WETTABILITY AS A FUNCTION OF PORE SIZE BY NMR

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ABSTRACT

Reservoir wettability affects fluid distributions, capillary pressure, electrical properties, relative permeability and ultimate hydrocarbon recovery. In the petroleum industry, laboratory measurements based on capillarity theory such as the Amott-Harvey test (AH) and U.S. Bureau of Mines (USBM) test are used to describe rock wettability. Wettability indices derived from these two methods are based on different physics and calculations, and frequently do not provide consistent results. This paper describes a wettability characterization method based on NMR measurements [1]. NMR is uniquely sensitive to surface wetting conditions of oil and water at the pore scale due to surface relaxation effects on NMR relaxation (T_2). A new NMR wettability inversion algorithm, as well as a new approach to the underlying forward model for a mixed wet and mixed saturated pore spectrum will be presented in this paper. Results from these new techniques are shown on a set of reservoir and outcrop carbonate core-plugs with a variety of laboratory controlled saturation and wettability conditions, along with a variety of core cleaning preparation techniques. From *a priori* knowledge of the wettability state of the rock samples tested, NMR wettability index developed in this study is believed to be more representative than the industry standards of AH and USBM methods. Consequently, it could become the measurement of choice for reservoir wettability characterization.

INTRODUCTION

Wettability is a key parameter in the reservoir description of a hydrocarbon bearing rock and governs the fluid distribution and flow in porous media [2–5], yet there are few methods for quantifying it. The two most common are the indices based on capillarity theory: (1) Amott-Harvey (AH), and (2) U.S. Bureau of Mines (USBM).

NMR is a technique that has been proved to be very sensitive to rock/fluid interfaces. The most widely used application is the measurement of the pore size distribution when the pores are 100% saturated with a single phase fluid, such as water. In such cases, the interpretation is straight forward and is based on the fact that as the pore size decreases, the surface-to-volume ratio increases, resulting in shorter (*i.e.,* enhanced) T_2 relaxation times. This technique was initially developed for cells [6] and then applied to other porous materials. It is also known that the NMR T_2 relaxation times are highly sensitive to the presence of mixed saturated (oil and water) and mixed wet pores, such as in the case of core-plugs in their native state taken from an oil zone. For these reasons, NMR should be a promising technique for determining wettability index owing to its inherent

and direct sensitivity to wettability on the pore scale [7]. Additionally, the lab NMR method can be accomplished in a fraction of the time required by the AH or USBM methods.

The goal of this work was to validate the inversion technique in the laboratory under a controlled set of conditions. Traditionally this has been done by correlating the NMR wettability index under investigation against the various industry standards [1,8], and thereby determining whether the NMR method is valid. Given the nature of the AH and USBM indices [9], such benchmarking may be misleading.

THEORY

In this section, principles used to extract wettability and saturation as a function of pore size from NMR T_2 data are provided.

The basic physics is that the NMR relaxation time T_2 of a fluid contained in a porous medium is shortened from its bulk value as a result of surface interactions. In a pore system fully saturated by a single phase, the extent of relaxation enhancement is related to the pore diameter d as $T_2 \propto d$, i.e., smaller pores correspond to shorter T_2. In the case of a mixed saturated pore system, the amount of T_2 shortening from surface relaxation for a given phase is directly proportional to the fraction of surface wetted by that phase. As a result, an unambiguous NMR wettability index for the rock can be derived from an average of the wettability across the pore system. The T_2 for a given phase in a pore also depends on the saturation of that phase in that pore. This implies that T_2 is sensitive to both wettability and saturation in the pore, as well as the pore size distribution itself.

Taking the extreme case to illustrate these principles, in a strongly water-wet scenario, when a drop of oil is forced into a water filled pore, the oil drop is isolated from the surface and therefore behaves as a bulk fluid with long T_2. Meanwhile, the T_2 of the water is shortened since the pore size for the water phase is effectively reduced by the presence of the suspended oil in the pore. A similar argument holds for the reverse situation of a water droplet suspended in an oil wet and oil saturated pore. These are the basic underlying principles which make up the forward model in the wettability model on which the inversion is based. This forward model is described in detail in ref. [8].

