Applications of a New Technique to Acquire Spatially Resolved NMR Petrophysical Data

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ABSTRACT

NMR pore size distributions derived from measurement of the NMR T_2 relaxation are extensively utilized both in the laboratory and in well-logging tools. Typically these measurements are made to quantify the type of fluid in the pore network (clay bound, capillary bound or free fluid). In addition to pore size, the NMR relaxation time T_2 is also sensitive to fluid type (oil versus water) and the wettability of the rock. There are a number of applications that can benefit from resolving this information spatially. This work presents a new method for acquiring T_2 distributions spatially in a rock sample. Three applications will be investigated to explore the capabilities and usefulness of the new technique. The three applications are: 1) a simple quick method for determining the irreducible water saturation, 2) monitoring fluid redistribution following centrifugation, and 3) investigation of sample heterogeneity.

The irreducible water saturation can be predicted by using the new NMR technique to measure the T_2 relaxation as a function of position down a core plug after the plug has been centrifuged to create a range of saturations in the plug. The relationship between the geometric mean of the T_2 and saturation leads to a prediction of the irreducible saturation. This technique has the advantage of not actually requiring the plug to reach irreducible saturation to determine its value as this may not be possible on some rocks.

Fluid movement following centrifugation of a sample is complex and can depend on many factors including the permeability, gravity, and capillary pressure. A measurement of the T_2 spatially yields information about which pores are occupied and how the movement or redistribution of fluid takes place. In the final application, the new technique simply looks for variations spatially in the pore size distribution (and relaxivity) that may not appear on a simple saturation profile on various rock types.

INTRODUCTION

NMR techniques have proven to be an invaluable tool in the oil and gas industry. Quantitative NMR measurements are sometimes the only way to determine certain petro-physical properties [2]. The oldest and most established techniques are NMR relaxometry techniques. The principal NMR relaxation times are T_1 and T_2. The T_1 process is the recovery of longitudinal magnetization while the T_2 process is the decay of transverse

magnetization. Although the T_1 and T_2 processes occur simultaneously, for purposes of this publication we will only discuss a T_2 measurement in more detail. The T_2 relaxation time is defined by the rate of exponential decay of the transverse magnetization. This is expressed as follows:

$$M_{XY} = M_{XY0} e^{-t/T_2} \tag{1}$$

In the above, M_{XY} is transverse magnetization, M_{XY0} is the initial transverse magnetization, t is time, and T_2 is the relaxation time. The most widely accepted T_2 measurement method is the famous Carr Purcell Meiboom Gill pulse sequence (CPMG). The pulse sequence consists of a 90 degree radio frequency (RF) pulse to place the magnetization in the transverse plane then a series of 180 degree refocusing pulses to continuously refocus echoes which decay with the time constant T_2. The pulse sequence diagram is shown below:

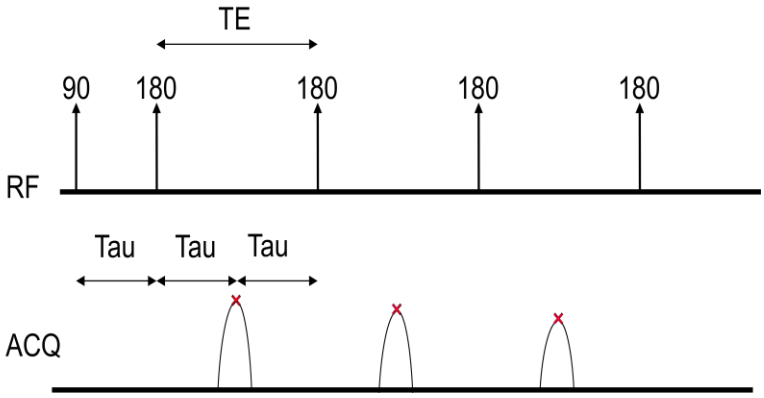

Figure 1. Standard CPMG Pulse Sequence

The CPMG sequence begins with a 90 degree RF pulse which tips the magnetization to the transverse plane. Immediately after the 90 degree pulse, the transverse magnetization is at its maximum and starts to decay rapidly due to the different magnetic fields each hydrogen experiences. After a time, tau, a sequence of 180 degree pulses spaced by two times tau (called the echo time) is applied to refocus the dephasing caused by an inhomogeneous magnetic field leaving only dephasing due to spin-spin interactions (i.e. T_2). This process is repeated and the decay of the echo amplitudes yields T_2. The decay of each echo is governed by a different exponential NMR time constant T_2^*.

