

Aging Time Control by NMR Relaxation

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Abstract

It may become possible to simultaneously determine the full water-oil capillary pressure bounding curve cycle in the laboratory by two methods: (1) directly, by traditional fluid displacement through oil- and water-wet membranes, and (2) indirectly, by NMR relaxation. The coreholder has to be made of nonmagnetic, e.g., peek, material and placed inside the static magnetic field of the spectrometer. It may be possible then to estimate the branch of the capillary pressure curve from the relaxation data at each equilibrium point of the membrane method. Or, if the capillary pressure curve from the membrane data is fitted to a correlation, the parameters of the correlation could possibly be estimated from the NMR data.

If crude oil displaces formation water in a primary drainage process, the wettability may gradually change as the fluid-core system ages during the equilibrium wait periods, even at laboratory conditions, unless the system has been pre-aged. The standard way of doing this is to flood a water saturated core with crude down to a selected water saturation and then age for an appropriate time interval.

In this paper we report the results of an investigation into the possibility of using the NMR T_2 -data to determine the appropriate aging time for a crude oil/brine/rock system. We have used Berea cores and crude from the Snorre field. The main results are that the T_2 -distribution of continuously aged cores at 90 °C seems to be rather insensitive to wettability changes for water-wet systems, as determined by spontaneous water imbibition, but becomes a good indicator as the fluid-rock system approaches intermediate wetting conditions. A procedure with aging intervals at 90 °C interrupted by cooling and NMR relaxation measurements at 35 °C exhibits a gradual change in the geometric mean T_2 -value, right from the start.

Also, it is suggested that the aging procedure should perhaps be a slow, quasi-equilibrium process down to the selected water saturation to have a well-defined, equilibrated rock-fluid system similar to that in the reservoir, before the capillary pressure bounding loop measurements are initiated.

Keywords: Porous media, wettability, aging time, nuclear magnetic resonance.

1. Introduction

1.1 Capillary pressure curves from NMR-logs

The possibility of estimating capillary pressure curves from NMR logs has been discussed by Marschall *et al.* (1995), Volokitin *et al.* (1999), Lowden (2000), and Altunbay *et al.* (2001). The primary drainage capillary pressure curve may be estimated from the simple expression $p_c \propto 1/T_2$, where p_c is the capillary pressure and T_2 the spin-spin relaxation time, measured on a 100% water-saturated core. If there are both nonwetting oil and water in the core, the p_c

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estimate may be adjusted, Volokitin *et al.* (1999). Procedures for determining capillary pressure curves for nonwetting oil and water during an imbibition process, and for drainage and imbibition in a mixed-wet system, have not been reported.

In a reservoir there may exist large field-wide variations in wettability, Hamon (2000) and references therein. Also, at any location within the reservoir a unique history of drainage–imbibition sequences may have taken place. To infer saturation distributions from NMR-logs, other logs may help to determine the free water level and the capillary pressure from density differences and height above the free water level. In addition, it may be necessary to calibrate the NMR log interpretation procedure by laboratory experiments.

In a current research program, we plan to place a restored-wettability core inside a coreholder of nonmagnetic material in an NMR-spectrometer. Bounding and scanning capillary pressure curves will be recorded by the membrane method and fitted to a correlation, e.g., Skjaeveland *et al.* (2000). Simultaneous measurement of the T_2 -spectra will be performed and linked to the parameters of the p_c -correlation. The idea is then to extrapolate this benchmark interpretation by NMR-log data to estimate in situ p_c -curves for adjacent or similar locations in the reservoir.

As explained by Masalmeh (2001), wettability changes will introduce additional hysteresis effects in the capillary pressure curve for nonwater-wet systems. An increase in contact angle from 0° for a completely water-wet system will reduce the capillary pressure level. The residual oil saturation will decrease and the residual (irreducible) water saturation will increase, as demonstrated by measurements. And, as noted by Masalmeh, wettability itself may be a function of the initial oil saturation. Reformulated in terms of the p_c -correlation (Skjaeveland *et al.*, 2000), a saturation reversal from primary drainage will initiate an imbibition process and lock the wettability and residual saturations to constant values during all consecutive scanning imbibition and drainage processes, unless the primary drainage curve is rejoined at the first reversal point. If so, a continuation along the primary drainage curve implies further gradual change in wettability and residual saturations.

