A MULTI-PHYSICS APPROACH FOR MONITORING A WETTABILITY ALTERATION ON CARBONATES DURING AGING

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ABSTRACT

Wettability influences the multiphase flow of fluids in oil reservoirs: it is measured in laboratory with specific procedures including assessment of its initial value. If core plugs cannot be used at native state, a good cleaning is required before any core analyses. Then, a restoration of the initial wettability conditions of cores by aging them is required to get representative data of the reservoir. USBM and/or Amott-Harvey tests are generally performed to assess the wettability of the cores. In this study, NMR and resistivity measurements were carried out at different time intervals while aging to monitor the wettability alteration of carbonate rocks (dolomites–limestones). Our goal was to highlight the mineralogy effect on the aging process.

Four carbonate outcrop cores were cleaned using two different methods, respectively conventional and forced cleanings. USBM and Amott-Harvey wettability indices were calculated to evaluate the efficiency of each cleaning. Then, their cementation factor m was calculated and NMR T_2 relaxation was performed as a reference on fully brine saturated samples. A dead crude oil was injected into the plugs to reach S_{wi}; NMR T_2 and resistivity were measured again. Two static aging methods were tested at same temperature. Resistivity and NMR T_2 were recorded at different time intervals while aging.

A comparison between different cleaning and aging procedures showed that forced cleaning and aging under confining stress are required. The combination of resistivity and NMR T_2 measurements showed the progress of aging and corresponding wettability alteration. The slow progress of wettability alteration observed in this study may be attributed to the slow process of aging using stock tank oil rather than live oil. The multi-physics approach demonstrated that dolomite seems to be more prone to become oil-wet than limestone. Additional works need to be done to confirm the role of mineralogy on aging.

INTRODUCTION

Wettability is one of the most important properties affecting oil recovery efficiency of a reservoir. The understanding of wettability modification is crucial for the accuracy of all core measurements and studies because of its effects on capillary pressure, relative permeabilities, waterflood behavior, electrical properties and EOR studies. The dynamic mechanisms involved in wettability changes are still not clear and require more investigations.

Generally in core laboratories, measurements are made on cores which could be in one of the following states: native state, cleaned state or restored state. The methods most often used to measure and verify wettability in core plugs are Amott-Harvey and USBM. Depending on the number of capillary pressure steps and rock properties, both methods are time consuming especially if their purpose is to monitor the change of wettability in the plugs while aging, in which case these methods have to be repeated several times.

Numerous papers dealing with wettability restoration and aging procedures have been published, essentially on sandstones. A wettability literature survey was done by W.G. Anderson [3, 4, 5, 6, 7, 8], who reported the effects of wettability on different parameters such as capillary pressure, relative permeability, and the electrical properties of porous media. Baldeo Singh [11] conducted aging experiments showing that the cores aged at low pressures gave more oil-wet behavior than the cores aged at higher pressure. Villard et al [9] performed aging tests at different conditions and concluded that the extent of wetting alteration can be varied by changing the aging time and temperature. De Pedroza et al [2] showed that the degree of wettability alteration was found to be dependent on the amount and the physicochemical properties of asphaltenes present in the crude oil. They demonstrated that core cleaning before the aging could affect the degree of wettability alteration after the aging. From the NMR point of view, Al- Mahrooqi et al [12] showed that NMR T_2 distribution could provide valuable information regarding rock wettability, rock/fluid interactions and phase distributions. Later, Al-Mahrooqi et al [1] presented another study using a combined wettability measurement through shift in NMR T_2 relaxation times and changes in electrical impedance in consequence of changes of rock/fluid interactions on sandstones. In this study, samples were first cleaned with conventional method (Dean-Stark extraction). Johannesen et al [10] showed that NMR measurements indicated a linear relationship between wettability and T_2 relaxation times for the oil phase in chalk core plugs. Different aging techniques were tested showing varying degrees of wettability uniformity. Meissner et al [14] reported that wettability restoration by a static method (cores immersed in crude oil) was not effective for the moderately low-permeability limestone cores in comparison with siliciclastic cores due to the complex pore structure of carbonates. Ferno et al [13] presented experimental wettability alteration results using static and dynamic aging methods. They showed that dynamic aging with continuous crude oil injection exhibited greater reduction in water-wetness of strongly water-wet chalk plugs than the one achieved with the static aging.