The input data for wettability inversion is a fully polarized NMR echo train, described as a linear combination of exponentials with different relaxation rates $1/T_2$ that carry information about the surface contact of fluids within a porous rock. Specifically, $1/T_2 = 1/T_{2bulk} + 1/T_{2S}$ where T_{2bulk} is the fluid's bulk relaxation time and T_{2S} is the surface relaxation time. The surface relaxation time can be calibrated to pore size via petrographic microscopy, mercury injection or specialized NMR experiments [17-19]. In this work, the relaxivity to oil and to water has been taken to be equal.

The method to be developed in this study does not require that a pore geometry be specified, but instead is incorporated into the model by the measured, fully water-

saturated relaxation time (T_2) distribution. The method requires NMR data for two states of the core plug (i.e., the fully water saturated state and any mixed saturated state) as well as two bulk fluid samples (water and oil). One output of the analysis is the native state T_2 distribution, which is decomposed into oil and water T_2 spectra, respectively. A second output is a wettability index as a function of the pore size spectrum. The wettability function was chosen to be the one proposed by Looyestijn in reference [8] which is monotonic. This wettability function can be averaged over the pore size distribution to provide a single wettability index for the core plug on a -1 to +1 scale (to be consistent with the traditional convention). The saturation function also follows Looyestijn [8] except in selected cases as is discussed in the advanced interpretation section.

EXPERIMENTAL METHODS

Sample description, preparation and handling

Sample selection and preparation was focused on having the widest possible range of wettabilities to be able to test the technique's applicability. The reservoir cores were originated from a carbonate reservoir in Saudi Arabia. The selected core plugs had a porosity ranging from 9.18 to 30.15 p.u. and permeability ranging from 0.5 to 2580 mD. The plugs used in this study were obtained in their native state. After making NMR, AH, USBM* and saturation measurements on the native state plugs, they were cleaned by standard Soxhlet cleaning using toluene and methanol followed by drying at 80 °C until weight stabilization and porosity and permeability measurements were made.

Plugs were then divided into three groups as follow: four plugs were made strongly oil wet by fully saturating in reservoir crude and ageing; four plugs were used directly after standard Soxhlet cleaning, and eight plugs were used after forced cleaning following a procedure proposed by Masalmeh and Jing [10]. The forced cleaning consisted in the following steps: samples were placed in a Soxhlet apparatus and cleaned using hot refluxing toluene to remove the residual hydrocarbon for three weeks until the effluent became transparent. The plug samples were further cleaned using an azeotropic mixture (84 vol% chloroform, 14.2 vol% methanol and 1.8 vol% water) for four weeks until the effluent became transparent again. After Soxhlet cleaning was completed, the samples were mounted in a core holder at a confining pressure of 400 psi, and were then flooded with 100% chloroform at a back pressure of 75 psi. After flooding with nominally three pore volumes of chloroform, the core-plug was left to soak overnight. The procedure was repeated again with the next chloroform flood. In total more than 15 pore volumes of chloroform were used until the effluent became colorless. To further improve the effectiveness of forced cleaning, alternating solvents (Chloroform, THF, and Methanol) were used in the core flood. Given the complexity and challenges associated with cleaning carbonate rocks, Indiana Limestone outcrops were incorporated into the study to ensure fully water wet plugs were represented.

Fluids used for wettability measurements

For the native state plugs, crude oil from the same reservoir as the rock sample was used for the entire AH and USBM* cycles as well as for the NMR measurements. For the

conditioned plugs, to avoid further ageing that may occur during the AH and USBM* measurements, the crude oil used for ageing was substituted with a synthetic oil, Soltrol, before starting the standard drainage and imbibition cycles described in the next section. Potential interactions between crude oil and Soltrol was checked to ensure data quality. The different starting points for the aged samples (fully oil saturated) and for the cleaned samples (fully water saturated) ensured that the fluid distribution in the plugs were very different. As described later, the saturation history was considered when choosing constraints in the inversion algorithm.

AH and USBM* data acquisition

The USBM* wettability index is computed by comparing areas under the total displacement of water by oil (A_1) and the total displacement of oil by water (A_2) capillary pressure curves: which is bound by $-1 < I_{USBM*} < +1$. Note that the USBM* index [8] was used, which is different from the traditional index $I_{USBM} = \log (A_1/A_2)$. The purpose of using the USBM* index is that it is bound between -1 and +1, convenient when comparing with AH and the NMR index.

In the Amott test results, whichever fluid is spontaneously displaced in more quantity indicates the non-wetting phase. The AH index is determined from the combination of the Amott and centrifuge data by calculating the intermediate values of displacement index for water, which is bound by $-1 < I_{AH} < +1$.