The p_c -correlation,

$$p_c = \frac{c_w}{\left(\frac{S_w - S_{wr}}{1 - S_{wr}}\right)^{a_w}} + \frac{c_o}{\left(\frac{S_o - S_{or}}{1 - S_{or}}\right)^{a_o}}, \dots\dots\dots (1)$$

and the associated hysteresis scheme were developed for the case of constant wettability. For crude oil displacement of initially 100% water saturated and water-wet cores, this may possibly be achieved in the laboratory by a very slow primary drainage at reservoir conditions down to the first saturation reversal. At any stage, the fluid-rock system should be in equilibrium and aging is not required. The c 's in Eq. 1 express the entry pressures for water- and oil wet systems. They are generally wettability dependent and related to the wettability indices. The a 's represent the "pore-size distribution index" which, in principle, should be independent of wettability. The Land equation (Land, 1968), for estimation of residual saturations from initial values, is suggested modified by Sarwaruddin (2001) and others referenced therein.

These considerations imply that cores for calibration and benchmarking of NMR-log interpretations need to be carefully prepared with due attention to the saturation processes and wettability variations that may exist in the reservoir. For example, if the reservoir during a geologic time period is developed by a downward drainage process, the initial saturation distribution may be modelled in the laboratory by a primary drainage curve, sufficiently aged at each small saturation step.

1.2 Aging time

Zhou *et al.* (2000) present a comprehensive study on interrelationship of wettability, initial water saturation, aging time, and oil recovery by spontaneous imbibition and waterflooding. They use Berea cores, synthetic formation brine, a Prudhoe Bay, Alaska crude oil to develop mixed-wet cores and a refined oil to obtain constant, strongly water-wet conditions. The cores were drained (primary drainage) directly to residual water saturation and then aged at 88°C for up to 10 days. Others workers report similar treatments but have chosen different aging times, to mention a few: Masalmeh (2001) 28 days; Anderson (1986) cites 10 references to support an equilibration time on the order of 1000 hours; Zhou *et al.* (1993) up to 2 days; Zhang *et al.* (1999) 3 weeks. There is an interesting discussion on the SCAWeb Forum on this topic

1.3 NMR and wettability changes

Several authors have reported wettability changes determined by NMR relaxation measurements, e.g., Zhang *et al.* (1999), Howard (1998), Manalo *et al.* (2000).

During an aging period, a relocation process takes place in the pore network. The oil phase makes contact with the rock surface, and the oil protons relax faster. At the same time, the water protons relax slower although the water phase may still have contact points with the rock surface. The overall effect is to shift the composite distribution of oil and water protons to lower relaxation times. Even if the aging process should make the rock surface completely oil-wet, the faster bulk relaxation of water protons will probably shift the composite distribution to lower relaxation times.

The the relaxation time distributions may exhibit structure and details and it may be difficult to decide by inspection if an overall shift has taken place. Also, the data program which translates the observed decay of magnetization into a distribution of protons with different relaxation times relies on some form of smoothing procedure, e.g., Tikhonov regularization (Tikhonov and Arsenin, 1977). It is therefore of interest to apply an overall measure of the average relaxation time of the distributions, e.g., the geometric mean relaxation time, as suggested by Manalo *et al.* (2000), Borgia *et al.* (1996).

2. Experiments

2.1 Fluids

We used dead crude from the oil-wet Snorre sandstone field in the North Sea, the same type of oil as Hammervold *et al.* (1996) employed in the capillary pressure scanning curve measurements of the fresh, oil-wet reservoir core. The crude has a density of 0.8531 g/cc and a viscosity of 16.2 cp.

