Spatial resolved nuclear magnetic resonance in the study of rock heterogeneity and centrifuge capillary pressure

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This paper was prepared for presentation during the 13th International Congress of the Brazilian Geophysical Society held in Rio de Janeiro, Brazil, August 26-29, 2013.

Abstract

Taking rock and saturation heterogeneity into account is of great importance both in well logging and petrophysical core analysis programs. This work presents some results achieved with novel low field laboratorial nuclear magnetic resonance techniques that can spatially resolve transversal relaxation time (T_2) and signal amplitudes along core plugs length. The measurement quality is firstly tested with a mixture of bulk fluids (water and oil) and then performed in sandstone and carbonate rocks under fully and also partially (centrifuged) saturated conditions. These one dimensional rock imaging techniques can efficiently inform about sample pore-size distribution heterogeneities and also monitor in situ rock saturation with important centrifuge capillary pressure implications.

1. Introduction

1.1. NMR petrophysics

Nuclear magnetic resonance has been used widely in the last couple of decades as an important formation evaluation tool both in geophysical well logging and laboratorial petrophysics. While for laboratorial analyses core plug samples extracted from well walls and whole cores are placed within a solenoidal coil positioned between two magnet poles, Figure 1a, at the well site, it is the equipment that literally is placed inside the geological formation. In NMR logging, coils and magnets are specially designed to be able to respectively project the electromagnet and magnet field outside its cylindrical body, reason why the technique is also called inside-out NMR, Figure 1b.

At hundreds of gauss, a permanent magnetic field B(r) makes the ^{1}H isotope nucleus, or protons, present in reservoir fluids, precess around its direction (z) at a Larmor frequency $\omega(r)$ proportional to the applied field, $\omega(r)=\gamma B(r)$, where γ is the proton gyromagnetic ratio and r is spatial position. After spins fill the magnetic field presence, an energy equilibrium state is eventually achieved resulting in a spin maximum magnetization. Fluids polarized magnetization can be detected by applying an electromagnetic wave $B_1(t)$ perpendicularly to B(r), Figure 1, and with the same Lamor frequency (in resonance) which is in the radiofrequency (RF) range. $B_1(t)$ flips the magnetization to a direction where the precessing spins can produce an oscillating decaying signal induction at a coil called RF probe (laboratory) or antenna (logging).

a) b)

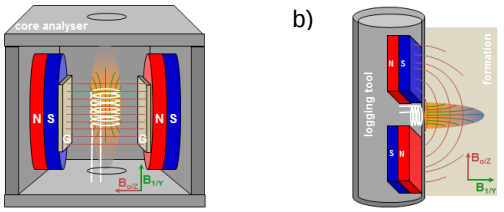

Figure 1 – General magnet/coil configurations of a a) benchtop NMR analyzer and b) a NMR logging tool.

By manipulating the spins with different polarizing periods and radiofrequency flipping pulses it is possible to extract primarily NMR information such as signal amplitudes, longitudinal (spin-lattice, T_1) and transversal (spin-spin, T_2) relaxation curves, and also self-diffusion coefficient, D_o. Raw NMR data is usually processed with a one or two dimensional inverse Laplace transform (ILT) algorithms from where relaxation and diffusion distribution curves can arise and be used in a variety of petrophysical models. With them many rock-fluid properties can be estimated or even quantified, such as pore volume, porosity, porous distribution, capillarity, permeability, fluid typing, viscosity, connectivity, wettability, among others.

NMR maximum signal amplitude S_0, for example, is one of the most reliable methods to quantified porosity since it is directly proportional to fluid quantity (^{1}H spin density). It is preferably acquired together with transversal relaxation time signal decaying $S(t_j)$, described by multiples exponential T_2 time constant, as follow:

$$S(t_j)=\sum_{i=1}^{n}A(T_{2,i})e^{\frac{t_j}{T_{2,i}}} \Leftrightarrow \text{Error}=\sqrt{\sum_{j=1}^{m}\left(M(t_j)-S(t_j)\right)^2}+\alpha\sqrt{\sum_{i=1}^{n}\left(A(T_{2,i})\right)^2},$$

where $A(T_{2,i})$ is the T_2 relaxation distribution with n bins. This distribution is calculated by ILT through minimization of the Error function where $M(t_j)$ is the raw experimental measured signal with m bins and α is a regularization parameter, which is dependent of signal-to-noise (S/N) ratio, typical of an ill-posed problem.