CPMG T_2 measurements are used extensively in NMR logging as well as in laboratory core analysis to quantify the total porosity and to determine the pore size distribution of the sample. The T_2 relaxation time is related to the pore size distribution by the following equation [4]:

$$\frac{1}{T_2} = \frac{1}{T_{2-Bulk}} + \rho \frac{S}{V} + (\gamma G T_E)^2 \frac{D_O}{12} \tag{2}$$

Where $T_{2\text{-Bulk}}$ is the bulk fluid relaxation time, ρ is the relaxivity parameter, S is the surface area of the pore, V is the volume of the pore, G is the gradient of the magnetic field, TE is echo time, and D_0 is the diffusion coefficient. In rocks, the first and third (diffusion) term in Equation (2) can be ignored due to $T_{2\text{-Bulk}}$ being long and TE being very short. Equation (2) thus reduces to:

$$\frac{1}{T_2} = \rho \frac{S}{V} \tag{3}$$

According to Equation (3), the T_2 relaxation time is directly related to the pore size (S/V) via the relaxivity parameter, ρ. Using standard pore size cut-offs, we can divide the pore size distribution into clay bound water (CBW), capillary bound water (BVI) and free fluid (FFI)[2].

Magnetic resonance imaging (MRI) resolves the NMR signal by spatially altering the magnetic field with magnetic field gradients. A wide variety of pulse sequences (combinations of gradient, excitation, refocusing, and acquisition events) have been created to highlight different NMR properties and/or localize the location of hydrogen.

Spatially Resolved T_2 NMR Technique

Performing a bulk CPMG measurement on a rock sample gives the pore size distribution of the sample as a whole. In the case of heterogeneous samples, one might be interested in T_2 distributions from specific parts of the sample. Such requirement can be fulfilled by using a spatially resolved T_2 measurement. Spatially resolved T_2 is also a good tool to examine intentional variations (experimentally created) in T_2. The methodology for obtaining this measurement is to utilize spin echo (SE) single point imaging (SPI) methods.

The spin echo single point imaging (SE-SPI) technique was first proposed by Li et al. [1] to measure the T_2 distribution at different positions in a sample. Petrov et al [3] simplified the original technique combining a CPMG pulse train in combination with magnetic field gradients and coherent phase cycling. The pulse sequence diagram is shown below.