A mixture of two synthetic mineral oils, Marcol 82 and EDC 95/11, was made with an average viscosity close to that of the crude oil. The density of the mixture was 0.8379 g/cc and the viscosity 17.6 cp

The composition of the synthetic formation water was 20 g/l NaCl, 2 g/l $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$, 0.4 g/l KCl and 0.8 g/l CuSO_4 . This gives us a density of 1.0139 g/cc and a viscosity of 1.18 cp.

2.2 Cores

Eight coreplugs, No 1–8 in Table 1, were cut from the same long Berea core. Their properties are shown in Table 1. The residual water saturations, S_{wr} , were established by flooding the cores with crude oil. There are some variations between the plugs and we could perhaps have tried to establish a more uniform residual water saturation. According to Zhou *et al.* (2000), a decrease in S_{wr} results in a decrease in water wetness, since the oil phase then gets in closer contact with the rock. Another Berea coreplug, No 9 in Table 1, was treated similarly but separately for preliminary check on wettability changes.

Table 1. Properties of Berea Cores

No	$L[\text{cm}]$	$d[\text{cm}]$	$dWt[\text{g}]$	$wWt[\text{g}]$	$V_b[\text{cc}]$	$V_p[\text{cc}]$	ϕ	S_{wr}	$k[\text{md}]$	Aging[d]
1	4.4	3.8	109.1	120.4	49.8	11.2	22.4	32.8	175	47
2	4.8	3.8	109.1	120.8	53.3	11.6	21.8	27.7	166	42
3	4.7	3.8	106.7	118.0	52.2	11.2	21.4	28.3	188	38
4	4.7	3.8	107.0	118.3	52.2	11.1	21.2	24.2	144	33
5	4.7	3.8	108.6	119.9	52.7	11.2	21.2	24.1	152	28
6	4.8	3.8	109.5	121.0	53.5	11.3	21.2	22.4	151	24
7	4.7	3.8	108.6	118.7	52.9	10.0	19.0	17.2	160	12
8	4.6	3.8	106.4	117.3	51.6	10.8	20.8	27.5	147	
9	4.6	3.8	107.0	119.8	54.3	12.6	23.2	23.2		

2.3 Experimental procedures

After cutting, cores 1–8 were cleaned in an ultrasonic brine bath, then in a Soxhlet apparatus with toluene and methanol for two days and dried at 105 °C for several days. They were cooled in an exsiccator with silica gel.

The dry cores were vacuumed and saturated with synthetic formation water. Porosity was determined from the change in weight. The cores were flooded with about 15 PV of brine at four different rates for permeability measurements listed in Table 1. They were then left immersed in brine for several days.

Cores 1–7 were flooded (primary drainage) with 2–2.5 PV of crude oil in each direction at a flow rate of 5ml/min in a Hassler coreholder sleeve. The confining pressure was about 15 bar. Core 8 was treated accordingly but with the mixed mineral oil. This core remains 100% water wetted, Jadhunandan and Morrow (1995), and is a reference for the spontaneous imbibition tests.

Cores 1–7 were immersed in crude oil in a closed container and aged at a temperature of 90 °C for time intervals listed in the last column i Table 1. The aging started with Core 1, and with an increasing delay time for the others. All 7 cores were removed from the heating cabinet and subjected to T_1 and T_2 measurement on the same date with the same parameters, i.e., echo spacing T_E of 0.5 ms and the number of echoes and wait times T_W appropriately

selected to match the relaxation characteristics of the cores. All measurements were performed at a stabilized temperature of 35 °C by a Resonance Instrument MARAN 2 spectrometer which operates at 2 MHz. The T_2 -values were measured by a CPMG pulse sequence and T_1 -values by inversion recovery. The mapping of the decaying magnetization into relaxation time distributions was performed with the Resonance Instrument software package. Spectra were also recorded just before and right after primary drainage, before aging started.

Core 9 was prepared in a similar manner as for Cores 1–7 except for the 15 PV brineflooding at four different rates to determine permeability. The aging consisted of interrupted periods. At certain points in time, the core was removed from the heating cabinet at 90 °C, equilibrated at 35 °C, and the T_2 -distribution was recorded before it was put back into the heating cabinet again.

