# **Measuring Relative Permeability With NMR**

M.J. Dick<sup>1</sup>, D. Veselinovic<sup>1</sup>, T. Kenney<sup>1</sup> and D. Green<sup>1</sup>

<sup>1</sup>Green Imaging Technologies, Fredericton, NB, Canada

This paper was prepared for presentation at the International Symposium of the Society of Core Analysts held in Trondheim, Norway, 27-30 August 2018

#### Abstract

Whether they are used to complement existing measurements, or completely replace traditional methods of data acquisition in lieu of speed, accuracy, cost-effectiveness and/or other benefits, NMR techniques have become an indispensable tool in oil and gas exploitation. This project will utilize NMR capabilities in unsteady state relative permeability measurements.

In conventional relative permeability measurement, fluid saturations, pressure differences and fluid flow rates are monitored and, from these values, relative permeability can be calculated. Typically, the fluid saturation monitoring relies on mass or volume measurements acquired using acoustic and/or optical instruments. We propose a more elegant technique to monitor the saturations via NMR  $T_2$  distributions. This NMR relative permeability measurement should be more accurate than the conventional method since the saturations are measured directly in the rock rather than from mass or volume measurements. An NMR relative permeability measurement has been developed and will be presented in this paper.

To test the feasibility of this method, a carbonate initially saturated with dodecane was confined in an NMR overburden probe. NMR invisible  $D_2O$  based brine was then pushed through the rock at a fixed rate. A  $T_2$  spectrum was recorded every few minutes and the resulting pore volumes were employed to retrieve the oil produced from the rock as a function of time. Simultaneously, the pressure drop across the rock was also measured as a function of time. The resulting data set was then employed to determine a relative permeability plot for the rock. An overview of how this experiment is conducted, how the pressure drop across the rock is recorded simultaneously with the NMR  $T_2$  distributions and other experimental hurdles in this measurement is presented.

#### Introduction

Relative permeability analysis is one of the most important petrophysical parameters assessed in special core analysis experiments. Traditionally, relative permeability is measured via one of two methods: steady state and unsteady state [1,2]. In the steady state method, two immiscible fluids are injected simultaneously into the core plug at constant flow rates but with different flow rate ratios. The pressure drop across the core plug is monitored and once it has stabilized, the relative permeability is determined by

application of Darcy's law [3]. Waiting for the pressure drop across the core to stabilize makes the steady state relative permeability experiment time consuming and as a result, the unsteady state relative permeability measurement is generally the quicker method. In the unsteady state relative permeability measurement, the core plug is saturated with one immiscible fluid. The other fluid is then injected into the core and displaces the first fluid. The pressure drop across the core and the average saturation by material balance (via collection of effluent volumes at the outlet) in the core are monitored as a function of time. For example, if the core is initially saturated with oil and brine is used to displace it, the amount of oil ejected as a function of time would be recorded. The monitoring of the amount of oil ejected can either be done by eye or with the assistance of optical/acoustic instruments which monitor the position of time is known, a modified version of Darcy's law is employed to derive the relative permeability curve [4,5].

NMR can also be employed to measure the average saturation in-situ and hence derive the relative permeability of a rock core sample. It is known that NMR is a very effective and accurate way of measuring the capillary pressure curve [6]. Previous attempts to measure the relative permeability via NMR [7-9] have tried to take advantage of this strength by measuring both capillary pressure curves and saturation profiles as a function of pressure across the core. This data is then coupled with a modified version of the Darcy equations to yield the relative permeability.

In this paper, we present a completed unsteady state relative permeability measurement where the saturation of the core has been tracked as a function of time using NMR  $T_2$ volume measurements. This is a very similar measurement to the conventional unsteady state measurement. However, we believe this measurement should be more accurate than the conventional method because it directly measures the in-situ saturation profiles in the core rather than relying on the material balance method, where factors such as dead volumes and instrument uncertainties can lead to errors in estimating core saturations. In addition, we think that this measurement should outperform other NMR relative permeability measurements as it uses the same well-established method for determining relative permeability and doesn't rely on less well-established methods involving the measurement of capillary pressure.