Transversal relaxation rates are faster and usually preferred than longitudinal ones. They are dependent of bulk, surface and diffusive relaxation mechanisms such that $1/T_2=1/T_{2b}+1/T_{2s}+1/T_{2d}$. Bulk relaxation rate is an inherent fluid physical-chemical property and is relatively small compared to surface one. Diffusion relaxation rate, $1/T_{2d}=(\gamma G_i TE)^2 D_0/12$, can be tremendously minimized at lower frequencies (which produce low internal magnetic gradient G_i) and high sampling rate 1/TE acquisition of the relaxation decaying signal. Considering the so-called fast diffusion regime, translational time rates of a wetting

phase can be related to surface-to-volume ratio so that $1/T_2 \approx 1/T_{2s} = \rho_2 S/V$, where ρ_2 is the matrix transversal surface relaxivity. Assuming constant relaxivity for a type of rock, T_2 spectra of a total saturated rock is a close approximation of its pore-size distribution. For spherical pores $1/T_2$ is proportional to $3/r_p$ and cylindrical ones to $2/r_p$, where r_p is pore radius.

1.2. Rock MRI considerations

Nuclear magnetic resonance imaging, commonly called MRI in clinical medicine, uses the frequency spatial dependence to generate one, two or three dimensional images. The original constant magnetic field variation is performed with powerful magnetic gradient coils individually dedicated for each perpendicular direction (x, y and z). Unfortunately, the generated imperfect gradients (G) are problematic in porous media due to the influence of diffusion relaxation and also the relative short relaxation life time in small pores. As frequency should preferably be low, the consequent low S/N ratio is a critical problem mainly because rocks have usual low fluid content. That is why rock MRI measurements are extremely long and noisy. All these issues are not a problem in human tissues which have comparatively long relaxation times and measurements are performed in much higher frequencies (tens of MHz).

Although the first NMR logging equipment was named magnetic resonance imaging logging tool (MRIL from NUMAR), no pore imaging (in conventional MRI terms) is currently done at the well site. Nevertheless, spatial resolved petrophysical diagnostic is indeed possible. Logging tool design dispenses gradient coils and amplifiers since an inherent magnetic gradient already exist. The magnetic field decays radially away, generating different resonance frequency $\omega(r)$ regions, from where different shell depths inside the formation (although just some inches) can be probed separately. This allows radially resolved signal amplitudes and T_2 distributions (T_1 and diffusion is also possible) acquisition which can be a valuable tool for heterogeneity studies and fluid typing in the mud-filtrate and native-fluid contact.

Although conventional MRI are still not ideal for rocks, many recently advances have been achieved, mainly with phase encoding techniques instead of usual frequency encoding ones. In this work novel one dimensional MRI-based measurement were performed in a regular low field NMR rock analyzer to extract spatially resolved NMR total signal and spatial resolved T_2 distributions along core plugs longitudinal length. Petrophysical meaning is discussed for totally and partially fluid saturated samples. Possible applications in core-logging integration and special core analyses (SCAL) programs are presented, especially those related to heterogeneity investigation, in situ monitoring and MRI-aided capillary pressure curves.

2. Methods

2.1. Pulse sequences

With modern pulsed NMR techniques total core plug fluid content can be reliably measured with an application of just a so-called 90^o radio frequency (RF) pulse after a sufficient wait time ($T_W > 5T_1$). This RF pulse brings fluid magnetization to the transversal plane (xy), parallel to the detecting coil orientation where an oscillating induction signal can be acquired. To get a complete transversal relaxation time decaying signal (T_2) a train of equally spaced 180^o pulses (double 90^o pulse time or amplitude) is applied after the first 90^o pulse. This train of pulses consecutively refocus the spins assembly allowing a proper full decaying transversal relaxation signal sampling, depending on number of refocusing pulses and the time between them (TE=2Tau). The refocusing event, also called spin echo, is acquired by the coil with a limit number of data points. This pulse sequence technique is called Carr-Purcell-Meiboom-Gill in reference to its idealizers and here it is simply called 'standard CPMG'. Figure 2a shows the corresponding pulse sequence diagram where RF events are separated from the coil acquisition signal events (just one red data point is shown per spin echo).

