3D-NMR, simultaneous determination of saturations and wettability

Benjamin Nicot, Georges Ibrahim, Gregory Dubes
TOTAL

This paper was prepared for presentation at the International Symposium of the Society of Core Analysts held in Vienna, Austria, 27 August – 1 September 2017

ABSTRACT

The knowledge of saturations and wettability is key in the assessment of the potential of an oil field. Saturations control the hydrocarbon volume in place, while wettability has a strong influence on the recovery. While many techniques are available to derive saturations, both from laboratory and log data, techniques for measuring wettability are limited to the laboratory and are expensive and time consuming.

Traditionally, laboratory measurements of wettability in the laboratory are based on spontaneous and forced drainage and imbibitions (USBM and Amott indexes). Many attempts have been presented in the past to replace these long and expensive measurements by other techniques. Among these techniques, NMR has a strong potential thanks to its natural sensitivity to saturations and wettability. On the one hand, NMR is sensitive to saturations through DT2 maps. On the other hand, T_1T_2 maps are sensitive to wettability. In these maps many authors have already reported that the wetting fluid has a high T_1/T_2ratio. However one question remains: which fluid is the fluid that exhibits a high T_1/T_2 ratio? The difficulty comes from the fact that there is no fluid identification in the T_1T_2 maps.

In this paper we demonstrate the interest of acquiring 3 dimensional NMR: Longitudinal relaxation time T_1, transverse relaxation time T_2 and Diffusion D. The correlated information between the three NMR parameters allows the simultaneous interpretation of the data in terms of pore size distribution, saturations (thanks to Diffusion), and identification of the wetting fluid (thanks to T_1). This method has been developed on bulk fluids, and successfully tested on real rocks.

INTRODUCTION

Nuclear Magnetic Resonance (NMR) has been used in the oil industry since the early beginning of NMR. Soon after the discovery of NMR by Bloch and Purcell in 1946, the oil industry recognized the interest of this technique and launched laboratory NMR studies of fluids in porous media. Since the first earth's-field NMR logging tool developed by Chevron in 1960, NMR measurements have evolved a lot. NMR can be used to measure different parameters: Quantity of signal, relaxation times T_1 and T_2, diffusion coefficient.

NMR first answer of interest to the oil industry is the quantity of signal. The quantity of signal measured by an NMR device is proportional to the amount of Hydrogen nuclei present in the sample. Therefore, on a rock containing liquids of know Hydrogen density, NMR measures porosity.

Relaxation times are used to get structural information on the rock. Since the discovery of reduced fluid relaxation times in porous media, NMR has been used as a pore size indicator [1], and NMR permeability predictors such as the Schlumberger-Doll Research [2] and the Timur-Coates [3] have been proposed.

Since the 2000's and the pioneering work of Venkataramanan et al.[4], two dimensional NMR has arisen, allowing to correlate two different NMR parameters. The most widely used NMR 2D map has been the Diffusion-T_2 map, allowing the determination of the water and oil saturations thanks to their diffusion contrast. The interest of performing a 2D measurement has been tremendous and dedicated logging tools have been developed to allow the acquisition of the 2D Diffusion-T_2 maps [5].

Meanwhile, the industry recognized the natural sensitivity of NMR to wettability and many papers have been published demonstrating the potential of using T_2 for getting wettability information [6][7][8]. However, the work from Korb et al. [9] demonstrated that a measurement of T_1 is very sensitive to wettability and Valori et al. [10] published a very strong dependence of the T_1/T_2 ratio of oil with wettability. The remaining difficulty being: how to measure the T_1/T_2 ratio of oil.

Here we propose to combine the benefits of the Diffusion-T_2 map to obtain saturations with the potential of the T_1T_2 map to obtain wettability. For this purpose we developed a 3D inversion allowing the simultaneous acquisition and inversion of T_2, T_1 and diffusion. We first give the principle of the method, and then demonstrate the concept on bulk fluids. Last, we provide experimental examples acquired on outcrop and reservoir rocks.

PRINCIPLE OF 3DNMR

The 3D NMR sequence used in this paper consists of three main segments. The first one is a simple inversion-recovery with a waiting time of ΔT_1. This segment is followed by a pulsed gradient stimulated echo of gradient **G** for diffusion encoding. Finally a classical CPMG sequence is used to characterize the T_2 distribution. The overall pulse sequence is presented in Fig.1 where K_{T1} is the longitudinal magnetization kernel, K_D is the diffusion kernel and K_{T2} is the transverse magnetization kernel.