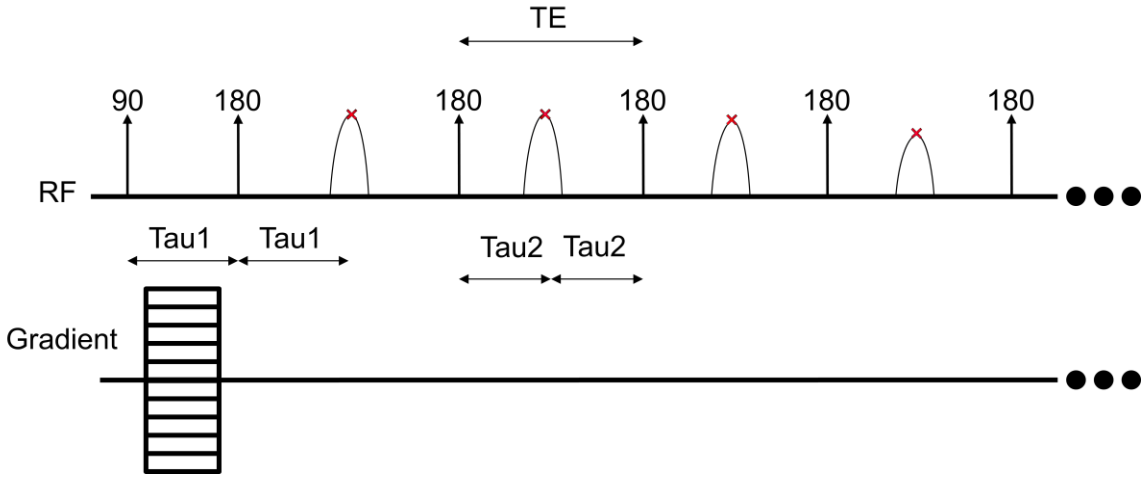

Figure 2. Spin Echo Single Point Imaging Pulse Sequence

This sequence uses two separate pulse interval times, tau1 and tau2. The second tau, tau2, is chosen to be as short as possible to measure the short T_2 signal in the rock and to ensure the third term in Equation (2) can be ignored. The longer first interval time, tau1, is required to have a magnetic field gradient switch on then off. A magnetic field gradient requires time to switch on and off due to the fixed power of the driving amplifier and the fixed inductance of the coil generating the magnetic field. There are also other factors such as induced eddy currents which further increase the delay required. A typical time for Tau1 is 0.5msec.

One key to the SE-SPI technique is to perform phase cycling to maintain the phase coherence of the signal acquired during the CPMG echo train. This type of phase cycling, known as XY phase cycling was first introduced by T. Gullion [5]. In an XY-4 phase cycling scheme the phase of the RF refocusing pulses (180 degree pulses) is cycled through $(XYXY)^n$. The signal intensity at each echo, m, for this SE-SPI sequence is given by:

$$M_{XY} = M_{XY0} e^{-2Tau1/T_{2p}} e^{-(m-1)2Tau2/T_2} \tag{4}$$

Where T_{2p} is the relaxation contrast for the first echo (usually the first exponential term can be ignored). To increase the signal to noise, multiple points can be acquired for each echo and averaged together. For this work only one point per echo was acquired.

Relationship between Saturation and T_2 Log Mean

Coates et al [2] first proposed a relationship between the irreducible water saturation and the geometric mean of the T_2 NMR given by:

$$\frac{1}{S_{WIR}} = mT_{2LM@Swir} + b \tag{5}$$

where the log mean T_2 is defined by:

$$LogMean = 10^{\frac{\sum \phi_j \log(T_j)}{\sum \phi_j}} \quad (6)$$

Coates also noted that the intercept, b, is usually 1. This describes a linear relationship of T_2 log mean of the rock at irreducible saturation versus irreducible saturation across many rocks. Li et al [1] expanded this idea to predict the irreducible water saturation by measuring the T_2 relaxation at different water saturation levels in a core plug. As the larger pores are drained, the local T_2 relaxation times will shorten. Extrapolation of this trend leads to a prediction of irreducible water saturation. The linear relationship between saturation and T_2 log mean as described by:

$$S_w = mS_{WIR}T_{2LM@Sw} + S_{WIR} \quad (7)$$

This relationship implies that the y intercept of the plot will yield the irreducible water saturation. By using a centrifuge, one can create a core plug sample with a distribution of many different saturation values. Then we can measure both the saturation and the T_2 using the spatially resolved T_2 technique described above. A plot of Sw versus T_{2LM} should be linear and have a y intercept equal to the residual water saturation.

After performing the experiments on 4 different rocks and using various centrifuge speeds to create the saturation differences in the rock, we have found that the linear relationship only holds for larger saturation values.

RESULTS

All magnetic resonance experiments were performed with an Oxford Instruments GeoSpec2 spectrometer operating at a resonance frequency of 2.4MHz. The radio frequency coil was 40mm in diameter equipped with Q-SENSE technology for shorter measurement times and improved signal to noise ratio. Q-SENSE uses techniques so that the probe Q (probe bandwidth over resonance frequency) is low to give fast ring down times (therefore the minimum tau that can be obtained) without compromising the signal to noise ratio. The centrifuge used was a Beckman Coulter J2-21 high speed centrifuge with a JS7.5 swing bucket rotor and a rock core sample handling kit from Green Imaging Technologies. The radius to the outlet face of the rock was 15.1cm.

All SE-SPI scans were acquired with a first tau, tau1, of 500usec and a second tau, tau2, of 100usec. The number of echoes was adjusted to acquired signal until echo had fully decayed. The spatial resolution was 64 points over a field of view of 7cm.

All samples were nominally 1.5 inches in diameter and 2 inches long. See Table 1 for a description of each sample.