#### Experimental

A carbonate core plug was chosen for the relative permeability study. The characteristic information on the rock is shown in Table 1 and the overall experimental setup employed in this study is shown in Figure 1. The core was initially saturated with dodecane and then the sample was confined hydrostatically at a pressure of 4500 psi (using Fluorinert as confining fluid) in an Oxford Instruments P5 overburden NMR probe [10]. Fluorinert is chosen as the confining fluid as it is NMR invisible in our experiment and thus won't interfere with any NMR signals from the core itself. The probe is then inserted into in an

Oxford Instruments GeoSpec 2-53 rock core analyzer [11]. Once inside the analyzer, the confining pressure is kept constant using a Vindum Pump [12].

The P5 overburden probe is designed for flow experiments and has inlet and outlet lines going to the top and bottom of the rock. The next step in the experimental setup was to connect flow lines to each end of the rock and prime the inlet line with dodecane. Priming of the line is important to avoid pushing air through the rock changing its initial saturation condition. Flow of liquids through the rock is achieved by employing a HPLC pump [13]. The outlet line is plumbed directly to a waste beaker.

The success of the relative permeability experiment hinges on accurate measurement of pressure, specifically the pressure difference across the rock core. To ensure this is an accurate measurement, pressure transducers are placed on the inlet and outlet flow lines, as well as, the confining pressure line. A data acquisition system was designed in which an Arduino microcontroller [14] continually logs the pressure from the transducers to an internal website. A server application then poles the website once a minute creating a log of the system pressures which also includes a time stamp for each measurement. This log is crucial in ensuring that accurate pressures can be correlated to the NMR  $T_2$  measurements.

The relative permeability experiment began by flowing 2% KCl in D<sub>2</sub>O brine through the rock at 0.1 ml/min using the HPLC pump. The D<sub>2</sub>O brine was chosen as a surrogate for H<sub>2</sub>O brine as D<sub>2</sub>O is NMR invisible. The invisibility of D<sub>2</sub>O is important to ensure that any NMR signal observed will only be from dodecane so that the relative saturation levels of D<sub>2</sub>O/dodecane can easily be derived from the observed NMR signal and the pore volume of the rock. The NMR measurements were begun simultaneously with the flow of  $D_2O_2$ . The relative permeability derived from this experiment will not be reflective of reservoir conditions as it does not employ live crude oil. However, there is no reason that this NMR relative permeability measurement could not be employed for use with live crude. A T<sub>2</sub> spectrum and an NMR saturation profile were both measured throughout the experiment and their NMR parameters can be found in Table 2. The  $T_2$ spectrum was used to monitor the bulk saturation levels in the rock, while the saturation profile was used to track the position of the D<sub>2</sub>O/dodecane front as dodecane was ejected from the rock. The saturation profile was not necessary for the calculation of relative permeability. It was recorded only for investigational purposes. Data acquisition and analysis of the NMR data were achieved via Green Imaging Technology software [15]. The software includes a loop function that allows the NMR measurements to be run continuously. The T<sub>2</sub> measurement takes about 3 minutes to complete while the NMR saturation profile has an 11 minutes experiment time. As a result, each NMR measurement was spaced by approximately 14 minutes. As mentioned above, pressure measurements were continually recorded and their time stamps allowed the pressures to

be correlated to the corresponding NMR measurements (which also have independent time stamps).

#### Results

Figure 2 shows three  $T_2$  measurements taken at three different times throughout the experiment. The y-axis is the NMR signal in units of equivalent water volume. The NMR spectrometer is calibrated in units of equivalent water volume. The black measurement was taken prior to the initiation of D<sub>2</sub>O flow. This spectrum is 100% dodecane saturated spectrum. The red spectrum was taken after D<sub>2</sub>O has entered the core but, as will be explained, prior to breakthrough of D<sub>2</sub>O from the outlet face of the core. The blue spectrum is taken towards the end of the experiment after breakthrough of D<sub>2</sub>O from the outlet face. The area under any of these spectra is the amount of dodecane in the core at the time of the measurement. As will now be explained, plotting the area under these T<sub>2</sub> spectra as a function of time is how the data was derived to calculate the relative permeability of the core in this experiment.