The problem is to find the relaxation times T_1, T_2 and the diffusion coefficient D, knowing the magnetization M. Mathematically, the problem can be written in the following way:

$$\mathbf{M(i,j,k)} = \sum_{\mathbf{l,m,n}} \mathbf{K_{T_1}(i,l)K_{T_2}(j,m)\,K_D(k,n)F(l,m,n)} + \boldsymbol{\varepsilon}\mathbf{(i,j,k)} \quad (\mathbf{1})$$

Where $M(i,j,k)$ denotes the magnetization resulting from the i^{th} ΔT_1, the k^{th} G, and measured at the j^{th} nTE. The discretised kernels and the discretised density function F are calculated using the discretisation over T_1, T_2 and D.

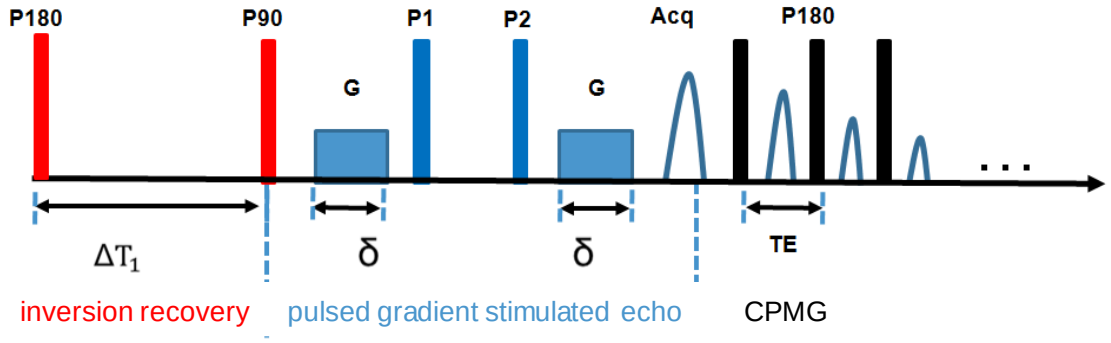

Fig.1: The 3D NMR sequence: ΔT_1 is the inversion time in the inversion recovery segment; in the PGSTE segment δ and G are the duration and amplitude of the gradient pulse; in the CPMG segment TE is the inter echo time. This sequence is run by varying ΔT_1 for encoding T_1 and varying G for encoding diffusion. (time intervals not to scale). Typical parameters are presented in Table 2.

The objective of the inversion is to find the optimal F that best fits the data M. In 2D, the problem can be written as a matrix product and solved using the method proposed in Venkataramanan et al. [4]. In 3D the matrix product no more holds. The mathematical formulation of the problem is given in [11]. We have proposed a way to solve this problem [12] based on a relatively simple strategy detailed in Figure 2:

1. Transform the 3D problem into a 2D problem by combining 2 Kernels. The key idea is to transform a third degree tensor into a second degree tensor by unstacking its elements into the first horizontal plane.
2. Solve the 2D problem using the classical approach proposed in [4],
3. Transform the 2D solution into a 3D solution

This way, the mathematical problem presented in equation (1) (that was impossible to solve) is transformed into a more simple 2D problem defined in equation (2)

$$\mathbf{R(M)} = (\,\mathbf{K_D} \otimes \mathbf{K_{T_1}})\,.\,\mathbf{R(F)}\,.\,\mathbf{K'_{T_2}} \quad (\mathbf{2})$$

All the details of the inversion strategy are given in [12].

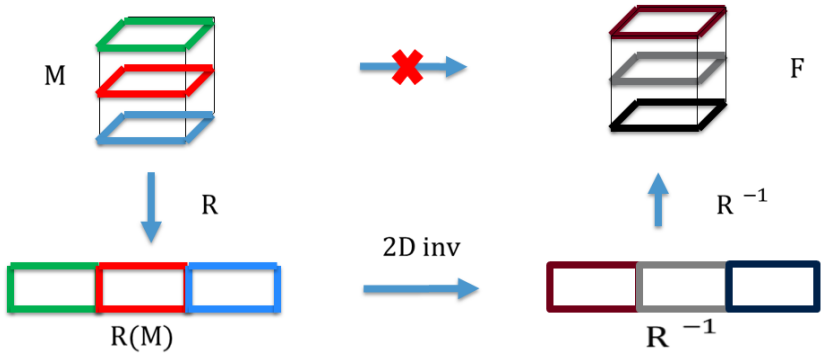

Figure 2: Principle of the 3D NMR inversion: We first transform the problem into a 2D problem by unstacking the 3D tensor into a 2D matrix, solving the problem in 2D, and then re-stacking the results into a 3D matrix to get the solution

EXPERIMENTAL

The NMR acquisitions were performed on a 2MHz Geospec from Oxford Instrument equipped with 1D gradients. Typical pulse length is 13µs for the 90° pulse and 6ms for gradient pulse. Typical inter-echo time is 200µs, and signal is acquired to a minimum SNR of 40, using the sequence described in Fig.1 and the parameters given in

Table 2. Typical acquisition time can range from 10hours on bulk fluids to a few days on rock samples.