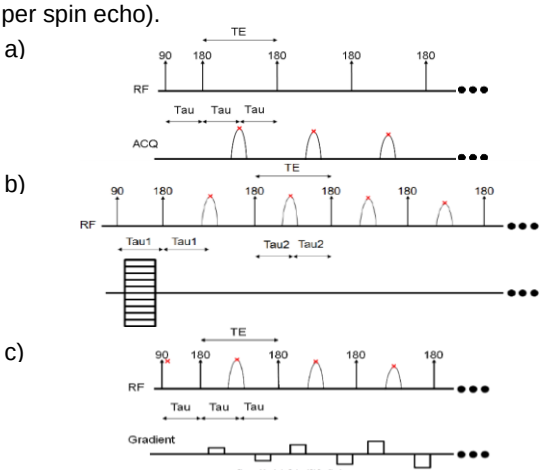

Figure 2 – Pulse sequence diagrams for a) standard CPMG; b) spatial CPMG and c) fast Profile.

Core plug spatial resolved translational relaxation curves were measured with one dimensional pure phase encoding Spin Echo Single Point Imaging (SE-SPI) method. It consists of a regular CPMG pulse sequence combined with magnetic field gradients and coherent phase cycling. In this 'spatial CPMG' sequence the time to the first spin echo (2Tau1) is much longer than usual TE, not enabling very high relaxation rates acquisition (T_2<5ms). This delay is inevitable because magnetic field gradient pulses require time to switch on and off due to the fixed amplifier power and coil inductance. Figure 2.c is another SE-SPI based method with consecutively increasing phase encoding and phase unwinding gradients that are applied around each echo. In a similar way, gradient pulses with theirs induced Eddy (or Foucault) currents also increase TE compared with standard CPMG. Although complete pulse diagrams explanation is beyond the scope of this paper, the latter can be assumed as a higher resolved fast Profile sequence compared to the spatial CPMG.

2.2. Samples, equipments and software

A cylindrical (3.8cm diameter per 2.8cm length) phantom sample was prepared with a non-emulsified mixture of water and oil, both having the same hydrogen index. The water phase (1.0g/cm^3 density, 14.8cm^3 volume and 1.3cm length) was doped with copper sulfate to low down its relaxation time (due to its paramagnetic ions) and to be

clearly differentiated from the higher relaxation times of the light paraffinic oil used (0.7g/cm^3 density, 17.1cm^3 volume and 1.4cm length). Free fluid relaxation is measured in a non-porous-confined condition so that only bulk relaxation time is measured.

Two 1.5in core plug samples were cleaned by solvents in a Soxhlet extractor and routine-petrophysics analyzed in a Core Lab (USA) automated gas (N_2) porosimeter-permeameter. The 4.8cm sandstone (S1) has 23,8% porosity and 2600mD permeability against 21% and 100mD of the 5,9cm carbonate (C1). Both samples were full brine (50kppm of NaCl) saturated and later desaturated in an air-brine drainage process at a Core Lab modified Beckman Coulter Ultracentrifuge (Optima L-XP Series).

NMR measurements were carry out on an Oxford Instruments (England) Q-sense Maran DRX spectrometer with 46mT (2,2MHZ) vertical bore at 32^{o}C temperature. Though not a MRI tomographer, this low field benchtop core analyser is equipped with one dimension (y) gradient coil that provide a maximum gradient strength of 60gauss/cm. The probe was a 51mm diameter with 12μs 90^o pulse duration and 20μs dead time.

NMR pulse sequence were already implemented in a Green Imaging Technology (Canada) software package compatible with the Oxford NMR hardware. Inverse Laplace and Fourier transform algorithms, together with additional data processing such as cumulative T_2 curves, geometric (or logarithmic) means, modeled capillary pressure curve, was also done with the same software support. In all plots, machine signal amplitudes are converted into fluid volume trough a calibration sample with well-known volume.