The types of mineral surfaces in a reservoir are also important in determining wettability.

It is known now that generally carbonate reservoirs are more oil-wet than sandstone ones. A lack of published data on carbonate rocks drove us to perform aging tests and monitor resistivity changes due to the aging effect, i.e., wettability change, on two different carbonate rock types i.e. dolomite and limestone. NMR T_2 relaxation data was also used to highlight a change of fluid distribution occurring at the pore-scale during wettability restoration. In the frame of the same study, conventional versus forced cleanings efficiencies were investigated using the USBM and Amott-Harvey methods. The experimental procedure followed in this study was similar to the one proposed by Al-Mahrooqi et al. [1] who worked only on sandstone rocks. Carbonates are made of more complex structure-mineralogy and have larger range of wettability than sandstones, making conventional cleaning inappropriate and aging understanding more difficult.

THEORY, MATERIALS AND EXPERIMENTAL PROCEDURE

Theory

In this study, two different physics were used to highlight the change in wettability of core plugs; resistivity and NMR.

Resistivity is known to be sensitive to saturation history and wettability as well. Archie's laws express an empirical correlation between electrical resistivity index and brine saturation. It indicates that the resistivity index is only a function of the conducting phase saturation for a given formation, as shown by the Archie's equations:

$$FF = \frac{R_o}{R_w} = \frac{a}{\phi^m} \quad \& \quad RI = \frac{R_t}{R_o} = \frac{1}{S_w^n} \tag{1}$$

where FF is the formation factor, R_o and R_w are the resistivity of the 100% brine saturated formation and resistivity of the brine phase respectively, R_t is the resistivity of the formation at saturation S_w, ϕ is the porosity, RI is the resistivity index, a is empirical constant (taken equal to 1 in the following calculations), m is the cementation exponent, and n is the Archie's saturation exponent.
Experimental observations have already indicated that wettability and saturation history could significantly alter the correlation of resistivity index with the brine saturation.

Using NMR, the theory of T_2 in porous media is basically summarized in the following equation:

$$\frac{1}{T_2} = \frac{1}{T_{2,bulk}} + \rho \frac{S}{V} f(I_w) \tag{2}$$

where T_2 is the measured relaxation time of the fluid in the rock, $T_{2,bulk}$ is the relaxation time of the bulk liquid, S and V are the surface and volume of the pore respectively, ρ is the relaxivity of the rock surface and $f(I_W)$ a function of the wettability that can have values between 0 and 1. The equation 2 can be easily understood if interpreted as follows: the observed relaxation time of the liquid in the rock is a weighted average of bulk relaxation) and surface relaxation. The weight is proportional to the S/V ratio, and therefore contains information on the pore size. The term $f(I_W)$ can be interpreted as the fraction of surface of the pore in contact with the liquid considered (and therefore connected to the wettability of the sample). The equation 2 is valid for each of the fluids. In case of mixed fluids (water and oil), the measured response is a combination of the two fluids. In the following study, the purpose is not a quantification of wettability indices with the NMR relaxometry like proposed by Fleury and Deflandre [15] or Looyestijn and Hofman [16] but just a monitoring approach for a wettability change while aging.

