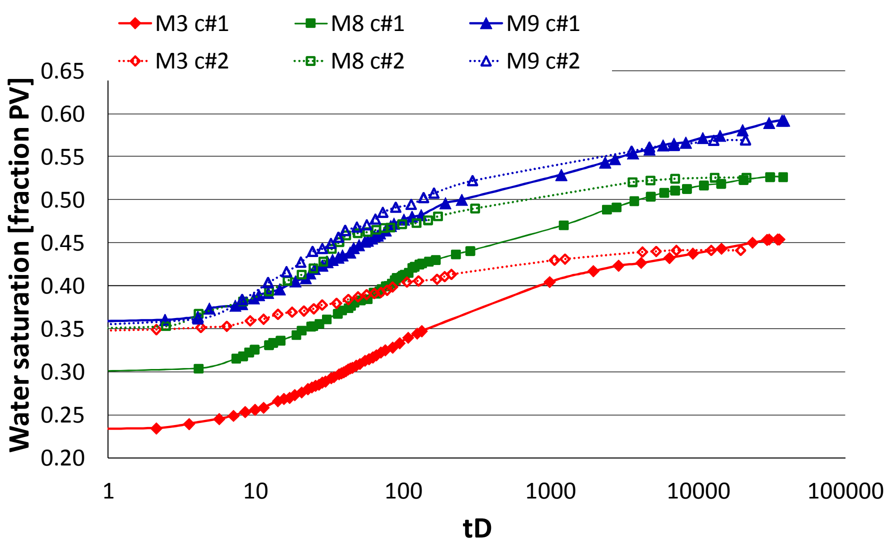

Figure 3. Development in water saturation S_w vs. t_D during spontaneous imbibition (c#1 and c#2) for core plugs aged 6 days at low/medium/high initial S_w: $M3^{L6}$, $M8^{M6}$, $M9^{H6}$.

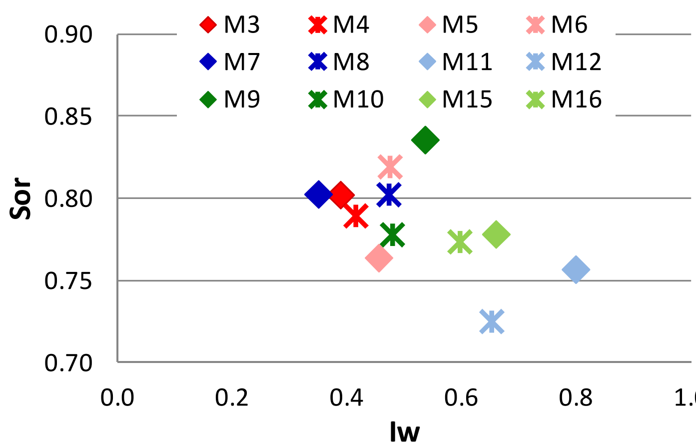

Figure 4. $S_{or,WF}$ vs. I_w for all core plugs after c#1 of spontaneous and forced imbibition.