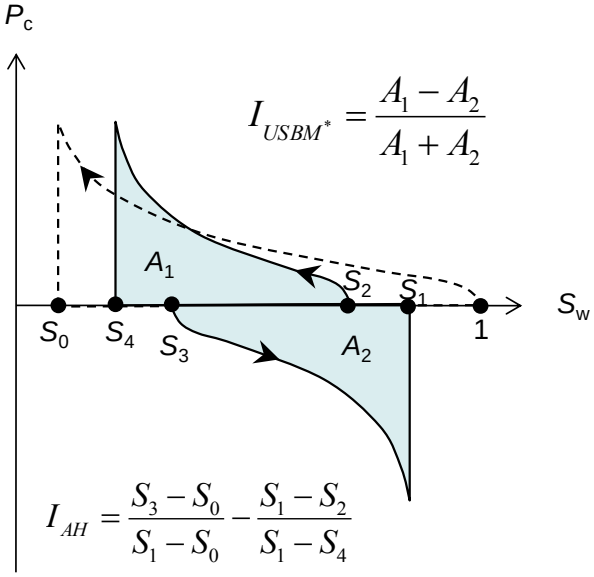

Figure 1: Pc curve illustration and definition of the areas and saturations used to compute I_{AH} and I_{USBM*}. Different parts of the capillary pressure curve measurement are shown: S_0 to S_3 represents spontaneous imbibition of water after primary drainage; S_3 to S_1 represents waterflooding; S_1 to S_2 represents spontaneous oil imbibition; and S_2 to S_4 represents forced oil imbibition. Note that S_4 (the forced oil end point) often is not the same as S_0 (the end point of primary drainage and the starting point of spontaneous imbibition). The definition of I_{AH} is based on S_0.

For both I_{AH} or I_{USBM*}, values of −1 and +1 indicate strongly oil-wet and strongly water-wet wettabilities, respectively. It is generally accepted that values below -0.3 are considered preferentially oil-wet, whereas values greater than +0.3 are considered preferentially water-wet. Values of around 0 are mixed-wet.

NMR data acquisition

The NMR measurements were performed at each extreme point of the AH cycle, namely irreducible water saturation and residual oil saturation [20] given by S_4 and S_1 in Figure 1, respectively. All the experiments were performed at ambient conditions on a 2 MHz NMR spectrometer from Oxford Instruments. The T_2 relaxation time measurements were performed using the CPMG sequence [11,12] with an inter-echo time of 400 µs. A total of 25000 echoes were acquired for an overall CPMG duration of 10 s. The wait time between successive scans was 13 s to allow full recovery of the magnetization even of the longest bulk components. Although not required by the inversion, the D-T_2 data (correlation between diffusion and T_2) and profile (1D porosity image) along the length of the plug were also measured. These data were useful in visualizing the wettability hypothesis and as a quality control. The signal-to-noise ratio was always higher than 250 for the T_2 data and 150 for the D-T_2 data.

QUALITATIVE INTERPRETATION FROM 2D NMR

The D-T_2 data from a representative case is presented in Figure 2. Three D-T_2 distributions are presented in this plot: (1) the bulk water peak in blue, (2) the bulk dead crude oil distribution in brown, which lies along the diagonal correlation line, and (3) the native state data for this plug shown in red. The 2 prominent features for the native state data, in red, are (1) the leftward shift of the signal from the bulk water peak along the water line, which corresponds to water wetting the rock, and, (2) a similar shift for the oil signal, which corresponds to crude oil wetting the rock. It is also useful to look at the T_2 and D projections shown on the bottom and to the right. The arrows in the T_2 projections show how wetting causes the bulk fluid responses to shift to the left, i.e., to shorter T_2, as a result of surface relaxation. What is significant here is that both fluids are found to shift to the left, which indicates that both fluids are to a certain extent wetting the pore surface. Therefore according to D-T_2, this native state plug is mixed-wet, which agrees well with the native state wettability indices (NMR, AH, USBM*), which range between -0.3 and -0.2 for this case.