Table 1. Summary of rock samples used

Sample	Diameter (cm)	Length (cm)	Porosity (p.u.)	Permeability (mD)	Lithology
1	3.8	5.0	15.8	Unknown	Low Perm Sandstone
4	3.8	5.0	17.2	Unknown	Low Perm Sandstone
52A	3.8	5.2	10.6	~1	Sandstone
116A	3.8	5.1	16.4	~3	Sandstone
109S	3.8	5.0	15.5	102	Sandstone
20A	3.8	5.0	13.4	4	Sandstone

Sample 20A was intentionally improperly saturated with brine after being cleaned. For this sample only the outside ends of the sample were fully saturated leaving the middle of the rock at a much lower saturation. The result is consistence with the NMR porosity being much lower than the Helium porosity. Figure 3 show the spatially resolved T_2 of this rock.

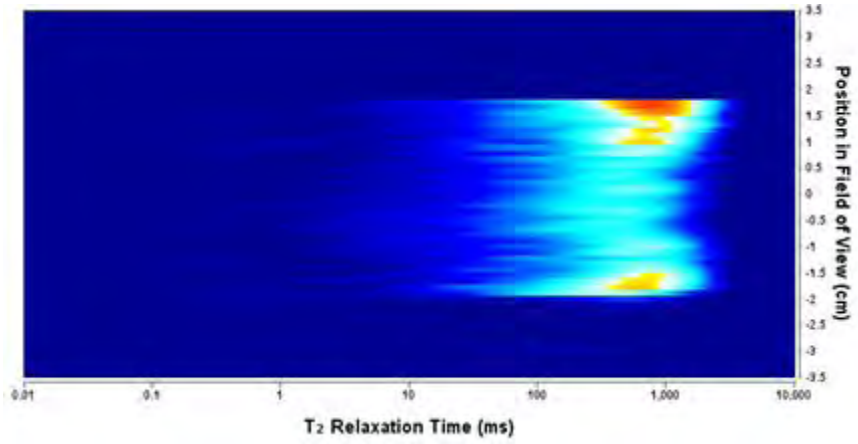

Figure 3. Spatially resolved T_2 of a incorrectly brine saturated core plug (Sample 20A)

The plot is a two dimensional intensity plot with the X axis being the T_2 relaxation time and the y axis being the long axis position of the sample. The intensity is the amount of water that has the corresponding T_2 and position. Although the rock has a uniform pore size distribution throughout the rock, one can easily see the larger pores are not fully occupied with water in the center of the sample (showing lower signal intensity).

Figure 4 compares the SE-SPI technique to a standard SPRITE acquired profile. The SE-SPI profile is created by summing all T_2 signal at each position.

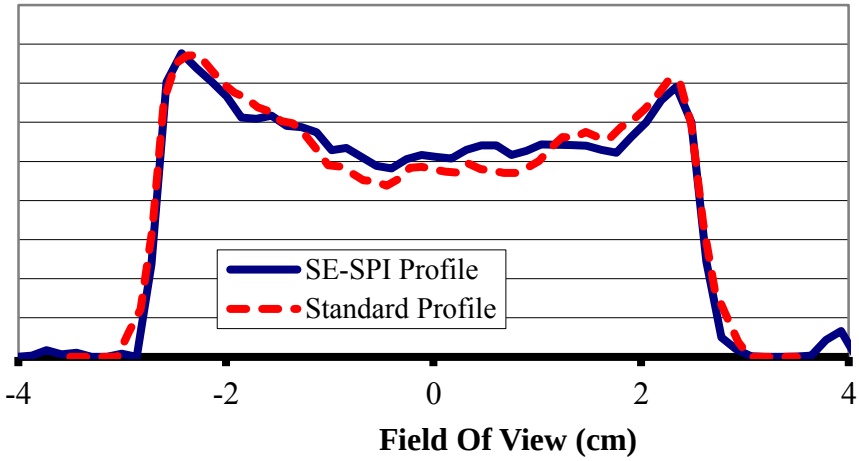

Figure 4. Comparison of a profile formed from the SE-SPI data and a regular SPRITE acquired profile for sample 20A

Figure 5 compares the bulk T2 from SE-SPI versus a conventional CPMG sequence. The SE-SPI bulk T2 is constructed by summing all spatial data for each T2 value.