EXPERIMENTAL

Porous Media

4 carbonate core plugs, two dolomites and two limestones, were selected and separated in 2 sets:

- Set 01 was composed of one dolomite D6 and one limestone L4
- Set 02 was composed of one dolomite D1 and one limestone L5

After two different cleaning methods (conventional and forced cleanings), conventional core analysis such as porosity and gas permeability were measured prior to plugs saturation with brine. Then their cementation factor *m* (at S_w=1) was measured using the ambient resistivity meter. The properties of all four plugs are reported in Table 01:

Plug Label	Lithology	Set	Porosity %	N_2 Permeability Kg (mD)	Cementation factor *m*
D6	Dolomite	01	19.56	83	1.98
L4	Limestone	01	17.02	8.35	2.05
D1	Dolomite	02	13.50	185	1.93
L5	Limestone	02	20.18	766	2.19

Table 01: properties of core plugs.

Fluids

A NaCl brine of 200kppm was prepared and used for plugs' saturation. This choice was not arbitrary: water density and resistivity had to match with the formation brine of the reservoir. The brine resistivity R_w at reservoir temperature was calculated from the measured resistivity at laboratory temperature using Arps relation.

Crude oil from a carbonate reservoir was used as oil phase. Before using it, the stock tank oil was filtered at 10µm to remove the heaviest components. This particular crude oil contained asphaltene components as shown in Table 02:

	Density (g/cc)	Viscosity (cp)	Total acid number (mg KOH/g)	Total base number (mg KOH/g)	Asphaltene content %
Brine	1.1499	1.65			
Clean Oil	0.9044	57.06	0.45	0.14	2.62
Used Oil	0.9052	NA	0.14	0.14	1.27

Table 02: fluids properties at 20^{o}C.

The properties of the same oil are given before it was injected into plugs and after it was collected at the effluent side. It clearly indicates that some asphatenes (and acides) had been absorbed by the core plugs during their de-saturation.

Procedure

It was decided to run two sets of aging experiments in parallel using a static method at two different conditions and for two different carbonate rock types (limestone / dolomite).

Prior to the aging process, all core plugs were first cleaned through conventional cleaning procedure. In addition to that, the set 02 was also cleaned extensively. The USBM and Amott-Harvey indices were estimated both after the conventional cleaning and the forced cleaning. After the wettability assessment, the two sets of plugs were cleaned again with the method which gave the best results in term of extent of water-wetness.

After the conventional dry measurements (porosity and gas permeability), the four plugs were saturated with the same 200kppm NaCl brine. Their cementation factor *m* was calculated at ambient pressure and temperature using the ambient resistivity meter at frequency of 20kHz. NMR T_2 relaxation was performed as a reference on all four fully saturated samples.

The crude oil was injected into the plugs. The 4 carbonate plugs were de-saturated using a centrifuge at a constant capillary pressure of 32psi corresponding to 3000rpm. In order to get a fluid redistribution that yields a quasi uniform profile, plugs were re-loaded in the centrifuge after flipping them. After that, resistivity and NMR T_2 relaxation were measured and a first saturation exponent at S_{wi} before aging was determined on all plugs. All following resistivity measurements were carried at the same frequency (20kHz).

Two different static aging methods were chosen to age the core plugs:

- 1/ the two core plugs from set 01 were aged in a beaker full of crude oil at 62^{o}C. This technique was chosen because quite a number of published papers report the use of core submersion in a crude oil at elevated temperature. The changes in resistivity were measured while plugs were undergoing aging, with a 4-points ambient resistivity meter. At regular time intervals, plugs were cooled down and brought to the ambient resistivity meter and then to the NMR system to measure the resistivity and NMR T_2 relaxation, respectively.
- 2/ the two core plugs from set 02 were aged in a HPHT resistivity system. Plugs were loaded in a 4-points resistivity core holder under 800psi net pressure and 62^{o}C of temperature. The NMR T_2 relaxation was measured only before and after the plugs had been aged for two reasons: first, to avoid a change in water saturation during the processes of loading, unloading and transportation of the core plugs (observed with method 1/); second, to acquire resistivity data at short time intervals (for instance, every three hours).

A system of 4-points electrodes was preferred to a 2-points system because 4 leads pattern usually gives better accuracy (no contact resistance). A LCR meter was used to measure the resistance between the electrodes at 20kHz.