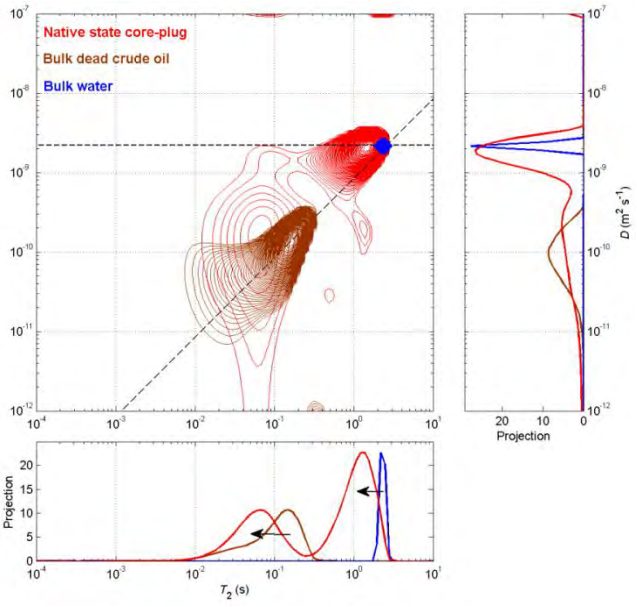

Figure 2: D-T_2 map for a selected representative plug in the native state (red), the bulk water peak (blue), and the bulk dead crude oil distribution (brown). All data were taken at ambient conditions. The horizontal dashed line corresponds to the water line, while the diagonal dashed line corresponds to the dead crude oil correlation line as described by Lo *et al.*[16] Also shown are the T_2 (bottom) and D (right) projections of the 2D map. The arrows in the T_2 projection panel illustrate how both fluids are wetting the surface, i.e., that the plug presented here is mixed-wet.

ADVANCED INTERPRETATION: WETTABILITY INVERSION

Forward Model

The amplitude of each exponential in the NMR forward model is the partial porosity $P_i = P(T_{2S,i})$ with a particular surface relaxation time $T_{2S,i}$. The partial porosities are normalized with total porosity such that their sum always equals to one. For each partial porosity, we define partial saturation S_i as the volume fraction of water occupying that partial porosity. Similarly, partial wettability W_i is the surface fraction of water-wetted pore wall within the i^{th} partial porosity. Both partial saturation and wettability are bound as $0 \leq Si \leq 1$ and $0 \leq Wi \leq 1$. Figure 3 illustrates how S_i and W_i can vary inside a triangular pore during the geological lifetime of a reservoir rock during drainage and imbibition processes [13].

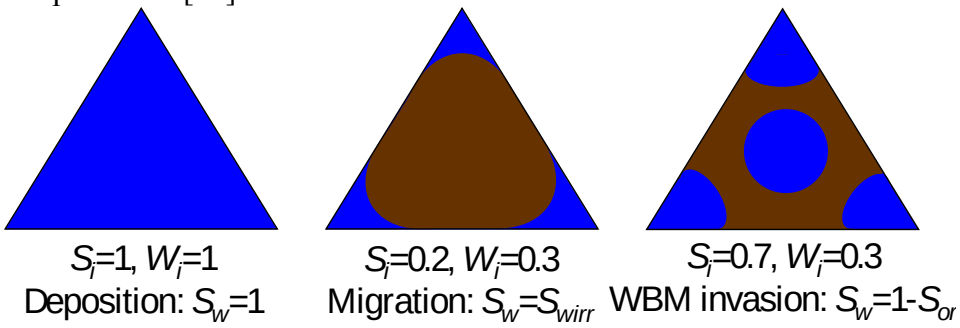

Figure 3: Schematic representation of the different states of a triangular pore after deposition (left), oil migration (middle), and flushing caused either by WBM (Water Based Mud) filtrate invasion or water injection (right). S_i is defined as the volume fraction of water in that i'th pore (bound by $0 \leq S_i \leq 1$), while W_i is the surface fraction of the pore wall that is wetted by water (bound by $0 \leq W_i \leq 1$).

The overall saturation is the average of S_i weighted by the pore size distribution, while the similarly calculated NMR wettability index is scaled to the traditional -1 to 1 interval.