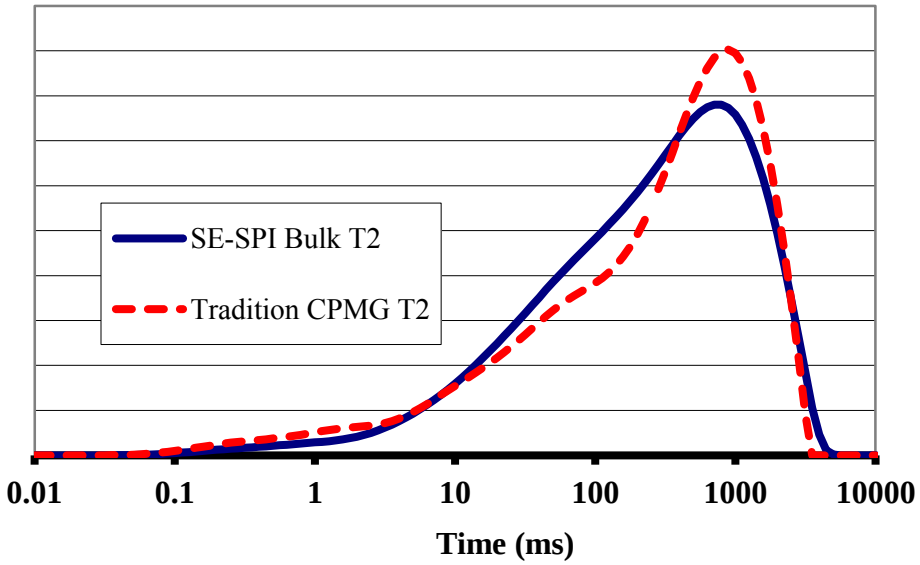

Figure 5. Comparison of bulk T_2 data acquired from SE-SPI data and a traditional CPMG T_2 measurement

Four of the samples (1, 4, 52A, and 116A) were used to investigate the relationship between saturation and T_2 log mean. For this investigation the following procedure was followed:

1. The rocks were cleaned, dried and fully saturated with 2% NaCL brine.
2. A bulk T_2, 1D saturation profiles, and spatially resolved T_2 measurements were performed on the samples.

3. The samples were spun in a centrifuge at 2500 rpm, 5000 rpm, and 7500 rpm for 24 hours. After each speed the measurements of Step 2 were repeated. Note that samples were not flipped during the centrifugation step.

Figure 6 shows the spatially resolved T_2 measurements for sample 52A. The other samples show similar results.

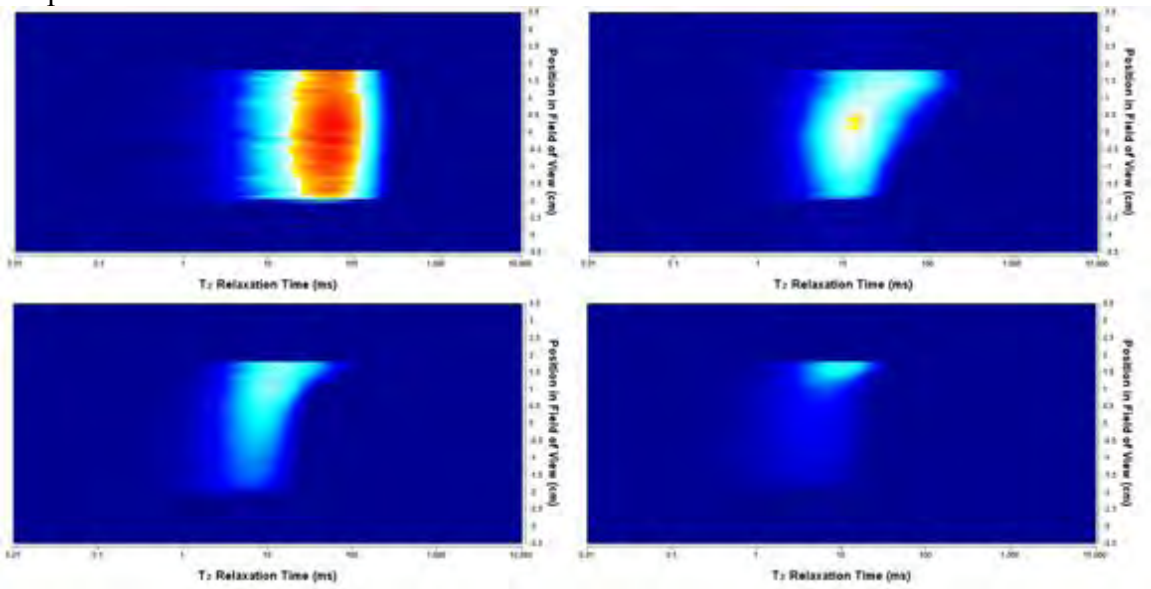

Figure 6. Spatially resolved T_2 measurements at 100% brine saturated, and after increasing centrifuge speeds (moving left to right and top to bottom).