RESULTS

Cleaning assessment

Two different cleanings were tested on set 02: conventional cleaning (Dean-Stark and Soxhlet extraction) and extensive cleaning. The extensive cleaning method consisted in a conventional cleaning followed by a sequential injection of toluene and methanol into the plugs loaded in heated core holders. The injection process continued until the clear toluene at the outlet of the plugs was observed indicating possibly that all the oil in the core plugs had been removed.

The efficiency of each cleaning was then determined by calculation of USBM and A-H wettability indices by centrifuge using dodecane as non active oil.

	After convention cleaning		After forced cleaning	
	USBM WI	A-H WI	USBM WI	A-H WI
D1dolomite	0.35	0.05	0.38	0.45
L5 limestone	0.58	0.74	0.61	0.78

Table 03: USBM and Amott-Harvey wettability indices after conventional cleaning.

Table 03 shows clearly that, after the forced cleaning, an appreciable improvement in the A-H wettability index was observed for the dolomite plug in comparison with the one after the conventional cleaning. That improvement in the WI of the dolomite plug could be attributed to the large spontaneous oil production in imbibition. Since that assessment highlighted the positive effects of the aggressive cleaning, all plugs were conventionally cleaned and also flush cleaned.

De-saturation

After the conventional analysis (Table 01), NMR T_2 relaxation was performed as reference on fully saturated samples. Crude oil was then injected into the plugs using a centrifuge (at 3000rpm). When the brine production stopped, the plugs were flipped and run again in centrifuge. At this stage, new measurements of resistivity and NMR T_2 were carried out for the purpose of reference before aging. Resistivity measurements of de-saturated samples from set 01 were carried out using the ambient resistivity meter. As for set 02, resistivity measurements were carried out in the resistivity core holder of the apparatus at 62^{o}C and at 800psi of confining pressure. Because NMR T_2 distribution signals are “qualitatively” similar for the four samples, only NMR T_2 relaxation from set 02 (D1 and L5) are presented:

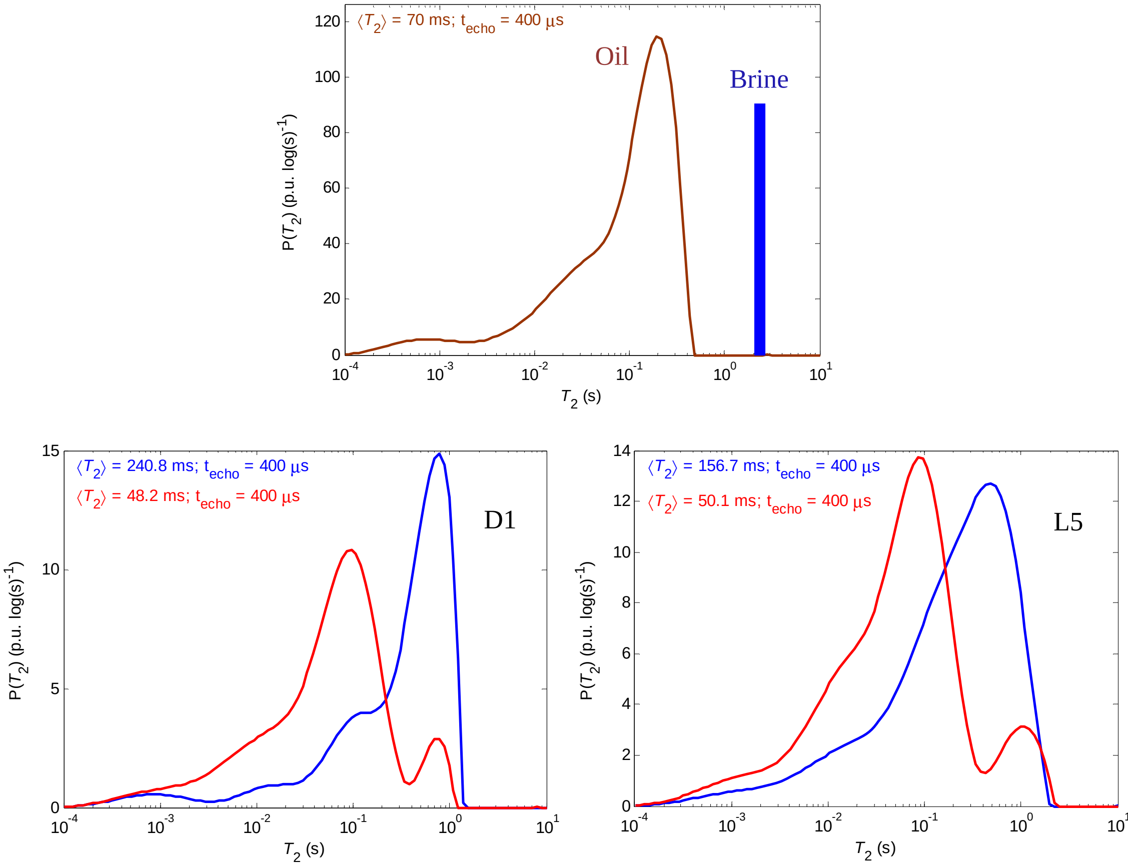

Figure 01: T_2 bulk oil (top) and T_2 distributions of D1 (bottom left) and L5 (bottom right) – plugs fully brine saturated in blue, core plugs de-saturated in red.

In Figure 01 (bottom), the shift in the T_2 distributions between the fully brine saturated stage (blue) and the de-saturated stage (red) shows that pores that were initially filled by brine have been filled by oil. Due to the higher viscosity (shorter T_2 bulk values) of the oil compared to the brine, the spectrum is shifted on the left. The remaining signal above about 300ms can only be interpreted as remaining water in the plugs. This is because the maximum T_2 value of the bulk oil (Figure 01 top) is about 300 ms and the porous matrix can only enhance the relaxation process of the liquid compared to the bulk.

At this reference time, thanks to the resistivity measurements (performed at 20kHz), saturation exponents n of all samples were calculated using the Archie's law (equation 02).

	SET 01		SET 02	
	D6	L4	D1	L5
S_{wi} (%)	32.0	59.8	28.2	57.5
n	4.69	7.88	2.73	2.10

Table 04: Irreducible water saturation and saturation exponents after de-saturation.

The ambient resistivity meter was used to determine the saturation exponents of plugs from set 01. All values were found to be higher than expected. These unexpectedly high values of *n* can be attributed to the design of the ambient resistivity meter.

For set 02, using the HPHT resistivity apparatus, the values of saturation exponents *n* are more representative of the state of the de-saturated core plugs. Values are close to 2 but a little bit higher. The aging process had probably already started in the plugs before the first resistivity measurement was made on them. Moreover, as shown in Table 02, asphaltene in the clean oil had been absorbed by the plugs during the de-saturation process, which can partly be attributed to the *n* values higher than 2.

Aging & Measurements

Set 01

The two core plugs were immersed in crude oil kept in the oven at 62^{o}C. During the aging process, they were removed from the oven at regular time intervals for resistivity measurements with the ambient 4-points resistivity meter, and NMR T_2 relaxation measurements.

The temperature of the core plugs was brought down to the room temperature before they were used for the resistivity and NMR measurements. After 12 days immersed in crude oil at ambient temperature, the samples were placed in the oven at 62^{o}C. The resistivity measurements made on the plugs after keeping them in the oven for some days, started showing an increase in resistivity, hence increase in *n* (Figure 02).