The water saturation is bound by $0 \leq S_{NMR} \leq 1$, where $S_{NMR} = 1$ implies 100% water saturated rock, while $S_{NMR} = 0$ implies 100% oil saturated rock. The wettability index I_{NMR} is bound by $-1 \leq I_{NMR} \leq 1$, where $I_{NMR} = 1$ implies an entirely water-wet rock, $I_{NMR} = -1$ implies an entirely oil-wet rock, and $I_{NMR} \approx 0$ implies a neutral wettability of a mixed-wet rock. Generally, the model parameters for the inversion are the partial porosities P_i, partial saturations S_i and partial wettabilities W_i which is an extremely ill-posed problem. However, we can obtain the pore spectrum P_i by a subsequent NMR measurement on a force-cleaned, fully water-saturated sample. Moreover, the number of free parameters associated with S_i and W_i can be further reduced by representing the saturation S_i and wettability W_i with a function as suggested by Looyestijn *et al.* [1,8]. In this work, two types of functions were considered:

$$X_i = \frac{X_0 - X_\infty}{1 + \left(\frac{T_{2S,i}}{T_{RX}}\right)^{\alpha_X}} + X_\infty \tag{1}$$

$$S_i = Ae^{-\frac{\left(T_{2S,i} - T_{2c}\right)^2}{w^2}} \quad \text{(a)} \qquad S_i = 1 - Ae^{-\frac{\left(T_{2S,i} - T_{2c}\right)^2}{w^2}} \quad \text{(b)} \tag{2}$$

(1) Where X can be S for saturation or W for wettability: the original four parameter function proposed; is monotonic and characterized by two plateaus at short and long T_{2S}, (whose amplitudes are determined by X_0 and X_∞)and a smooth transition between the two plateaus at $T_{2S}= T_{RX}$ and of slope α_X Figure 4. These functions reasonably match the expected distributions of fluids and wettabilities in native core plugs.

(2) A Gaussian function for the saturation S_i allowing a non-monotonic saturation profile along the pore spectrum. Non-monotonic behaviour in saturations may occur as a result of multiple drainage and imbibition events caused by tectonism and/or oilfield development. (a) and (b) are the two cases of positive or negative Gaussian depending if the initial saturation was Sw=0 or Sw=1 respectively. A is the amplitude, w the width and T_{2c} the center.

A representative example of the forward model and inversion output for S_i and W_i across the pore spectrum data is given in Figure 4. The pore cartoons illustrate how the big pores (right) would look like according to S_i and W_i, and similarly for the small pores (left).

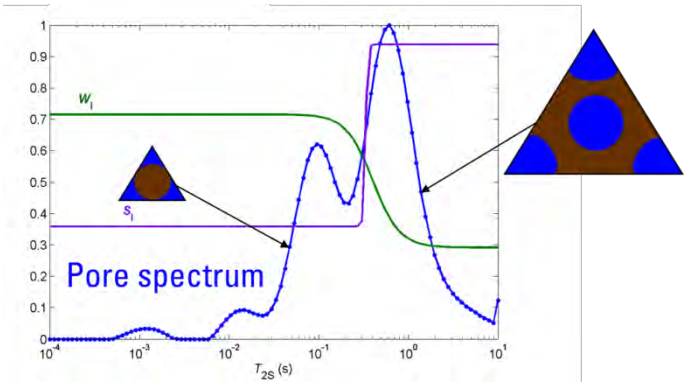

Figure 4: A representative example of the forward model and inversion output. S_i (purple line) and wettability W_i (green line) across the pore spectrum data (blue dots). The pore cartoons illustrate how the big pores (right) would look according to S_i and W_i, and similarly for the small pores (left). Note that the pore cartoons are meant as an illustration and do not correspond to the precise values of S_i, W_i, and pore size.

Inversion

The theory of surface relaxation, including the one for wettability is presented in terms of relaxation times T_2. This makes the representation clearer and easier to understand, even though the acquisition and inversion are performed in the time domain. To express the results in the T_2 domain, an inversion (Inverse Laplace Transform, or ILT) is required [14]. This processing method is known to be problematic and without a unique solution. This problem is solved by the use of a regularization parameter, which imposes a certain degree of smoothness to the T_2 distribution. This regularization parameter allows a stable solution at the price of details in the T_2 distribution. The use of a regularization parameter has proved to be fairly reliable; however, there is still an open question on the possibility of improving the data processing. Along these lines we avoid the Laplace Inversion altogether and invert in the time domain following the technique described in Ref. [15]. The outputs from the inversion are the 4 parameters determining the wettability W_i, and the 4 (or 3) parameters determining the saturation S_i, as function of pore size and the water and oil T_2 distributions.