The upper panel of Figure 3 shows the NMR signal plotted as a function of time, as well as, the pressure difference across the rock plotted as a function of time. This data was recorded as  $D_2O$  was being injected through the core sample at 0.1 ml/min. Therefore, the NMR signal observed is proportional to the amount of dodecane in the rock as any  $D_2O$  in the rock will be NMR invisible. During the first approximately 50 mins (Figure 3- black lines) both the pressure across the core and the amount of dodecane in core remains constant. This is because, as mentioned above, the inlet flow lines were primed with dodecane. As a result, it takes about 50 minutes for the  $D_2O$  to reach the core. The dead volume in our system can be estimated as 50 min x 0.1 ml/min = 5 ml.

After 50 minutes, the NMR signal observed from the rock begins to decrease. This corresponds to  $D_2O$  reaching the core plug (Figure 3 - red lines) and beginning to replace dodecane in the plug. The amount of decrease in NMR signal is proportional to the amount of  $D_2O$  entering the rock and dodecane leaving the rock. During this same period, the pressure increases because the two immiscible fluids are both being pushed through the rock simultaneously. The pressure increases until approximately the 80 minutes mark when a peak pressure of 1400 PSI is reached. This peak corresponds to breakthrough of  $D_2O$  through the outlet face of the rock. The pressure slowly decreases after this as the rate of dodecane being evacuated by the  $D_2O$  decreases. This is reflected in a reduction in the rate of change of the amount of NMR signal observed. Eventually very little dodecane is produced from the core (Figure 3 – blue lines) and only  $D_2O$  is moving through the core (i.e.  $S_{or}$  is reached).

To derive relative permeability from this data, the NMR signal observed as a function of time must be converted to oil produced as a function of time. To do this, the NMR signal in units of volume of water is converted to oil produced by subtracting each measurement

from the pore volume of the rock. It should be noted that dodecane and water have a similar hydrogen index so there is no need to convert NMR signal in ml of water to ml of dodecane prior to doing the subtractions. The dodecane produced as a function of time is plotted in the lower panel of Figure 2.

Figure 4 shows the saturation profiles which were also recorded as a function of time during the experiment. The color scheme in this figure follows that of Figure 2. The black saturation profiles were recorded during the period before  $D_2O$  enters the rock core and as a result are all very similar. The red saturation profiles were recorded in the period immediately after  $D_2O$  enters the core. A  $D_2O$ /dodecane boundary is clearly visible moving across the core from left to right. Finally, the blue saturation profiles were recorded after the majority of the dodecane has been ejected from the rock. The amount of dodecane in the rock is again relatively stable. Although, these saturation profiles were not actively used in the calculation of relative permeability, they are still useful to visualize what is going on inside the core during the experiment. Also, the area under each saturation profile can be employed as an independent verification of the amount of oil produced as a function of time.

To derive the relative permeability from the experimental data, only those measurements taken after D<sub>2</sub>O breakthrough at the outlet face occurs are to be considered. Figure 5 shows both the oil produced (red plot) and pressure across the rock (blue plot) after breakthrough. The figure also includes least-square fits of the reduced data set to polynomial equations (black plots) of time. Once the expressions for oil produced and pressure difference as a function of time are known, the derivations of relative permeability follows the Johnson-Bossler-Naumann relative permeability calculation [2-5]. Figure 6 shows the relative permeability curves for oil (red plot) and water (blue plot) derived for this rock via a NMR measurement.