3. Results

3.1. Spatial resolved bulk relaxation

As an initial test the free fluid mixture of water and oil is measured. Figure 3a shows the whole sample transversal relaxation curve (red) and its multi-exponential fitted curve (blue). ILT incremental T_2 distribution (thicker red curve) in Figure 3b presents two well resolved peaks, each one corresponding with the bulk relaxation of individual fluids measured separately.

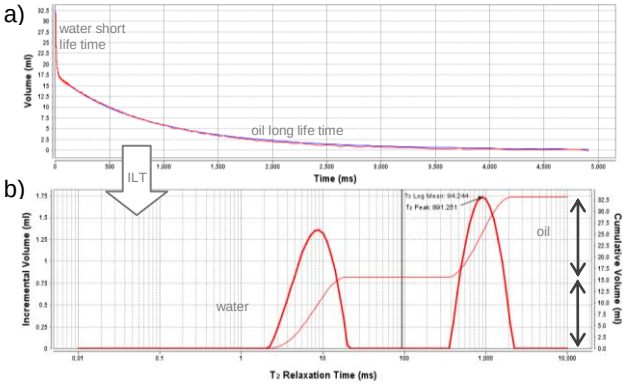

Figure 3 – Standard CPMG results of a mixture of doped water and mineral oil: a) raw transversal relaxation decay and its b) inverse Laplace transformed T_2 relaxation time distribution.

While time-domain maximum amplitude (equal to total T_2 distribution area) gives the whole sample fluid volume, the relaxation-domain cumulative T_2 curve (thinner red curve) informs about relative fluid volume portion corresponding to each individual phase with an error less than one cubic centimeter.

Figure 4a shows transversal relaxation distributions from three different slices of the sample. While the middle T_2 distribution is reading the interface slice where water and oil coexist, the T_2 distributions in the extremities are reading whether water or oil. The water phase stayed on the bottom due to its higher specific gravity. Figure 4b shows a corresponding counter map plot, or T_2 spatial map, where the red color indicates higher fluid volume density. Fast profile raw data was Fourier transformed to generate the one dimensional longitudinal fluid content profile, confirming each fluid phase length position in the T_2 map.

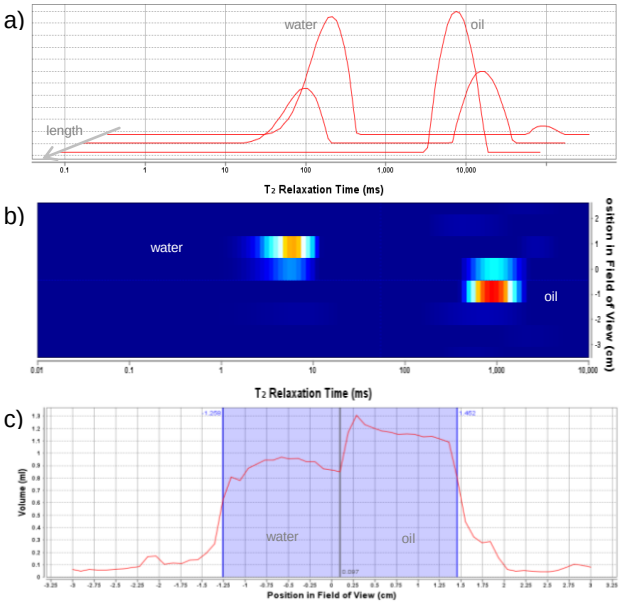

Figure 4 – Spatial NMR results of a mixture of doped water and mineral oil: a) three-slice T_2 distribution and its correspondent b) spatial T_2 map; c) longitudinal profile.