The fundamental idea behind the inversion used in this study is an intelligent grid search method. This has the advantage of providing many solutions indexed by their overall

quality of fitness to the data. The strength of the inversion is that one may introduce a constraint, as described above, on the saturation S_i across the pore spectrum to stabilize the inversion for wettability W_i across the pore spectrum. The saturation constraint is based on the saturation history of the rock as well as some petrophysical insight into the problem. Table 1 shows four common reservoir scenarios dictating helpful saturation constraints without any loss of generality:

Table 1: Description of the different constraints used in the inversion and their petrophysical justifications.

Saturation history and petrophysical insight	Saturation constraint
Strongly oil wet rocks with the cycle $\mathbf{S_W=0\rightarrow(1-S_{or})}$ have been first saturated and aged with oil, and then water flooded to S_{or}. Small pores are full of oil, while big pores contain more water.	S_i monotonically increasing
Strongly water wet rocks with the cycle $\mathbf{S_W=1\rightarrow S_{wirr}}$ have been saturated first with water, and then oil flooded to S_{wirr}. Small pores are water filled, while big pores have more oil.	S_i monotonically decreasing
Water-wet rocks with two cycles $\mathbf{S_W=1\rightarrow S_{wirr}\rightarrow(1-S_{or})}$ and oil-wet rocks with two cycles $\mathbf{S_W=0\rightarrow(1-S_{or})\rightarrow S_{wirr}}$ have a Gaussian saturation profile where irreducible fluids in small pores $T_{2S} < 10^{-1}$s have not been displaced. Native state plugs affected by drilling mud fall into the first of these categories.	S_i is a Gaussian profile centered in the large pores corresponding to $T_{2S} > 10^{-1}$s
Rocks saturation can be obtained independently from other techniques such as the amount of fluid production.	S_{NMR} lies within +/– 5% of an independent measurement

NMR WETTABILITY INVERSION RESULTS

Three examples covering a wide variety of wettability types and saturation states are shown below:

Strongly oil-wet rock $\mathbf{S_W=0\rightarrow(1-S_{or})}$

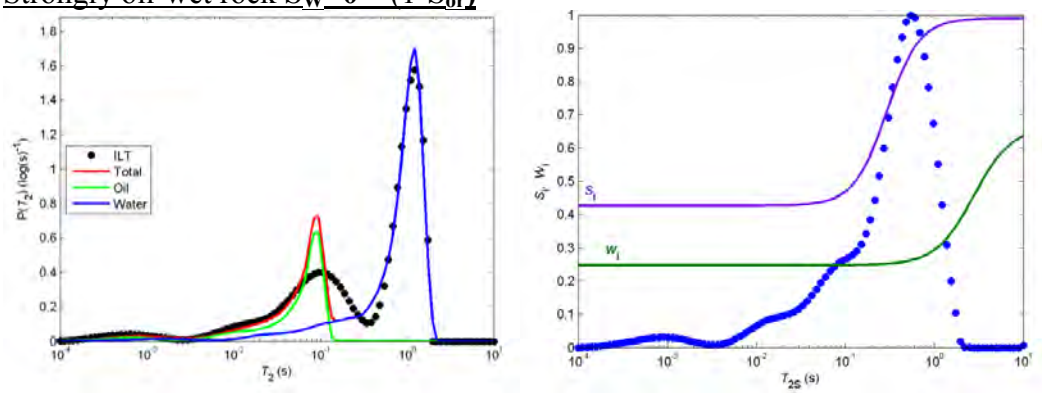

Figure 5: (Left) the T_2 distribution from ILT (black dots) and the oil and water distributions obtained from the inversion, as well as the sum of these distributions. (Right) The wettability (W_i) and saturation (S_i) functions obtained with the inversion together with the pore spectrum from $S_W = 1$ (Blue dots). The saturation steps at which the plug has been submitted before measuring the NMR response are $S_W=0\rightarrow(1-S_{or})$. The overall water saturation is $S_{NMR} = 0.74$ and the wettability index is $I_{NMR} = -0.47$.

Figure 5 (left panel) shows the presence of oil in the smaller pores ($T_2 < 10^{-1}$s) and water in the larger ($T_2 > 10^{-1}$s). This is consistent with the saturation history of the plug, that started fully oil saturated and has been de-saturated by centrifuging with water. Note that the total (oil + water) fit looks more "spiky" than the ILT. This total fit is not an issue as such since the misfit from the inversion is calculated in the time domain (not shown), and as already noted, the ILT tends to artificially smooth out the solution in the T_2 domain as a result of regularization. In the right panel, the saturation function S_i reflects the fluid distribution observed in the left panel. The wettability function W_i stays very low for most of the range of T_{2S} as expected for an oil wet plug. The W_i increases slightly in the range of T_{2S} values where there are no data.