Spontaneous imbibition for Cores 1–7 was measured in Amott cells. The cores were placed in the cells surrounded by formation water and the volume of displaced oil was measured over time. Core 8, having been flooded with mineral oil, was also exposed to formation water in an Amott cell to benchmark a completely water-wet system.

3. Results

3.1 T_2 -distributions

In Fig. 1 is shown, as an example, the T_2 -distributions of Core 6 just after primary drainage, i.e., flooding with crude, and after 24 days continuous aging. The distributions are almost identical. The 100% water saturated peak at 250 ms corresponds to bulk water and the peak at 350 ms corresponds to bulk oil. After 33 days of continuous aging, Fig. 2 for Core 4, the peak of the distribution has moved from 350 to 250 ms indicating that the crude oil phase has come in contact with the rock surface. Also, the whole distribution is shifted to lower relaxation times.

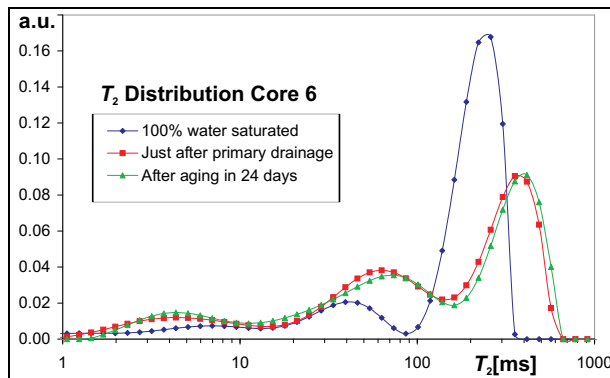


Fig. 1– T_2 -distributions for core 6.

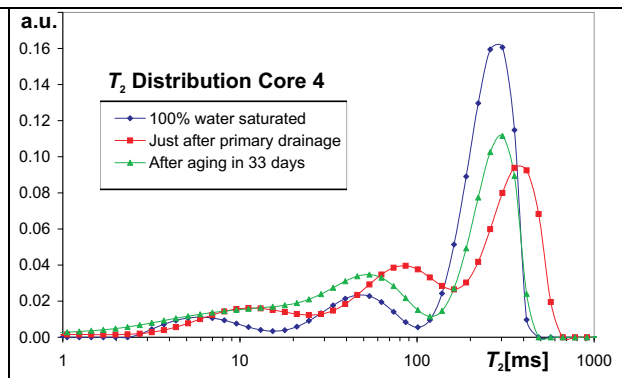


Fig. 2– T_2 -distributions for core 4.

3.2 T_1 -distributions

Figs. 3 and 4 show examples of T_1 -distributions after 28 and 33 days continuous aging, for Cores 5 and 6. The same trend is seen as for the T_2 -distributions. There is a shift to shorter relaxation times for the whole distribution as aging time increases.

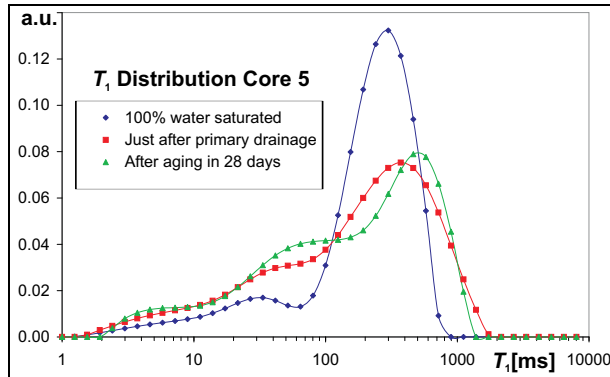


Fig. 3– T_1 -distributions for core 5.