RESULTS ON BULK FLUIDS, PROOF OF CONCEPT

The concept of 3D (T_1-T_2-D) NMR was tested on a sample made of bulk fluids: 40% of $MnCl_2$ doped water and 60% of synthetic oil. Since the two fluids have nearly the same hydrogen index, the apparent porosity of the sample is 100%. Its NMR properties are summarized in Table 1.

	$T_1(s)$	$T_2(s)$	$D(m^2s^{-1})$
Water	0.07	0.05	2.10^{-9}
Oil	0.2	0.2	10^{-10}

Table 1 : NMR properties of the fluid sample

On this sample, the acquisition of a 3D NMR cube was performed using the sequence described in Fig.1 and the parameters given in

Table 2.

	Min	Max	Nb of values
ΔT_1	5ms	3s	25
G	0	20 Gauss/cm	25
nTE	0	1s	5000

Table 2: Acquisition parameters

The result of the 3D inversion gives a cube of intensities; the axis of the cube being T_1, T_2 and D. An example of the cube is given in Figure 3. For practicity, the projections of the cube on the D-T_2 map and on the T_1-T_2 maps are displayed. In these projections, the water and oil signals can be clearly identified from the D-T_2 map: the round signal (signal A on Figure 3) corresponds to water. In the T_1-T_2 map this signal exhibits a high T_1/T_2 ratio; this is due to the use of $MnCl_2$ as a dopant. The elongated signal (signal B in Figure 3) corresponds to the mineral oil.

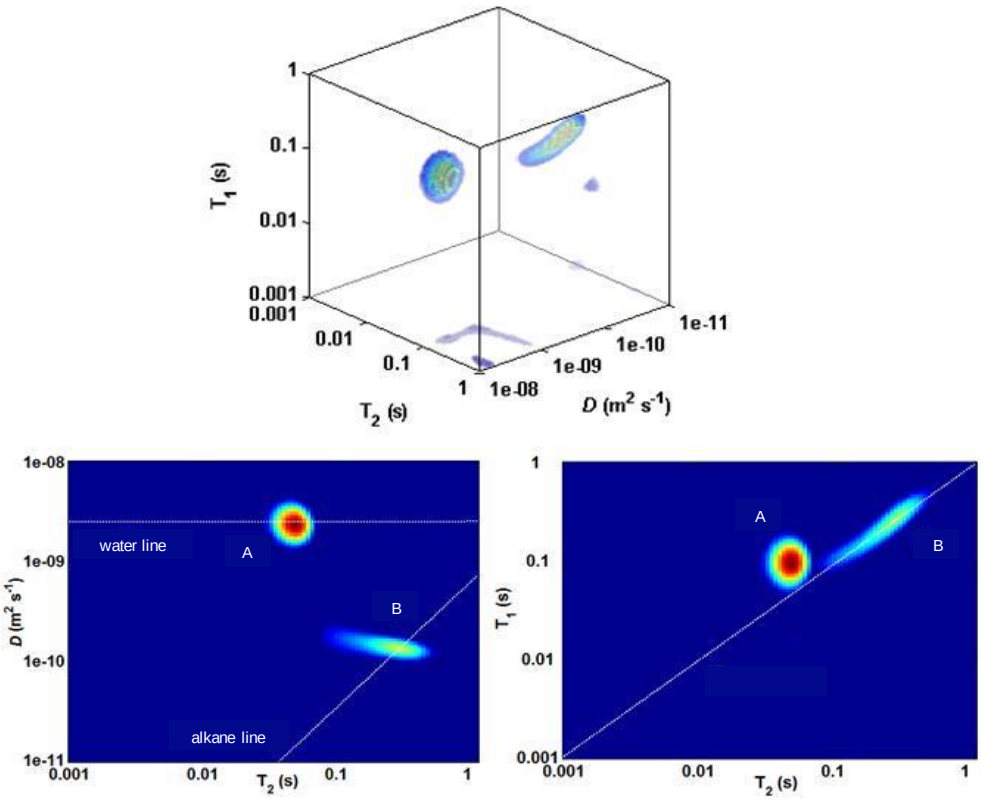

Figure 3: 3D inversion results carried on sample (2). Top panel shows a cube representation of the solution with a resolution of (100,100,100). Bottom left and right panels and represent the projection of the 3D solution on theDT2 and T1T2 planes.

RESULTS ON OUTCROP ROCK

This section discusses the results of the experiments on real rocks. We discuss petrophysical properties of the rock samples, crude oils properties, samples preparation and the results of processing.