3.2. Totally saturated rocks

3.2.1. Rock heterogeneity probe

Figure 5a shows the whole sample conventional T_2 distribution for the sandstone sample. As S1 was extracted from a very well-sorted formation, the whole sample T_2 distribution presents a big and selected pore family (794ms of T_2 main peak) with a cumulative total pore volume of 13.8cm^3. This pseudo-pore-size distribution has a T_2 geometric mean value of 463ms and a NMR porosity of 23.0%. Figure 5b show a spatial resolved T_2 distribution with an 8 pixel resolution in a 6 cm field of view. The curves on the x and y boxes indicate a slice T_2 distribution (in the middle of the core plug) and a specific pore-family profile (at 800ms T_2) extracted from the region cross-marked with a dotted white line. All T_2 slices are similar to each other and with the previous shown whole sample conventional T_2. Each slice cumulative T_2 pore volume plotted against slice position

(graphic not shown) generates a longitudinal profile quite similar to the main T_2 peak profile show at the y-slice. Figure 5c shows a 7.0cm field of view high resolution, 64 pixels, profile. This longitudinal profile has a flat pore volume plateau indicating a very homogeneous porosity rock structure along the core. This agrees very well with the T_2 mapping continuity, since pseudo-pore size distribution does not change from a slice to the other. Most probably the permeability in each slice, dependent of T_{2gm} and porosity, especially in sandstones, is also constant. The results show that S1 is a very homogenous and probably with a non-varying (or only a few) perm porosity system along its length.

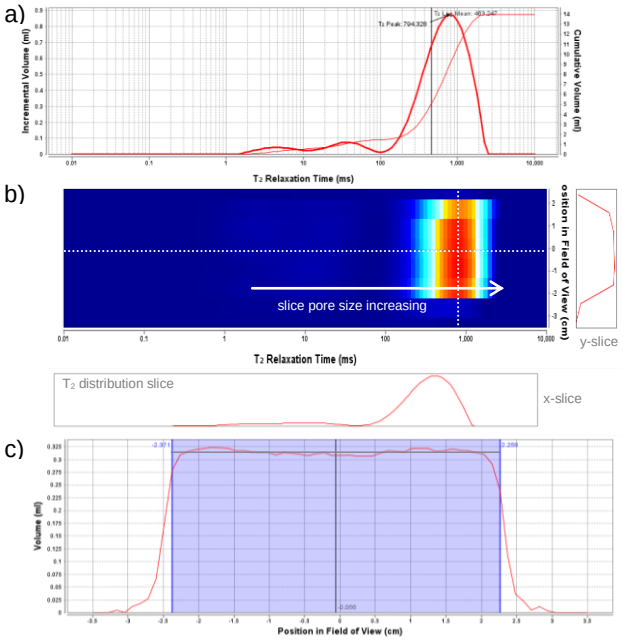

Figure 5 – Sandstone core plug sample: a) whole T_2 distribution b) T_2 mapping and c) longitudinal profile.

The carbonate core plug presented a whole sample broad pseudo-pore-size distribution, Figure 6a, typical of complex porous structure normally found in such lithology. T_2 distribution presented a 40ms main peak and 92ms T_{2gm} with cumulative total pore volume of 14,3cm^3. The result from longitudinal profile, Figure 6b shows a slight reduction in its volume amplitude from bottom to top core plug face. As the core gravity saturation index is 100% and the weight pore volume corresponds well with the total cumulative T_2 pore volume, it possible to conclude that there is a small but consistent reduction in pore volume from one core face to the other.

The slice stacked T_2 distribution in Figure 6a together with T_2 mapping, Figure 6b, show clear variation in T_2 distributions along carbonate core length. The slightly T_2 shift to shorter values from bottom to top face of the core suggest a pore size distribution variation with consequent differences in poro-perm properties compared. The top face presented a lower NMR porosity with shorter T_{2gm} what could result in shorter permeability.

3.2.2. More applications

In core-logging integration programs, it is common to compare whole sample T_2 distribution with pore-throat size distribution from mercury injection capillary pressure (MICP) main for extracting the transversal surface relaxivity parameter to convert T2 in pore-size. In the meanwhile, mercury porosimetry and capillary analysis is done in a small end piece of the core plug and the comparison can be non-representative in cases of strong heterogeneities. With spatial CPMG technique a specific portion of a core can measure and later send it for MICP analysis allowing a posterior much more reliable comparison. Furthermore, a proper multi-slice comparison approach can also probe possible surface relaxivity variation within a core sample.