#### **Conclusions and Future Work**

This paper summarizes a conventional relative permeability measurement done with NMR. As with any conventional relative permeability measurement it relies on waiting for the brine to breakthrough the outlet face before beginning the relative permeability calculation. This often restricts which saturations levels can be observed on the relative permeability curve. For example, in this experiment the relative permeability for both water or oil was not determined until the water saturation reached 0.4 (Figure 6). Traditionally, this is because until breakthrough occurs it is uncertain as to what is occurring in the rock (i.e. saturation, flow rate etc.). However, NMR has the added benefit of being able to visualize what is going on inside the rock. For example, the saturation profiles seen in Figure 4 show where in the rock the  $D_2O/dodecane$  front is before it emerges from the outlet face. We are now working at how to best exploit this NMR capability. A second NMR measurement to determine the flow rate as a function

of position within the rock is under way. This should allow relative permeability prior to breakthrough to be calculated.

## References

- 1. Bennion, D.B., and Thomas, F. B., Recent Improvements in Experimental and Analytical Techniques for Determination of Relative Permeability from Unsteady State Flow Experiments, SPE 10<sup>th</sup> Technical Conference and Exposition, Trinidad, 1991.
- 2. McPhee, C., Reed, J. and Zubizarreta, I., *Core Analysis: A Best Practice Guide*, Elsevier, Amsterdam (2015), 519-651.
- 3. Darcy, Henry, *Les Fontaines Publiques de la Ville de Dijon*, Victor Dalmont, Paris (1856).
- 4. Johnson, E. F., Bossler, D. P. and Naumann, V. O., "Calculation of Relative Permeability from Displacement Experiments", Trans. AIME (1959), **216**, 370-373.
- 5. Welge, H. J., A Simplified Method for Computing Oil Recovery by Gas Or Water Method", Trans. AIME (1952), **195**, 91-98.
- 6. Green, D., Dick, J., Gardner, J., Balcom, B. and Zhou, B., "Comparison Study of Capillary Pressure Curves Obtained Using Traditional Centrifuge and Magnetic Resonance Imaging Techniques", Society of Core Analysts, Calgary, Alberta, Canada 9-14 September 2007.
- 7. Romanenko, K. and Balcom, B.J.,"An Assessment of Non-Wetting Phase Relative Permeability in Water-Wet Sandstones Based On Quantitative MRI Of Capillary End Effects", Journal of Petroleum Science and Engineering (2013), **110**, 225-231.
- 8. Zhao, Y. The Guide to Measurement and Estimation Of Oil-Water Relative Permeability And Capillary Pressure From The Saturation Profile Of Capillary End Effect (CEE), Fredericton, NB (2014).
- 9. Liao, G., Romanenko, K. V., Marica, F., Balcom, B. J., *Measuring Two-Phase Relative Permeability from Unsteady-State Dynamic Flooding Experiments with Magnetic Resonance Imaging*, Fredericton, NB.
- 10. P5 Overburden Probe User Manual, Version 1, Oxford Instruments, Green Imaging Technologies.
- 11. Geo-Spec 2-75 User Manual, Version 1.8, Oxford Instruments.
- 12. VP-Series Pump User Guide, Version 1.2, Vindum Engineering Inc.
- 13. VersaGrad Model-12X6 Binary HPLC Pump Operator's Manual 90-2889 rev A, Scientific Systems Inc.
- 14. Arduino Microcontroler, www.arduino.cc.
- 15. GIT Systems and LithoMetrix User Manual, Revision 1.9, Green Imaging Technologies

## **Tables and Figures**

Table 1: Properties of core plug studied

Depth (m)	6390
Core Diameter (cm)	2.53
Core Length (cm)	4.88
Dry Core Mass (g)	60.59
Pore Volume (mL)	2.74
Bulk Volume (mL)	24.53
Irreducible Water Saturation	0.1972
Grain Volume (mL)	22.27
Grain Density (g/mL)	24.53
Archimedes Porosity (p.u.)	9.21
Brine Permeability (mD)	0.05