Rock and crude oils properties

We present the results from one limestone plugs from the French quarry Estaillades, exhibiting bimodal porosity.

The crude oil used was a viscous crude oil (viscosity~50cP).

Samples preparation

The sample first dried in an oven heated to 80°C, and porosity ϕ=28.5p.u. was determined in the lab measuring total volume and solid volumes, using a pycnometer. The plug was then vacuumed in a cell, and then saturated with brine at 200bars to remove any air. Due to the rock type, the brine was designed to avoid degradation (0.4mol/l NaCl + 0.33mol/l $CaCl_2$).

A suite of NMR acquisitions was performed on these samples at 100% brine saturation, referred later as Sw1.

Samples were set in Hassler cells for setting irreducible water saturation (Swi). The gas confining pressure was set to 75bars, keeping a 25bars pore pressure with crude oil. The plug was slowly swept, monitoring the fluids flowing out. The injected oil volume was equivalent to 4 times the porous volumes, estimated from NMR @ Sw=1. At this stage, the sample was close to (if not at) irreducible water saturation. A second suite of NMR acquisitions was performed after dismounting from the Hassler cell, referred as SwiBA (irreducible water saturation, Before Ageing).

The sample was then replaced in Hassler cell, in the same conditions. After a quick sweep equivalent to 1 Vp, a heating phase was started in order to age the plug and change wettability. Heating jackets were set on Hassler cells and temperature was controlled to 80°C at the plug. This operation lasted 3 weeks, and included a weekly oil sweep with an oil volume equivalent to 1 Vp. A third suite of NMR acquisitions was performed after dismounting from cells, referred as SwiA (irreducible water saturation, Aged).

NMR acquisition

The complete set of NMR acquisitions consisted in: 1D: T_2; 2D: T_1-T_2 map and D-T_2 map; and 3D: T_2-T_1-D cube

As shown in Figure 4, the T_2 distribution obtained on this sample saturated with brine partly overlaps with the bulk crude oil distribution. This already can help predicting that a single T_2 experiment would not be capable of distinguishing oil from water inside this rock.

In fact, as shown in Figure 4, a T_2 experiment is indeed not able to distinguish between oil and water, neither before nor after the ageing process. However, a small shift of the main T_2 peak towards shorter T_2 can be observed during the ageing. In order to interpret this shift into a wettability change, one would have to know if the peak corresponds to a water or an oil signal.

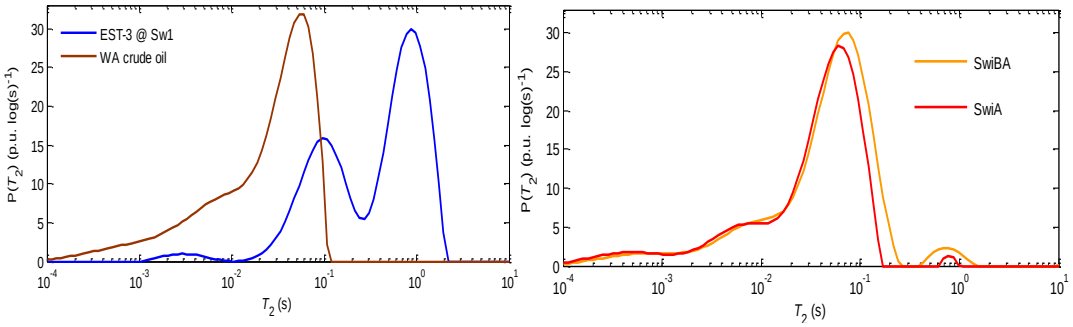

Figure 4 : Left : T_2 distribution of the Estaillade sample saturated with brine (blue) and of the bulk crude oil (brown). Right: T_2 distribution of the Estaillade sample at irreducible water saturation before ageing (orange) and after ageing (red).

In order to distinguish the NMR signals corresponding to oil and water, a diffusion contrast is required. Figure 5 presents the T_2 distribution, D-T_2 maps and T_1-T_2 maps obtained on this sample before and after ageing.
In this case, water and oil NMR signals have similar T_2, but the diffusion contrast is sufficient to separate the water and oil signals. This is the classical way to measure fluid saturations from NMR.
Looking at the T_1-T_2 map in Figure 5, a small increase of the T_1/T_2 ratio can be observed during ageing. As stated by Valori et al. [10], the T_1/T_2 ratio of the oil can be used as a proxy for wettability, however in this case it is not possible to know which fluid (water or oil) is responsible for the increase in T_1/T_2 ratio (i.e. which fluid is the most wetting fluid). The missing information is a connection between the D-T_2 map (allowing identifying the fluids) and the T_1-T_2 map (allowing the estimation of wettability).