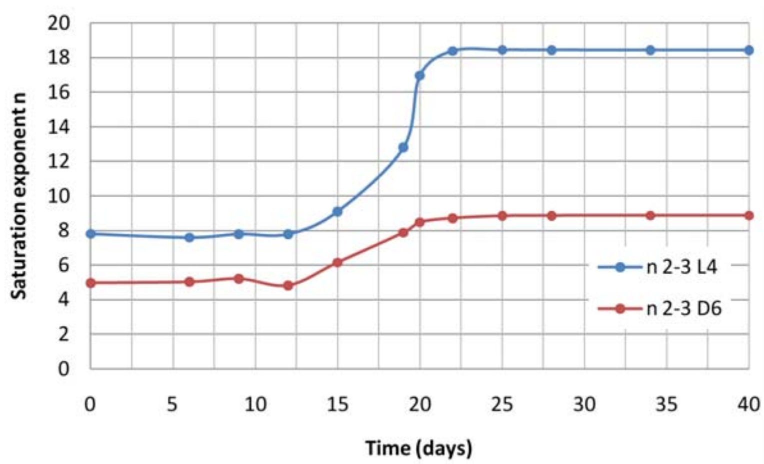

Figure 02: Saturation exponents *n* versus time for samples L4 and D6 from set 01.

The *n* increase appears only after applying a temperature of 62^{o}C and is more pronounced for the limestone plug L4 than the dolomite plug D6. This observation is contradictory with the amount of oil present in the samples ($S_{o[D6]}>S_{o[L4]}$). After 8 effective days of immersion in hot crude oil, *n* values became almost constant: the aging process for both core plugs was achieved.

It seems difficult to explain the difference in the behavior of limestone and dolomite samples because of the fact that the ambient resistivity meter is not suitable for multi-phase fluids saturated core plugs. High values of n are not only due to the real change of wettability of the plugs, but also to the experimental configuration of the resistivity system (limitation due to the poor contact between the two rods and the surface of the plug). This method can only be used as qualitative observation of a wettability alteration.

The results of NMR measurements on set 01, performed at different aging times, are shown in Figure 03.

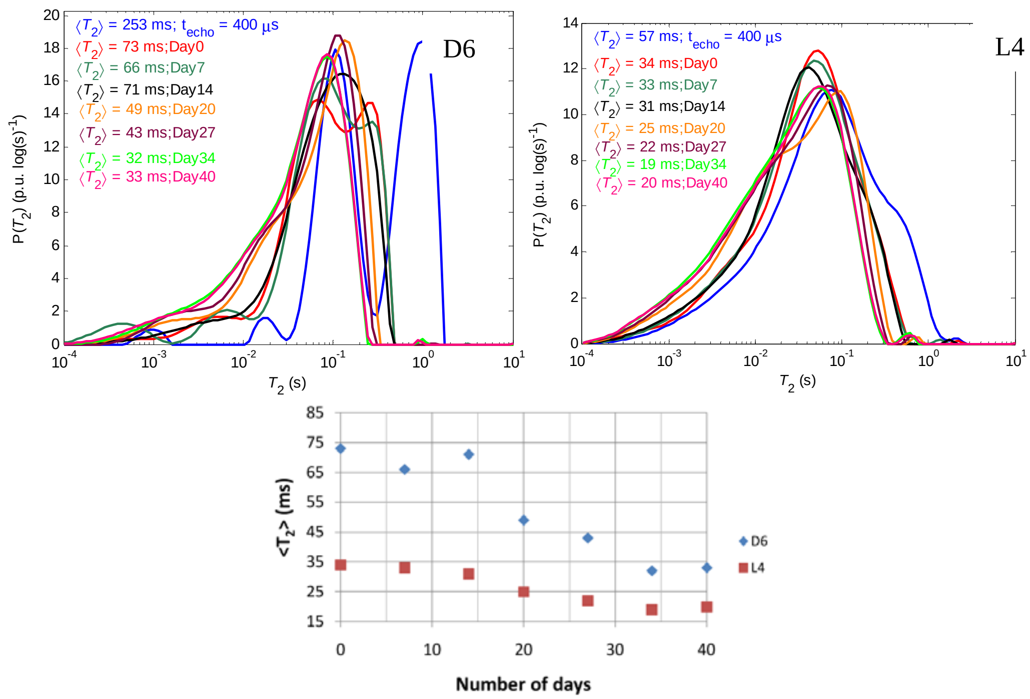

Figure 03: T_2 distributions (top) and T_2 log means (bottom) at different time intervals.