Strongly water-wet rock **S_W=1→S_{wirr}**

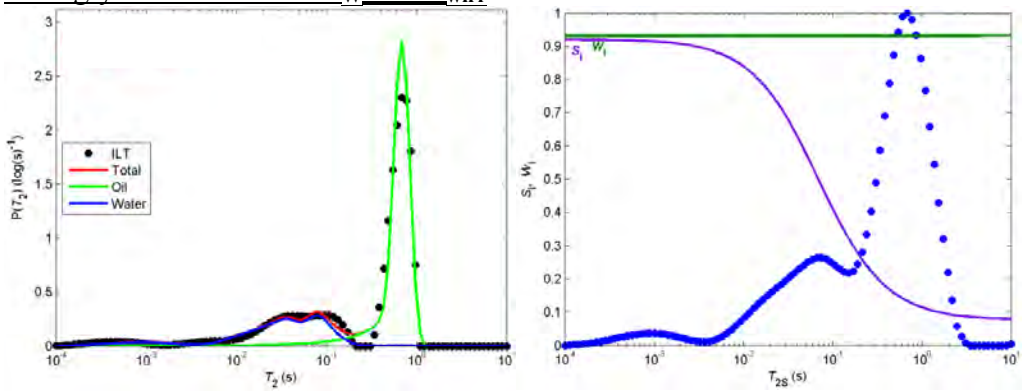

Figure 6: Same as Figure 5 but for a water wet plug at S_{wirr}. The saturation steps at which the plug has been submitted are S_W=1→S_{wirr}. The overall water saturation is S_{NMR} = 0.29 and the wettability index is I_{NMR} = +0.86.

Figure 6 is the situation that is completely opposite to the one shown in Figure 5. The oil and water distribution ranges are inverted and the wettability function is a constant at high values. These results are supported by the fact that the saturation history of the plug is opposite to that of the plug shown in Figure 5; the core-plug started as 100% water saturated and was partially desaturated by oil. The forced cleaning used for this core plug also is consistent with the high wettability value.

Water-wet rock **S_W=1→S_{wirr}→(1-S_{or})**

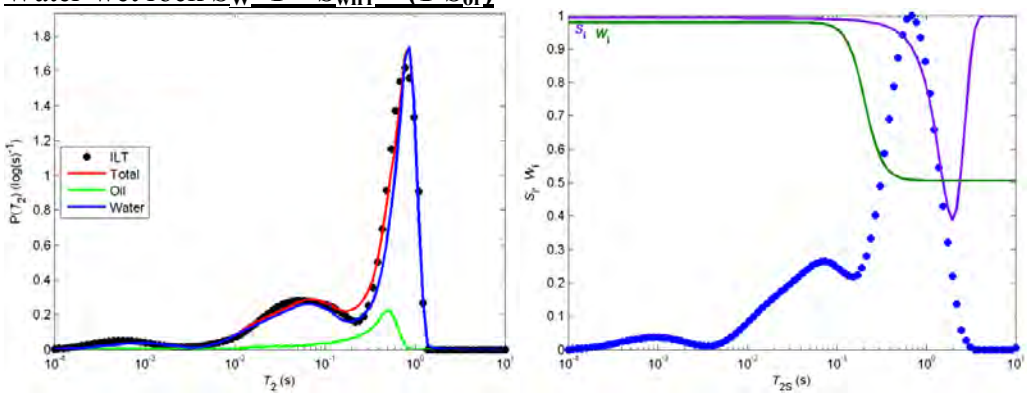

Figure 7: As Figure 5 but for a water wet plug at S_{or}. The saturation steps at which the plug has been submitted are S_W=1→S_{wirr}→(1-S_{or}). For this solution the overall water saturation is S_{NMR} = 0.89 and the wettability index from I_{NMR} = +0.32.

Figure 7 presents the cases in which the plug has been exposed to two changes of saturation $S_W=1\rightarrow S_{wirr}\rightarrow(1-S_{or})$ for the water wet plug. Because of this double change in saturation, the inverted Gaussian profile for S_i was adopted. In this case the inverted Gaussian function helps in resolving oil trapped in the intermediate size pores. Similarly, for the oil wet plug exposed to two changes $S_W=0\rightarrow(1-S_{or})\rightarrow S_{wirr}$ (data not shown), the fluid distributions show water trapped in the intermediate pores corresponding to a Gaussian (not inverted) profile.