The acquisition of 3D (T_1-T_2-D) NMR data can solve this issue. The 3D NMR cube acquired on the Estaillades sample before ageing is presented in Figure 6. The projections of the cube on the DT_2 and T_1T_2 maps are also presented. All the interest of the technique resides on the possibility to identify the oil signal on the DT_2 projection and to display the corresponding signal on the T_1T_2 projection. In this example, the oil signal has been selected and allows the determination of the T_1/T_2 ratio for the oil. The exact same methodology has been applied on this sample after ageing and the results are presented in Figure 7.

For this example, even if water and oil signal are superimposed in a simple T_1T_2 map, the use of 3D NMR allows the identification of the signals and the calculation of a mean T_1T_2 ratio for the oil. We found $T_1/T_{2oil(before\ ageing)}$=1.9 and $T_1/T_{2oil(after\ ageing)}$=2.2. According to Valori et al. [10] the T_1/T_2 ratio of the oil can be used as a proxy for a wettability index. This method has been used in Figure 10 to estimate the corresponding wettability indexes of $W_{(before\ ageing)}$=0.4 and $W_{(after\ ageing)}$=0.2.

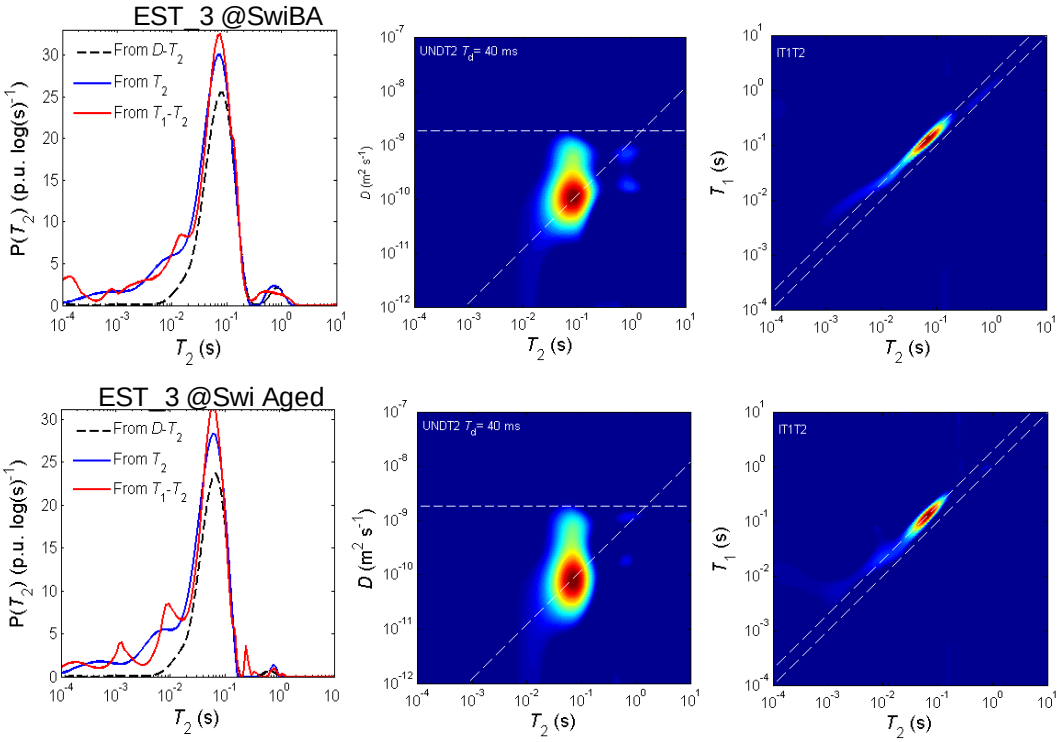

Figure 5: T_2, D-T_2 map and T_1-T_2 map obtained on the Estaillades sample before (top) and after (bottom) ageing