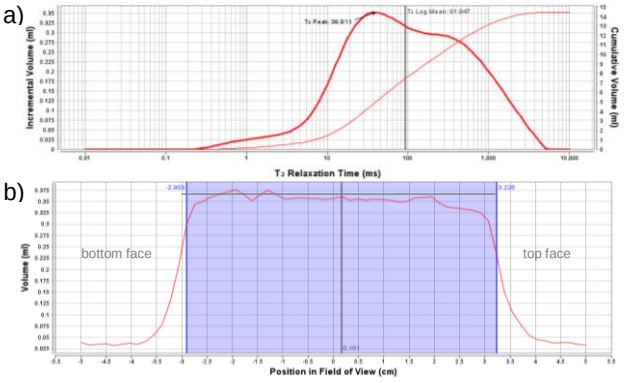

Figure 6 – Whole carbonate core plug measurement: a) T_2 incremental and cumulative distribution and its b) longitudinal profile

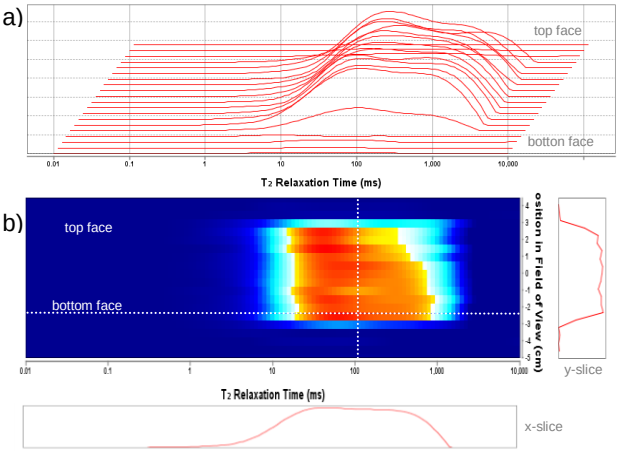

Figure 7 – Carbonate core plug sample: a) stacked T_2 slice distributions and corresponding b) spatial T_2 mapping with a slice T_2 (x-slice) and a profile of a pseudo-pore size (y-slice).

Another important application of the technique is the ability of monitoring perm porosity changes during one phase injection experiments such as in carbonated water (CO_2-enriched) injection. Due to the lack of fast and efficient in situ monitoring technique Spatial CPMG can help saving a lot of time once there is no need to clean the sample at each injection step and re measure its routine petrophysics. Furthermore it does not add uncertainties due to the possible structural changes during the cleaning process.

Homogeneity is a required assumption of many theoretical pretrophysical models from where many special core analyses are based. X-ray and gamma-ray

techniques for example have historically been used for monitoring S_w distribution within the core plug mainly for relative permeability experimentation. These new MRI based technique not only can efficiently do the same thing but can also measure spatial T_2 distributions.

3.3. Partially saturated rocks

3.3.1. Saturation probe

To pursue a quantitative desaturation process, core samples were spun at a specific speed, Figure 8, up to production stabilization, when centrifuge and capillary forces reach equilibrium. Figure 9 shows a spatial T_2 map from S1 sample before (totally saturated) and after (partially saturated) 3 hours spinning at 750 rpm. The gradual decreasing in pore volume amplitude from outer to inner centrifuge radius is because air-brine capillary forces increases expelling out free brine mainly in big pore families. Slice profiles from T_2 main peak shows a strong saturation gradient for this correspond big pore family.

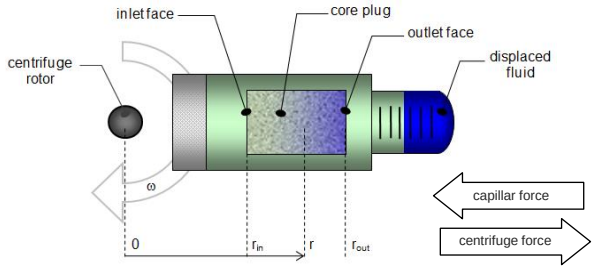

Figure 8 – Core plug inside a centrifuge drainage core cup and radial centrifuge forces.