A change in NMR T_2 distribution with increasing time interval is visible. There is a systematic drift towards shorter T_2 values (clearer on the T_2 log mean plots) as the samples get aged, possibly due to increasing trend in the oil-wetness of the plugs. If we refer to the equation 02, this is a sign of increasing values for the $f(I_W)$: in other word, a larger and larger surface is in contact with the oil phase as the aging time increases.

Set 02

In order to avoid resistivity offset after each loading-unloading sequence, NMR T_2 relaxation was measured before de-saturation, before and after aging. The two core plugs were placed in the HPHT resistivity system, under 800psi of confining pressure and temperature of 62^{o}C, to set in the aging process. Resistivity measurements were made and recorded every 3 hours in the vertical resistivity core holders.

Electrodes are referenced as numbers 1 to 4. The resistivity was measured continuously along the core plugs between electrodes 1 and 2, 1 and 3, 1 and 4 or between the two internal electrodes 2 and 3. The results of saturation exponent n measurement versus time for all four electrodes sets (1-2, 1-3, 1-4 and 2-3) are given below:

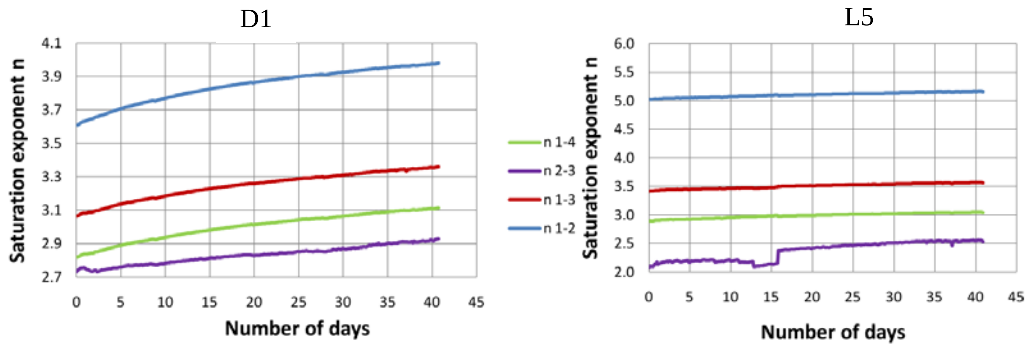

Figure 04: Saturation exponents of D1 and L5 at different stages during the aging period.

The above plots show an increase of saturation exponent n with time due to aging effect. The increase of n is more pronounced for the dolomite D1 than the limestone L5. In average, the increase in n is about 9% for the dolomite and 5% for the limestone. This was probably due to the higher oil saturation in the dolomite than in the limestone, 71.8% and 42.5%, respectively. Results between electrodes 2 and 3 (no contact resistance) are considered more accurate. Aging process is achieved for the limestone after 41 days whereas it is still ongoing for the dolomite.
It means that aging time, in the same conditions and with same fluids, can be different according to the mineralogy and the saturation. An additional experiment should be run with 1 dolomite and 1 limestone but at same saturation to highlight the real effect of the mineralogy.

The NMR T_2 distribution plots confirm a change in the wettability of the two plugs before and after they were aged with the crude oil (Figure 05).