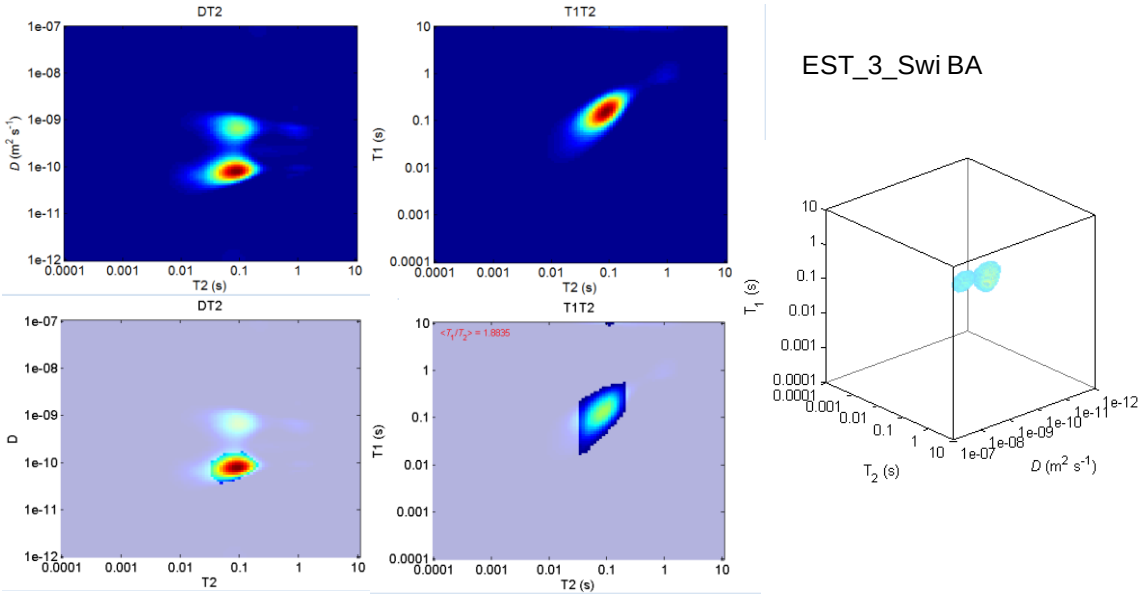

Figure 6: 3D NMR results obtained on the Estaillade carbonate at Swi before ageing. The 3D cube is presented on the right. On the left are presented the DT_2 and T_1T_2 projections. Bottom left are presented the

results of selecting the oil signal in the DT_2 projection and displaying the corresponding signal in the T_1T_2 projection.

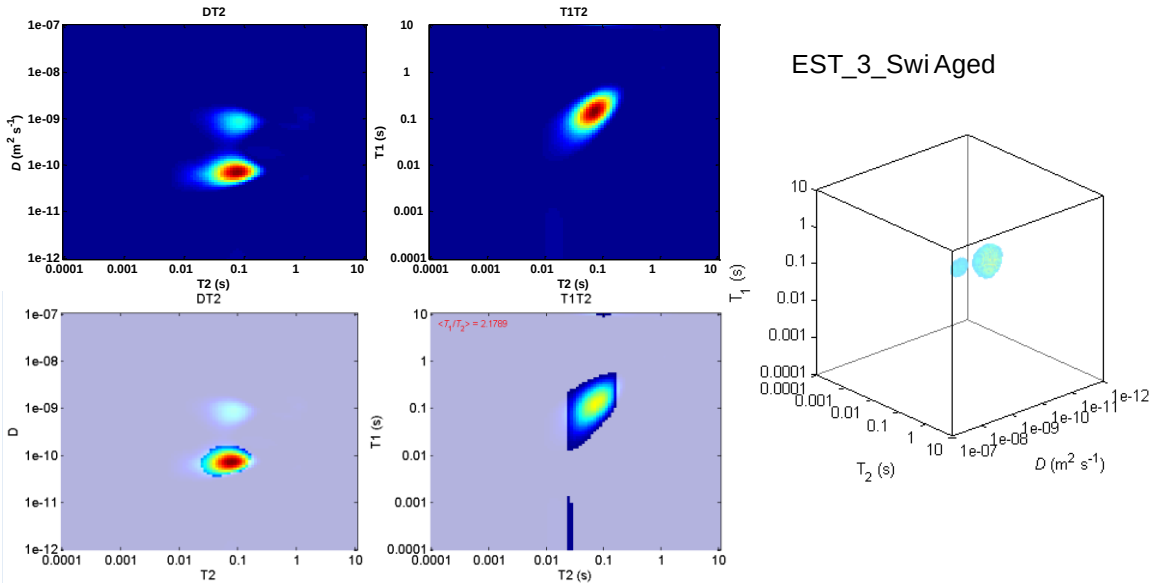

Figure 7 : 3D NMR results obtained on the Estaillade carbonate at Swi after ageing. The 3D cube is presented on the right. On the left are presented the DT_2 and T_1T_2 projections. Bottom left are presented the results of selecting the oil signal in the DT_2 projection and displaying the corresponding signal in the T_1T_2 projection.

RESULTS ON RESERVOIR ROCK

We present here the result of the 3D NMR acquisition on a real reservoir rock. This reservoir rock is a sandstone in the "as received" state, containing a light oil (~1cp). This reservoir is known to be strongly oil wet with Amott index of the order of -0.8.

On the same sample, Amott test and 3DNMR were performed. The Amott test was performed using mineral oil, after ageing at irreducible saturation in dead crude oil.