Figure 10a shows the stacked T_2 distribution slices of the spun (750rpm) core. Although whole sample irreducible water saturation (S_{wi}) is not yet reached at such speed, T_2 slices from outlet to inlet core face start converging to T_2 at S_{wi} because of higher capillary pressure. From each slice T_2 cumulative pore volume, water saturation (S_w) was calculated and plotted with correspondent T_2 geometric mean (T_{2gm}). Fitting data into an empirical linear model[11], $S_w=aT_{2gm}+S_{wi}$, irreducible water saturation (S_{wi}) is estimated in 6%. With such a value, the whole sample cumulative distribution delivered an air-brine based free fluid T_2 cut-off of 80ms, already expected for this clay-cleaned sand. With this approach, T_2 cut-off calculated without having to centrifuge the samples up to much higher speeds and this is especially important in friable and weak samples that cannot stand high centrifuge pressures.

Spatial T_2 mapping in homogeneous and standard-size samples spun at high speeds results in homogeneous low T_2 values maps (being all slices fairly the same), with just irreducible water in very small pore families, where capillary pressure is extremely high. Centrifuge saturation gradients can also be faded away by natural gravity forces and spatial T_2 mapping can also be used to monitor this process that usually takes longer for low perm samples (data not shown). Possible trapped water in one of the core sides can also be probed. This is important once it is common to use spun samples for many other SCALs.

3.3.2. Centrifuge capillary pressure

In conventional centrifuge capillary pressure curve (CCPC) the amount of liquid expelled from a core plug after reaching hydrostatic equilibrium is directly measured. From the expelled water the average water saturation at each centrifuge speed is known, and using an approximate solution based on theoretical model(s)[9], the saturation at the inlet face can be obtained. This saturation is plotted against the capillary pressure P_C at the inlet, Figure 8, which is calculated using the centrifuge rotational speed ω, fluid density difference $\Delta\rho$, and the distance r to the measured saturation points using the Hassler-Brunner equation, $P_c(\omega,r)=\Delta\rho\omega^2(r_{out}^2-r^2)/2$. This procedure is repeated 7 to 10 times to fully define the capillary pressure curve. This is very time-consuming, because each equilibrium step can take up to two days or more. But it still saves a lot of time comparing with porous plate method, the more direct and reliable one, where each equilibrium step can take weeks.

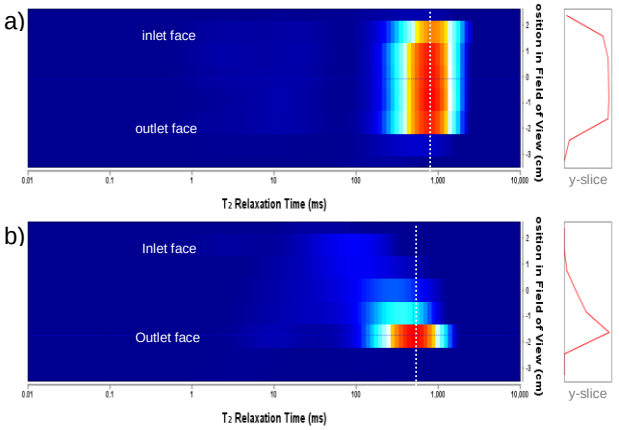

Figure 9 – Sandstone T_2 spatial mapping a) fully and b) partially (750rpm) brine saturated.

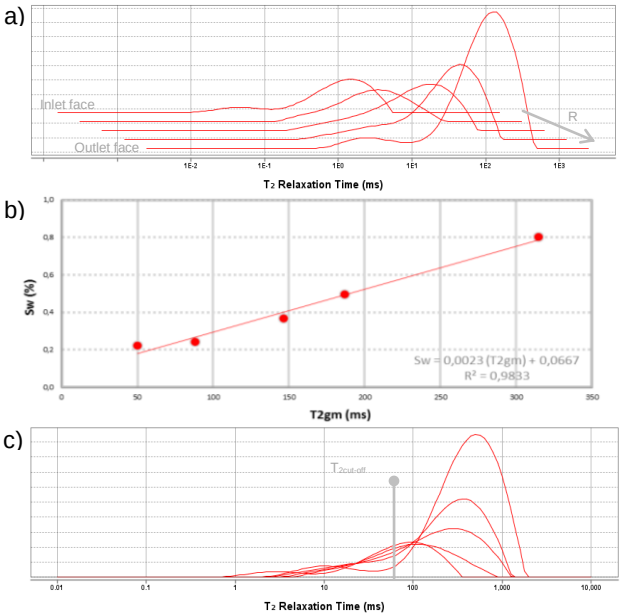

Figure 10 – a) Stacked T_2 distribution and b) each slice brine saturation and geometric mean cross-plot. c) All T_2 slices projected together and the T_2 cut-off just as a reference.