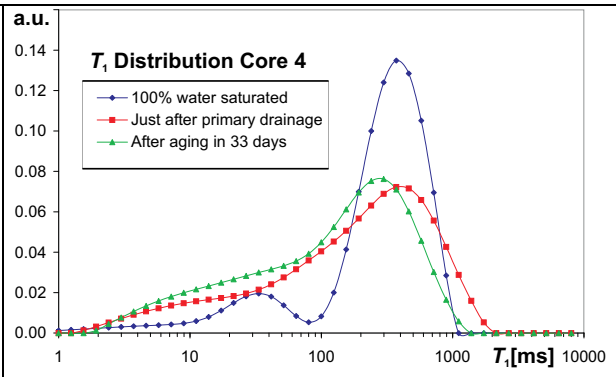


Fig. 4– T_1 -distributions for core 4.

3.3 Geometric mean relaxation time

In Fig. 5 are shown two pairs of the geometric mean of the relaxation times T_1 and T_2 as functions of continuous aging time, just before and right after the start of aging. As also is obvious directly from the spectra in Figs. 1–4, there is almost no shift in the mean geometric relaxation times before and after aging until the observations at 33 days, when there is a shift in T_{2g} of 34 ms. This shift is reduced to 7 ms for Core 2, after having aged continuously in 42 days, and increases again to 32 ms for Core 1, after 47 days aging. As will be shown below, these observations are consistent with the spontaneous imbibition performance of the same cores.

In Fig. 6 is shown the evolution of T_{2g} for Core 9. For each point, the core was cooled from 90 to 35 °C and measurements taken before it again was placed in the heating cabinet at 90 °C. The T_{2g} -value is gradually reduced from 109 ms to 61 ms after 20 days, and then increases slightly again.

It seems like continuous aging does not affect T_{2g} -values until a certain time is reached, here about 30 days. It is possible that wettability is continuously changing towards more oil-wet conditions with aging, but that the changes are not reflected in T_{2g} until about 30 days. For the other type of process, interrupted aging, Fig. 6, the T_{2g} -values change from the start of the aging process. Since T_2 -values usually are the information available from NMR-logs, it should be of interest to determine how sensitive the T_2 -distribution and the T_{2g} -values are to changes in wettability.

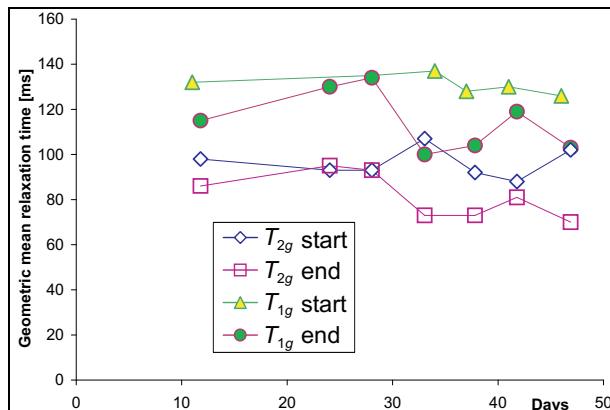


Fig. 5–Geometric mean relaxation times before and after continuous aging, Cores 1–7.