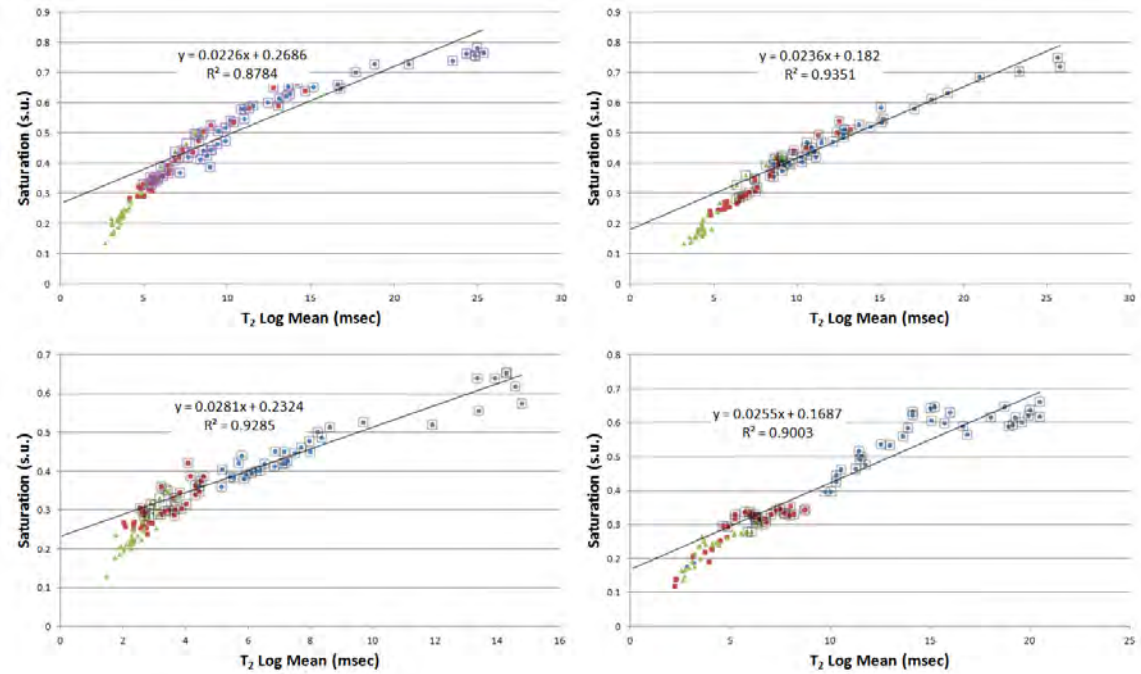

Figure 7. Plot of saturation versus T_2 log mean following centrifuging at different speeds. Samples are 1, 4, 116A, and 52A from top left going clockwise to bottom left.

Each centrifuge speed (2500, 5000, and 7500 rpm) is plotted in a different color. A linear fit is performed using only the data points highlight by square box around the point. The irreducible water saturation is the y intercept of the fit.

It was noted that all centrifuge data from each sample could be fitted by plotting saturation versus the logarithm of the T_{2LM}. Below is a plot of sample 1 with the X Axis plotted on a log scale.