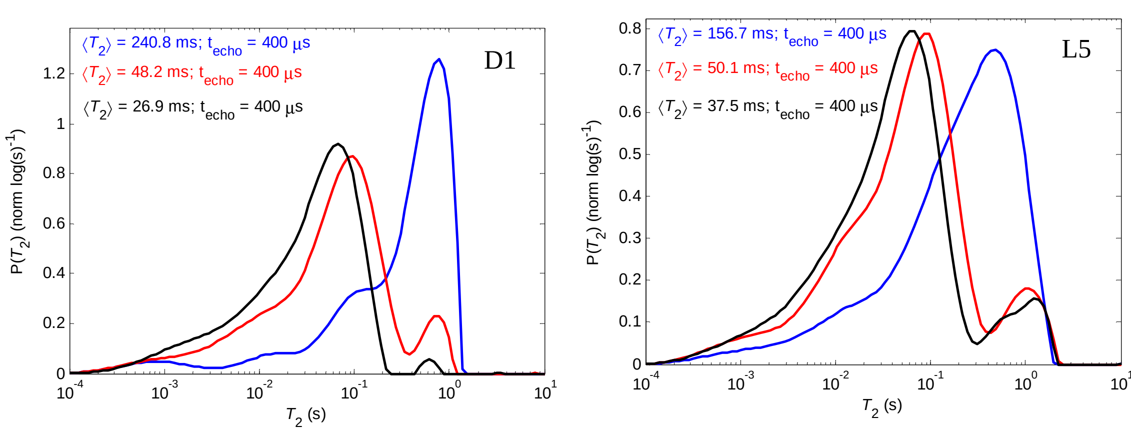

Figure 05: T_2 distributions on D1 and L5 at 100% brine saturation (blue), before aging (red) and after aging (black).

The leftward shift of the black T_2 curves relative to the red T_2 curves is a direct result of aging or decreasing water-wet nature of the plugs. T_2 log mean decreases from 48.2ms to 26.9ms and from 50.1ms to 37.5ms for D1 and L5 respectively. The change in the wettability of the two plugs was further confirmed through the USBM and A-H wettability indices (Table 05).

	USBM wettability index	Amott-Harvey wettability index
D1 dolomite	0.05	-0.02
L5 limestone	0.27	0.34

Table 05: USBM and Amott-Harvey wettability indices on D1 and L5 after aging.

The comparison of the wettability indices estimated through USBM and A-H methods, for the two plugs after conventional cleaning, extensive cleaning and after they were aged for 41 days shows that the plugs became less water-wet after being aged. The dolomite reached a neutral-wet state whereas the limestone passed from strongly water-wet to slightly water-wet condition.

SUMMARY AND CONCLUSIONS

Two different series of experiments were carried out to assess the aging process in carbonate (limestone and dolomite) using resistivity and NMR T_2 measurements. The main conclusions drawn from these experiments are given below:

- Conventional Soxhlet cleaning was found to be non-effective to make carbonate samples strongly water-wet, especially for the dolomite plug. Flow through core cleaning should be preferred to clean the carbonate plugs, essentially dolomite rocks.
- The presence of asphaltenes in the crude oil altered the wetting state of both plugs. The first deposition of asphaltenes occurred at the stage of plugs de-saturation, explaining values of n higher than 2 before the aging of the plugs.
- Immersion of plugs in crude oil with ambient measurements at different intervals of time was not found adequate due to the limitation of the experimental configuration.
- Static aging in resistivity core holder was found to be the best method, even if NMR T_2 relaxation could not be performed while aging in our configuration.
- NMR T_2 relaxation and resistivity methods showed a change in wettability. Both methods confirmed a gradual change in fluid distribution during the aging.
- Aging time can differ from one sample to another. The saturation exponent of the dolomite D1 was still increasing after 41 effective days of aging, although the saturation exponent of the limestone L5 was stable (Figure 04). Moreover, the slow progress of wettability alteration observed in this study may be attributed to the slow process of aging using stock tank oil rather than live oil.
- In the case study, it seemed to be easier to make dolomites more oil-wet than limestones. The role of mineralogy or chemistry can possibly explain this observation.
- The carbonate plugs should be at same oil saturation and under same experimental conditions for all wettability estimating methods to determine the real effect of rock mineralogy.

To conclude, the wettability alteration while aging can be identified and monitored in the lab using both resistivity and NMR T_2 relaxation measurements. Although a lot of experimental work has already been performed in the past by different labs and research institutions, additional work needs to be done to understand the dynamic mechanisms involved in the aging process of carbonate rocks.

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