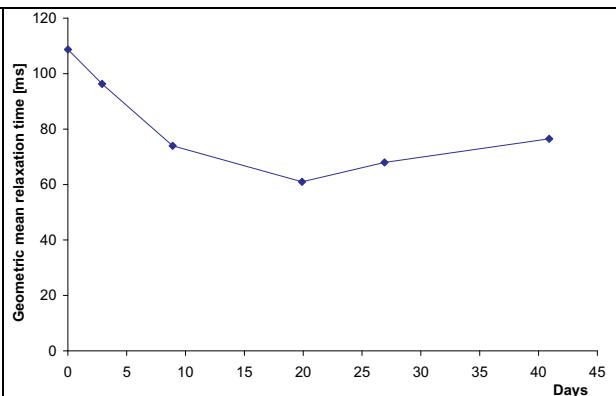


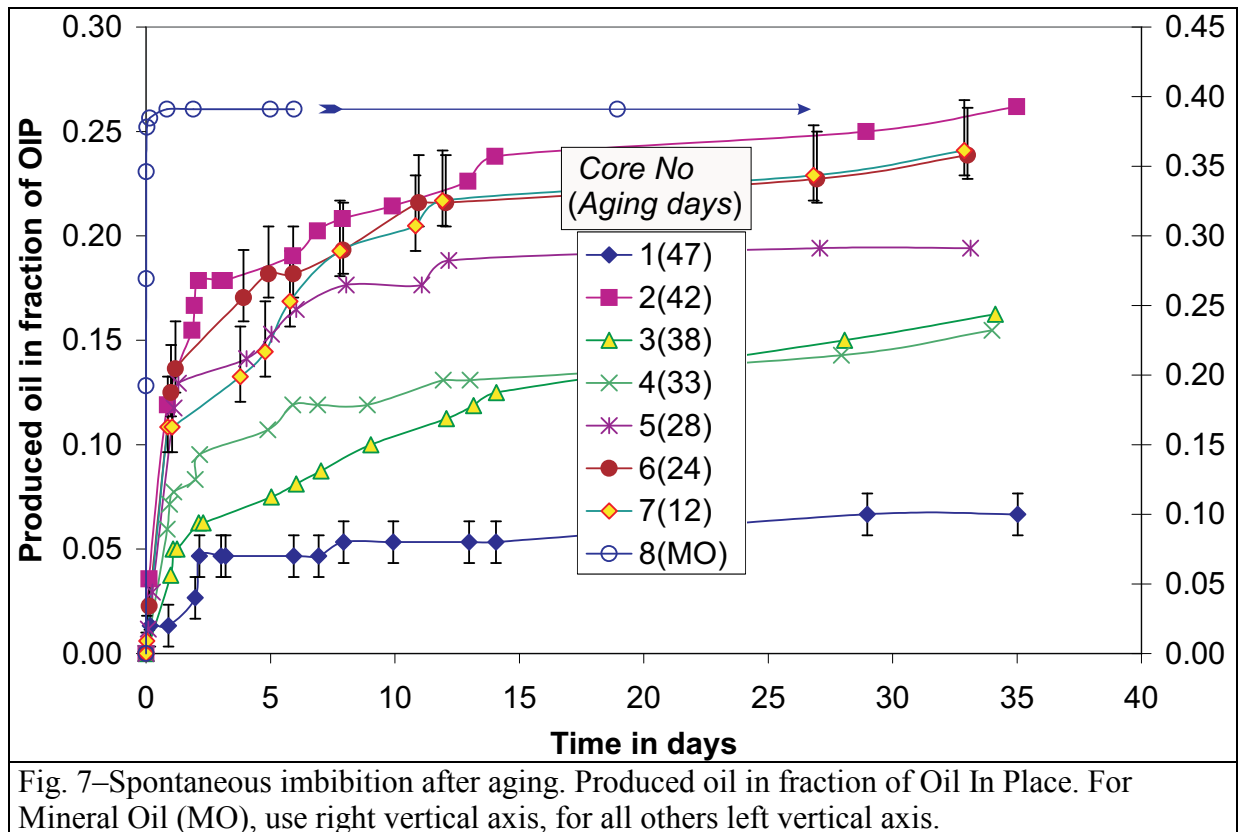
Fig. 6–Geometric mean T_{2g} , Core 9, at interrupted aging times.

3.4 Oil recovery by spontaneous imbibition

In Fig. 7 is shown oil recovery as function of time for the aged Cores 1–7 and Core 8, i.e., Amott tests. Core 8 instantaneously imbibes water and very fast the oil recovery reaches the level of 0.39. Except for Core 2 with 42 days aging time, the curves fall in sequence according to the aging period prior to imbibition and correspond fairly well with the shift in T_{2g} in Fig. 5. Specifically, Core 2, which in Fig. 5 has a small T_{2g} -shift similar to Core 5 (28 days), 6 (24 days), and 7 (12 days), groups together with these cores in Fig. 7 and not with Core 1 (47 days) which has a comparable aging time. Hence, after about 30 days in Fig. 5, the T_{2g} -shifts seem to correlate well with wettability. (We have no good explanation why Core 2 behaves out of order in the first place since all 7 cores are cut from the same long core and have been treated in the same manner.)