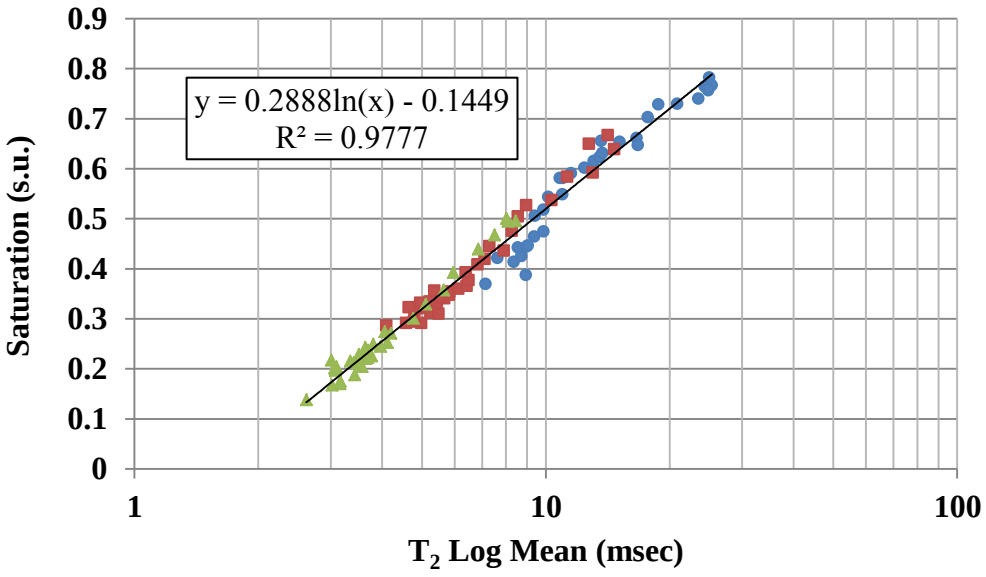

Figure 8. Plot of saturation versus the logarithm of the T_2 for sample 1.

A summary of the actual and predicted irreducible water saturation is shown in Table 2.

Table 2 - Summary of irreducible water saturation predication using T_2 log mean.

Sample	**T_{2LM}@100%Sw**	**Swir (s.u.)**	**Swir Predicted (s.u.)**
1	28.2msec	31.1	26.9
4	39.1msec	19.8	18.2
52A	36.4msec	19.3	23.2
116A	35.0msec	13.8	16.9

The irreducible water saturations were determined by using the saturation near the inlet face of the rock after centrifuging at 7500 rpm as measured by NMR.

In the final experiment the redistribution of fluid following centrifuging was investigated using sample 109S. For this experiment the following procedure was followed.

1. The sample were cleaned, dried and fully saturated with 2% NaCL brine.
2. A bulk T_2, saturation profile and the spatially resolved T_2 were measured.
3. The sample were spun in a centrifuge (at 1000rpm) to create a range of saturations within the rocks.
4. A spatially resolved T_2 was repeatedly measured until the saturation fully redistributed in the sample making the saturation approximately uniform.

Figure 9 shows the spatially resolved T_2 measurements taken for sample 109S. Each scan was acquired in 35 minutes.