Table 2: Acquisition parameters for  $T_2$  and saturation profile measurements

Parameter	<b>T</b> <sub>2</sub>	Parameter	Saturation Profile
Recycle delay (ms)	5000	Recycle delay (ms)	5000
Tau (µs)	55	Tau (µs)	500
Number of Echoes	45455	Gradient Duration (µs)	300
Filter Width (kHz)	125	Filter Width (kHz)	125
$T_2$ Max (ms)	1000	Resolution	64
90° Pulse Length (µs)	4.75	90° Pulse Length (µs)	4.75
180° Pulse Length (µs)	9.71	180° Pulse Length (µs)	9.71

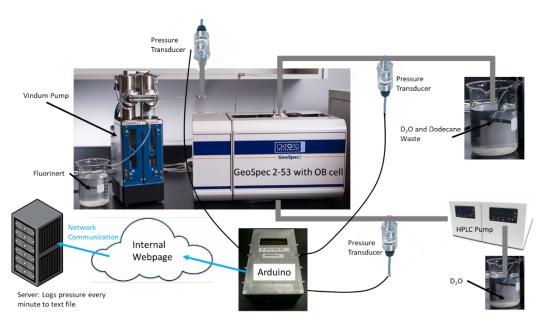


Figure 1: Experimental setup employed to acquire relative permeability via NMR.

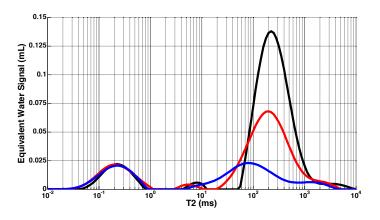


Figure 2: Typical  $T_2$  spectra recorded at various times during relative permeability experiment  $D_2O$  flooding.

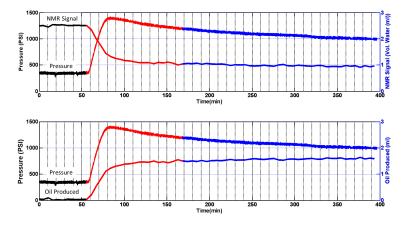


Figure 3: NMR signal and pressure across the rock core as a function of time (Upper Panel). Oil produced and pressure across the rock core as a function of time (Lower Panel).

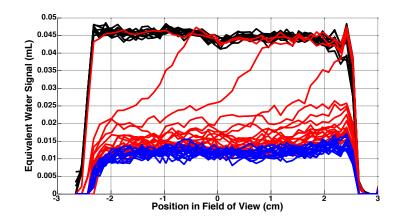


Figure 4: Series of saturation profiles recorded as  $D_2O$  was flooded through the core during the relative permeability experiment.

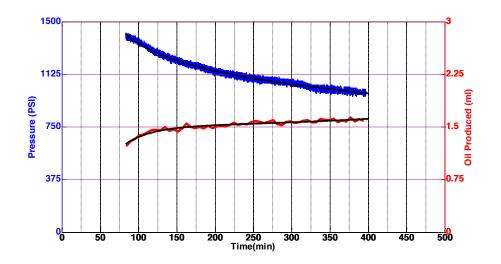


Figure 5: The pressure across the core (blue trace) and oil produced from the core (red trace) after breakthrough of  $D_2O$  from the outlet face of core.

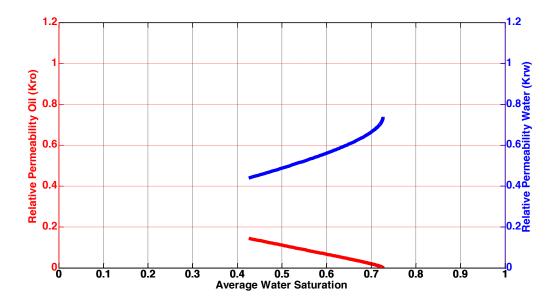


Figure 6: The relative permeability curves generated from NMR data. The relative permeability to water is shown in blue while the relative permeability to dodecane is shown in red.