When performing the 3DNMR acquisition, presented in Figure 8, the oil and water signals can be separated from the DT_2 projection. This allows the identification of the oil signal on the T_1T_2 projection. In this case, the oil and water signal do not properly separate in the T_1T_2 projection, this is due to a lack of encoding of the T_1 using the parameters presented in Table 2 (25 points). Once the position of the oil signal is known, a more simple T_1T_2 measurement can be performed, with a much stronger encoding of the T_1. As this stage, a 40 by 40 points encoding of a 3DNMR could have been done, but would have been very time consuming. In this case, the 3DNMR unambiguously assign the signal to oil and water thanks to the diffusion encoding. Once

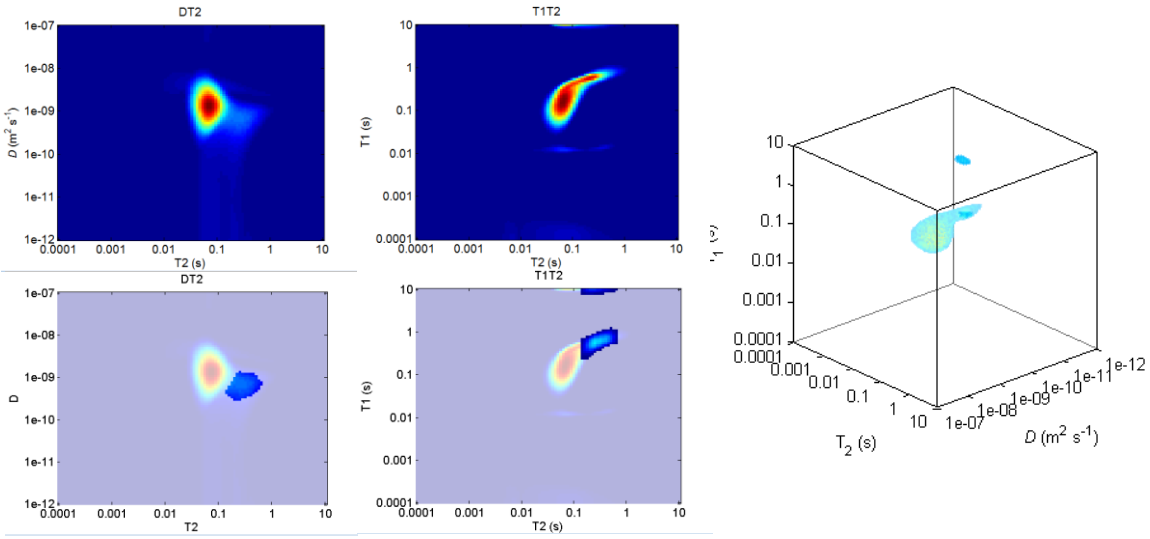

Figure 8: 3D NMR results obtained on the reservoir sandstone. The 3D cube is presented on the left. On the right are presented the DT_2 and T_1T_2 projections. Bottom left are presented the results of selecting the oil signal in the DT_2 projection and displaying the corresponding signal in the T_1T_2 projection.

In Figure 9 we present the 2D D-T_2 and T_1-T_2 maps acquired on this sample with the same parameters as in Table 2 but encoding using 40 points. This allows a much better resolution of the maps, for both DT_2 and T_1T_2. This T_1-T_2 map was then used to calculate the mean T_1/T_2 ratio for the oil. We found T_1/T_{2oil}=3.7, corresponding to a wettability index W=-0.75, following the Valori et al.[10] method presented in Figure 10.

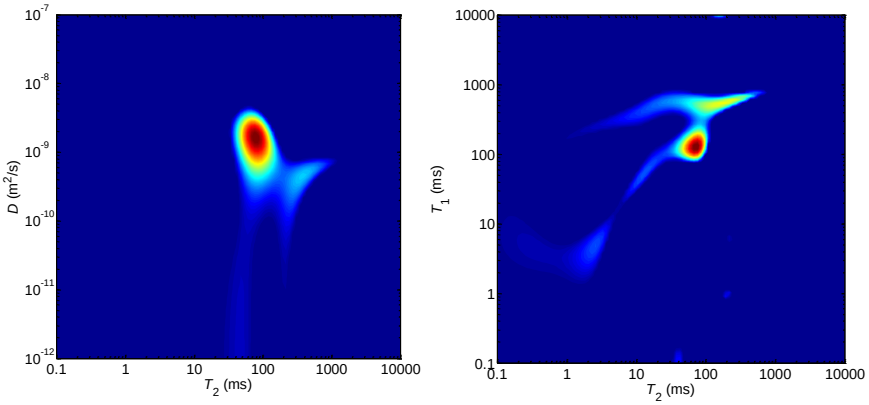

Figure 9: D-T_2 map (left) and T_1-T_2 map (right) obtained on the "as received" reservoir sandstone

In this example, it is arguable that a similar information could have been obtained with 2D maps only. In fact the use of 3DNMR in this case was to unambiguously identify the oil signal in the T_1-T_2 map. However, for samples like the Estaillades samples previously shown, where the oil and water signal have similar T2, 3DNMR is the only way to extract the T_1T_2 ratio of the oil, therefore the wettability.