COMPARISON OF WETTABILITY INDICES

A comparison of the various indices (NMR, AH, USBM*) versus the various core cleaning methods was made. As expected the forced cleaning and outcrops samples are strongly water wet, which is the case according to the NMR results shown in Figure 8 (top) (the one sample with I_{NMR} of about 0.3 is tight, difficult to clean to water wet due to preparation). The AH and USBM* results shown in Figure 8 (middle) and 8 (bottom) indicate less strongly water wet plugs, which may call into question the AH and USBM* techniques. A discussion of issues affecting these techniques is reported in reference [9]. In that context we now focus on the NMR results. The native state plugs show strongly oil wet with I_{NMR} down to -1, Figure 8a, while for the aged samples I_{NMR} is limited to about -0.6. The aged plugs were fully saturated with crude oil for a limited time (4 months), while the native state plugs have been aged over geological time at saturation closer to S_{wirr}. It is not thought that longer ageing time or the higher oil saturation would be more effective in the ageing process. The most likely reason for the inconsistency between the technique results is that the NMR inversion is more accurate on the oil wet side when the bulk fluids have a larger contrast in T_{2bulk}.

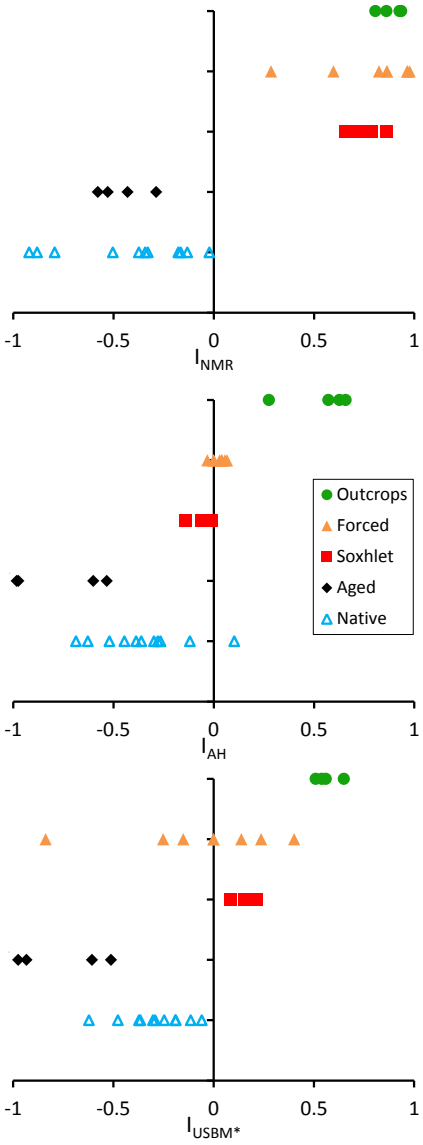

Figure 8: Different wettability indices (x axis) as a function of the sample preparation (y axis): native state (empty blue triangles), aged cores (black diamonds), soxhlet cleaned (red squares), forced cleaned (full orange triangles) and outcrops (full green circles). Wettability index from (top) NMR, (middle) Amott-Harvey, and (bottom) USBM* are shown.

CONCLUSIONS

A new approach to the NMR wettability inversion algorithm as well as a new approach to the underlying forward model, applicable over wide range of saturation and wettability

conditions has been presented. The approach takes the saturation history of the plug as well as some petrophysical insight into account to formulate a saturation filter on the set of solutions from the inversion, thereby stabilizing a solution set for the wettability across the pore spectrum. The approach incorporates a NMR inversion technique (which does not require an Inverse Laplace Transform or a regularization parameter), which further stabilizes the inversion algorithm for the wettability across the pore spectrum. These new techniques have been demonstrated on a set of reservoir and outcrop carbonate core-plugs with a variety of laboratory controlled saturation and wettability conditions, along with a variety of core cleaning preparation techniques. From *a priori* knowledge of the wettability state following forced cleaning and outcrops, the NMR wettability index is shown to be more representative than the industry standards for water wet rocks. Consequently, the NMR wettability index could be the measurement of choice for reservoir wettability characterization.

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