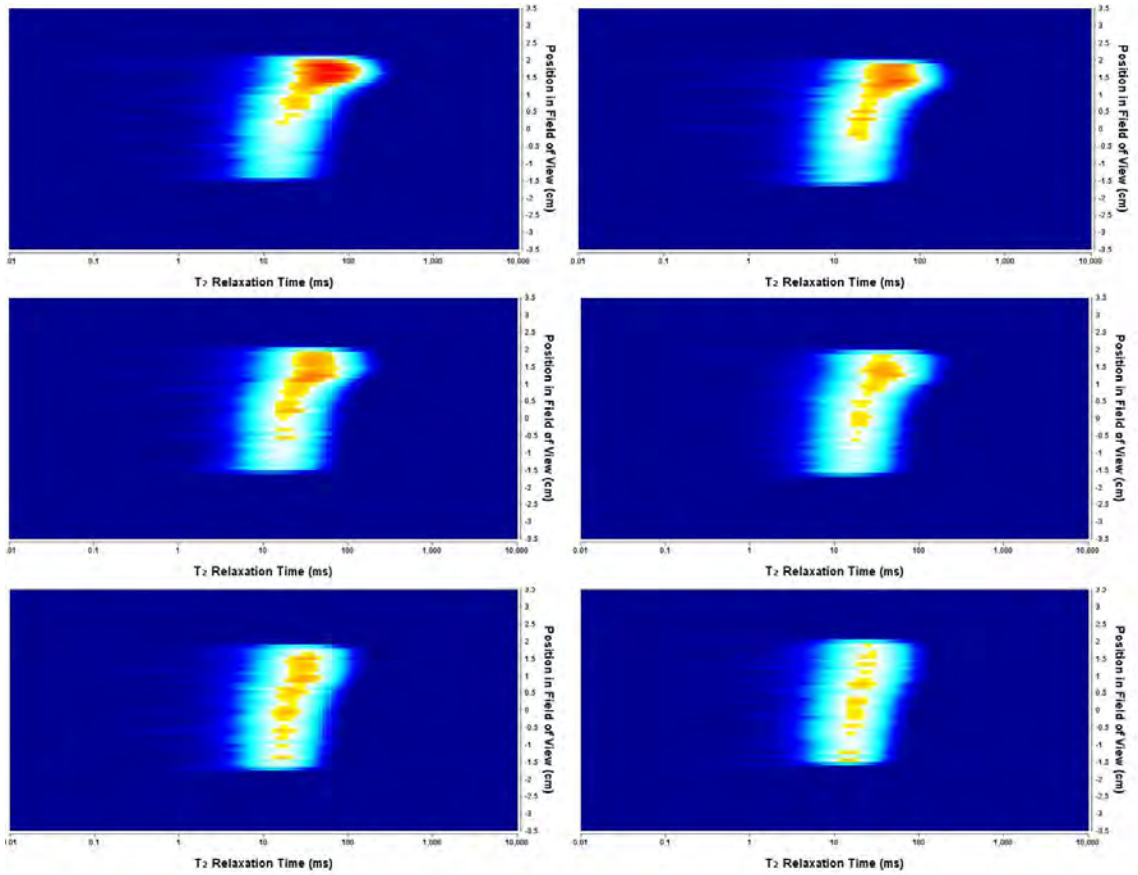

Figure 9. Time series of spatially resolved T_2 measurements (moving left to right and top to bottom).

The top left plot in Figure 9 shows is the sample after centrifuging for 24 hours at 1000 rpm. The remainder of the plots are acquired at 1, 2½, 6, 47h, and 145 hours after centrifuging. The fluid can be seen moving from the top towards the bottom of the plots (redistributing). The orientation during image acquisition (i.e. in the magnet) and during wait times was such that the higher saturation was at the top so that gravity was assisting redistribution.

CONCLUSION AND DISCUSSION

The spatially resolved T_2 technique described is a good tool for investigating spatially dependent saturation changes. A simple use is to investigate heterogeneous samples or samples that may have been improperly prepared. A far more interesting application involves using the technique to investigate experimentally created saturation variations. One application shows promise to predict irreducible water saturation in a rock by using a single low speed centrifuge step. This technique not only has the advantage of being fast but it may be possible to determine irreducible water saturation even if the forces required cannot be achieved due to centrifuge limitations or the friability of the sample itself.

Further work should be performed to fully understand the relationship between saturation and T_2 log mean. It seems there is a relationship between saturation and the log of the T_{2LM}. One possible explanation is that it becomes exponentially more difficult to remove water from smaller and smaller pores. This idea is supported by the exponential like shape of most capillary pressure curves.

The techniques described here are for air-brine systems. To use oil-brine systems modification will be required to distinguish the oil and water NMR signals. One simple modification is to use deuterium oxide for the brine which is not detected by the NMR system (only the oil will be detected).

The redistribution of fluid within the rock shows, not surprisingly, that the fluid moves from the higher saturation to the lower saturation. The time for redistribution is affected by the permeability of the rock and gravity. The fluid also seems to first move to the partially filled smaller pores from the larger pores.

REFERENCES

1. Li, L., Han, H, Balcom, B.J., "Spin echo SPI methods for quantitative analysis of fluids in porous media", *Journal of Magnetic Resonance*, (2009) **198**, 252.
2. Coates,G.R., Xiao, L., and Prammer, M.G., *NMR Logging. Principles & Applications*, Halliburton Energy Services, Houston, 1999.
3. Petrov, O.V., Ersland, G., Balcom, B.J., "T_2 distribution mapping profiles with phase-encode MRI", *Journal of Magnetic Resonance*, (2011) **209**, 39.
4. Klienberg, R.L., Straley, C., Kenyon, W.E., Akkurt, R., and Farooqui, S.A., "Nuclear Magnetic Resonance of Rocks: T_1 vs. T_2", SPE 26470, (1993).
5. Gullion, T., "The effect of amplitude imbalance on compensated Carr-Purcell sequences", *Journal of Magnetic Resoance Series A*, (1993) **101**, 320.