In Fig. 7, there is a wettability range between Core 8 (MO), which is 100% water wet, and Core 2, 6, and 7. It appears, from Fig. 5, that the T_2 -response and the T_{2g} -shift may be fairly insensitive to wettability changes in this range, for continuous aging. We plan to investigate more in detail the T_2 -response and the T_{2g} -shift as functions of wettability from 100% water-wet down to intermediate-wet conditions by including shorter aging times. A measure of wettability that differentiates well between high water-wet states is the relative pseudo work of spontaneous imbibition, Ma *et al.* (1999).

From Fig. 7, disregarding Core 2 (42 days), the wettability is still changing from 38 to 47 days, and seems to reach close to intermediate wetting conditions at 47 days.



4. Discussion

If in situ capillary pressure curves are to be inferred from NMR logs, the interpretation scheme probably has to be calibrated by laboratory measurements. Then it is important to properly prepare the cores with restored wettability. Gray (2001) comments that the wetting state depends on the selected water saturation which should be based on the capillary pressure related to height above the free water level. This water saturation should be established in the core before aging starts. Masalmeh (2001) ages the cores for four weeks after primary drainage before starting the subsequent imbibition-drainage experiments. He finds that as the plug becomes less water-wet, the trapped oil saturation will decrease and the trapped water saturation during secondary drainage will increase. Hence, the capillary pressure branches of (1) primary drainage, followed by aging, (2) primary imbibition, (3) secondary drainage do not form a closed loop. This is also the case for scanning loops originating on the primary drainage curve.

In the reservoir, the initial vertical distribution of oil and water is often assumed to be the result of a downwards primary drainage. This is a very slow, quasi-static process at constant temperature. When the reservoir is discovered, the rock-fluid system is in equilibrium at each height above the free water level. The reservoir is less water-wet and the water saturation decreases with height above the free water level. We believe that if these conditions are restored in the laboratory by a slow, quasi-static primary drainage at reservoir conditions, a saturation reversal at any point on the curve will initiate a closed loop system. That is, until the process, which could be an arbitrary sequence of drainage and imbibition subprocesses, returns to the point of first reversal, the wettability of the core is constant.

Our preliminary results in this paper show that T_{2g} -values seem to depend on the aging process itself, not just the aging time after flooding with oil. To restore reservoir wettability in crude/brine/rock system, the proper question is perhaps not just what the aging time should be after flooding with crude, but how slow the primary drainage process should be run down to a selected water saturation. In this case, the T_2 -spectrum may be sufficiently sensitive to act as an equilibrium indicator.

5. Conclusions

1. The sensitivity of the T_2 -distribution to wettability seems to depend on the selected aging process.
2. The T_2 -distribution seems to be fairly insensitive to wettability changes for states of high water-wetness.
3. At least 38 days of continuous aging is necessary for a brine-saturated Berea core that has been flooded to residual water saturation by crude from the Snorre field.
4. A proper aging process should possibly be a primary drainage at quasi-static fluid-rock equilibrium, rendering wettability as a function of water saturation along the primary drainage curve.
5. The geometric mean T_2 -value is probably a good measure of the wettability state of the core-fluid system, except perhaps at a high degree of water-wetness.
6. Capillary pressure correlations need to include wettability changes for the interpretation of NMR-logs.

6. Nomenclature

- a = parameter in correlation, dimensionless
- c = parameter in correlation, bar

d = diameter, cm
 k = permeability, md
 L = length, cm
 p = pressure, bar
 S = saturation, dimensionless
 T = relaxation time, s
 ϕ = porosity, fraction or per cent

Subscripts

b = bulk
 c = capillary
 g = geometric mean
 i = initial
 o = oil
 p = pore
 r = residual (irreducible)
 w = water
1 = spin-lattice
2 = spin-spin

Abbreviations

dWt = dry weight
wWt = wet weight

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8. References

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