Instead of an approximate solution for the inlet saturation, MRI-based techniques such spatial CPMG and fast profile, can aid conventional centrifuge capillary pressure. To get a so-called magnetic resonance imaging capillary pressure (MRICP), a partial saturated sample longitudinal profile (could be also from spatial CPMG) was acquired twice: one after centrifuging for 3-hours at 750 rpm and the other after centrifuging more 4-hours at 1500 rpm, Figure 11a. The centrifuged core volume profiles are divided by the 100% saturated volume profile. Associating each calculated water saturation point $S_w(\omega,r)$ with theirs correspondent capillary pressure $P_c(\omega,r)$, a full capillary pressure curve is formed, Figure 11b. Brooks-Corey Pc model was used to parameterize the data with a GIT orthogonal least square fit error function. An 8% irreducible water saturation was computed from the asymptotic $P_c(\omega,r)$ values.

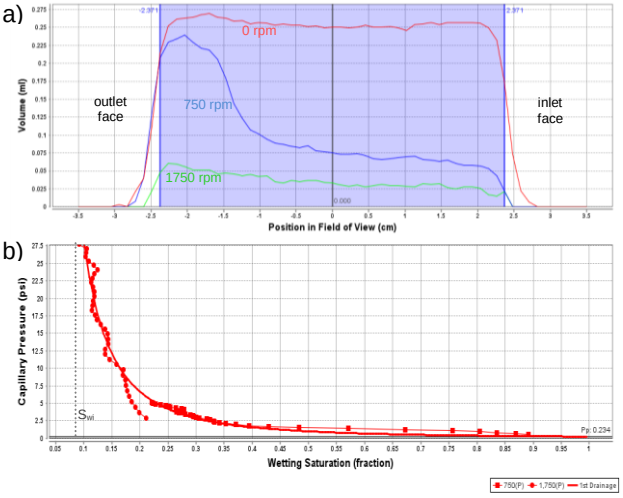

Figure 11 – a) Profiles of a sandstone sample in different saturation condition. b) Corresponding MRICP.

MRICP curves can be very important in rock multiphase fluid transport, being fundamental inputs for reservoir simulators and enhanced oil recovery programs. They can also be used in wettability index determination applying USBM method (additional imbibition curve is mandatory), geological face quality and rock typing. As any other capillary pressure curve method, pore-throat-size distribution and relative permeability can also be modeled from. Water-oil capillary pressure curves can be also measured if deuterium oxide (D_2O), non-NMR sensitive, is used instead of regular water.

4. Conclusions

Novel one dimensional MRI techniques based on pure phase encoding Spin Echo Single Point Imaging methods are better for porous media than traditional frequency encoding ones, though still not ideal for tight rocks with very high relaxation rates. The presented one dimensional pulse sequences were successfully tested in bulk fluids and in sandstone and carbonate core plugs.

In fully saturated rock samples, spatially resolved NMR is a probe for matrix heterogeneity since pore volume profiles and pseudo-pore size (T_2) distribution can be measured from different slices along core plug length. This is remarkable for supporting special core analysis programs in many day-to-day activities and also in core-logging integration for evaluating representativeness.

In partially saturated cores, spatially resolved NMR is an in situ fluid distribution monitoring tool with odd applications in controlled centrifuge core experiments. Irreducible water saturation, free fluid T_2 cut-off and capillary pressure are prime important implications. Other indirect applications of MRICP can be used to extract or model many petrophysical properties. In future work, more rock samples will be added, two saturating liquids will be used (heavy water and oil) and referred application will be deeply explored.

Acknowledgments

PETROBRAS and CNPQ for financial support. Green Imaging Technology and MRI Center of University of New Brunswick. Laboratory for application of NMR and Petrophysics at Fluminense Federal University. Cenpes Special and NMR Petrophysics Laboratories.

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