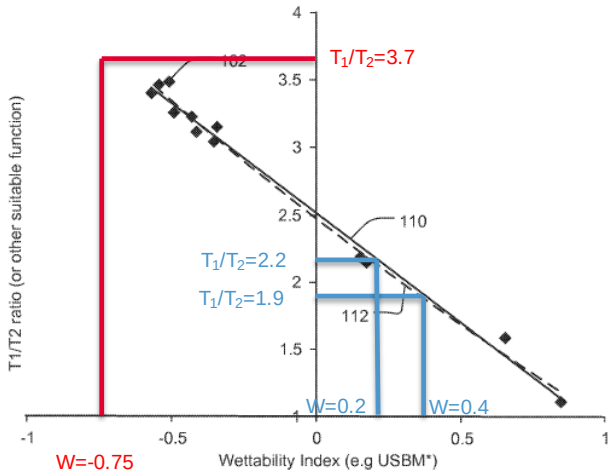

Figure 10: Correlation between the T_1/T_2 ratio of the oil with wettability (from [10]). The results from Estaillades carbonates before and after ageing are presented in blue, the results from the reservoir sandstone are presented in red.

CONCLUSION

We present in this paper the acquisition and processing of 3D NMR data: T_1, T_2 and Diffusion. We demonstrate that the interest of increasing the dimensionality of an NMR measurement is to unlock the fluids identification and to provide several petrophysical answers simultaneously. Examples are given on aged outcrop rocks and reservoir rocks that 3DNMR allows the joint determination of saturation and T_1/T_2 ratio of the oil, which is related to wettability [10].

Compared to the traditional measurement of wettability indexes, the proposed method is fast and does not require any change in saturation.

ACKNOWLEDGEMENTS

The authors thank Emmanuel Caroli and Jean Pierre Korb for fruitful discussions and Papa-Ousmane Mangane and Pierre Faurissoux for their support.

REFERENCES

[1] K. Brownstein and C. Tarr, "Importance of Classical Diffusion in NMR Studies of water in biological cells," *Physical Review A*, vol. 19, no. 6, 1979.

[2] W. Kenyon, P. Day, C. Straley and J. Willemsen, "A Three-Part Study of NMR Longitudinal Relaxation Properties of Water-Saturated Sandstones," *SPE-15643*.

[3] G. R. Coates, M. Miller, M. Gillen and C. Henderson, "The MRIL In Conoco 33-1

An Investigation Of A New Magnetic Resonance Imaging Log," in *SPWLA*, 1991.

[4] L. Venkataramanan, "Solving Fredholm Integrals of the First Kind With Tensor Product Structure in 2 and 2.5 Dimensions," *IEEE TRANSACTIONS ON SIGNAL PROCESSING*, vol. 50, no. 5, pp. 1017-1026, MAY 2002.

[5] C. Cao Minh, N. Heaton, R. Ramamoorthy, E. Decoster, J. White, E. Junk, R. Eyvazzadeh, O. Al-Yousef, R. Fiorini, B. Dacion and D. McLendon, "Planning and Interpreting NMR Fluid-Characterization Logs," in *SPE 84478*, 2003.

[6] M. Fleury and F. Deflandre, "Quantitative Evaluation of Porous Media Wettability using NMR Relaxometry," *Magnetic Resonance Imaging*, vol. 21, 2003.

[7] J. Chen, G. Hirasaki and M. Flaum, "NMR Wettability indices: Effect of OBM on Wettability and NMR responses," *Journal of Petroleum Science and Engineering*, vol. 52, 2006.

[8] W. Looyestijn, "Wettability Index Determination from NMR Logs," in *SPWLA*, 2007.

[9] J. Korb, G. Freiman, B. Nicot and P. Ligneul, "Dynamical Surface Affinity of Diphasic liquids as a probe of Wettability of Multimodal Pourous Media," *Physical Reveiw E*, 2009.

[10] A. Valori, B. Nicot, M. Hurlimann and H. Bachman, "Obtaining Wettability from T1 and T2 Measurements". Patent WO2013180752.

[11] C.H.Arns, «Multidimentional NMR inverse Laplace spectroscopy in petrophysics,» *Society of Petrophysicists and Well-Log Analysts*, p. 9, June 2006.

[12] G. Ibrahim and B. Nicot, "Solving Fredholm Integral for 3D NMR Inversion," *Submitted to Journal of Magnetic Resonance*, 2017.