COMPARISON STUDY OF CAPILLARY PRESSURE CURVES OBTAINED USING TRADITIONAL CENTRIFUGE AND MAGNETIC RESONANCE IMAGING TECHNIQUES

Derrick P. Green\textsuperscript{1}, Josh R. Dick\textsuperscript{1}, John Gardner\textsuperscript{2}, Bruce J. Balcom\textsuperscript{3} and Bing Zhou\textsuperscript{3}

\textsuperscript{1}Green Imaging Technologies, 46 Dineen Drive, Fredericton, NB, Canada
\textsuperscript{2}CoreLab, 6316 Windfern Road, Houston, TX, USA
\textsuperscript{3}University of New Brunswick, Fredericton, NB, Canada

This paper was prepared for presentation at the International Symposium of the Society of Core Analysts held in Calgary, Canada, 10-12 September, 2007

ABSTRACT

The current study is a market validation trial of the new Magnetic Resonance Imaging (MRI) method for acquiring capillary pressure. This study is meant to evaluate the methodology and workflow on site with CoreLab in Houston, TX. This study compares gas-water capillary pressure results measured using a traditional centrifuge method and the new MRI based method.

Traditional centrifuge capillary pressure measurements require the fluid(s) to reach equilibrium at many different speeds. This is very time consuming as each equilibrium step can take a couple of days. A new method, trademarked by Green Imaging Technologies (GIT) as GIT-CAP (GIT CAPillary pressure), centrifuges the core plugs then directly measures the water saturation distribution in the core plug using magnetic resonance imaging. The measured water saturation together with the known centrifugal force directly leads to a capillary pressure curve.

Traditional MRI methods have difficulty in relating the detected signal intensity to water or oil saturation. This is because traditionally the MRI image intensity depends on the environment of hydrogen atoms. In this work, we use a new MRI method, one dimensional centric scan Single-point Ramped Imaging with T1 Enhancement (SPRITE), in which the detected signal is directly proportional to the amount of water or oil present.

The new technique measures the capillary pressure curve more quickly and accurately. It is also 3-5 times faster due to the fact that only two to three centrifuge speeds are required (versus 7-10). In some rock types, this reduces the measurement duration from many weeks to days. The new technique is also potentially more accurate as it directly measures the water saturation in the rock instead of relying on a calculation using a measurement of the expelled water. The current study focuses on gas-water systems comparing traditional centrifuge capillary pressure measurements with the new MRI based method.
INTRODUCTION

Laboratory measurements of capillary pressure are typically performed by mercury injection, porous plate, or centrifugation [Dullien (1991)]. The porous plate method is considered the most direct and accurate method but is extremely time consuming since each capillary pressure point requires an equilibrium time that can take weeks or months. The mercury injection method is rapid and can reach very high capillary pressures but the test uses a non-representative fluid, mercury, and it is destructive. A common compromise between porous plate and mercury injection is centrifugation. This method uses reservoir fluids and decreases the equilibrium time by using a centrifuge.

The centrifuge method was first introduced by Hassler and Brunner (1945). It involves rotating fluid bearing rock core plugs at various speeds and measuring the amount of water expelled from the core at each of the rotational speeds. The measured expelled water can then be used to calculate the average water saturation in the core plug [Ruth and Chen (1995), Melrose (1986), Rajan (1986)]. At each speed, hydrostatic equilibrium is obtained balancing the centrifugal force with the capillary pressure in the rock core plug. This equilibrium time can take many days in some rock types. The capillary pressure curve is obtained by plotting the capillary pressure at various, usually seven to ten, rotational speeds versus the inlet face water saturation.

In the current investigation, we have used a new quantitative MRI method [US Patent application 11/262,658, Chen and Balcom, (2005, 2006)] to determine fluid saturation along the length of the sample instead of a non-quantitative spin echo based MRI [Baldwin and Spinler, (1998)], or nuclear tracer imaging techniques [Graue et al. (2002)].

Traditional Centrifuge Capillary Pressure

Hassler and Brunner (1945) proposed a centrifuge method to determine capillary pressure-saturation data from small core plugs. Neglecting gravitational effects and assuming the core length is negligible compared to the radius of rotation leads to an approximate solution to the basic equation relating capillary pressure and average saturation.

In a traditional centrifuge capillary pressure experiment, a fluid saturated core plug, confined in a special core-holder, is rotated at different rotational speeds. The relevant distances, denoted as r1, r2 and r, are the distances from the rotational axis to the inlet face, the outlet face, and any point along the core length, respectively, as shown in Figure 1(a). The core-holder contains another fluid which replaces the fluid displaced from the core. After reaching hydrostatic equilibrium, the amount of liquid expelled from the core plug is measured. The capillary pressure distribution and the water saturation along the length of the core are illustrated schematically in Figure 1(b) and 1(c). The fluid saturation distribution can be directly determined from MRI measurements but not by traditional measurements.
The basic concepts for capillary pressure measurement with a centrifuge are outlined. When a cylindrical core is placed in a centrifuge, a centrifugal acceleration $a_c = -\omega^2 r$, is generated, where $\omega$ is the angular rotation speed of the centrifuge and $r$ is the distance from the axis of rotation. Applying Darcy’s law at hydrostatic equilibrium, we have

$$\frac{dP_c}{dr} + \Delta \rho \omega^2 r = 0$$  \hspace{1cm} (1)

where $\Delta \rho$ is the density difference between wetting fluid and non-wetting fluid. The differential equation can be solved by simple integration and application of the Hassler-Brunner boundary condition, i.e., $P_{c2} = 0$, so

$$P_c(r) = \frac{1}{2} \Delta \rho \omega^2 (r_2^2 - r_1^2)$$  \hspace{1cm} (2)

The capillary pressure at the inner face of the core is given by

$$P_{c1}(r) = P_c(r_1) = \frac{1}{2} \Delta \rho \omega^2 (r_1^2 - r_2^2)$$  \hspace{1cm} (3)

The capillary pressure distribution results in a fluid saturation distribution along the length of the core. Neither of these distributions is actually measured with the traditional method. What is measured is the rotational speed, $\omega$, and the average fluid saturation, $\bar{S}$, within the core. The average fluid saturation of the core after centrifugation can be expressed as

$$\bar{S} = \frac{1}{r_2 - r_1} \int_{r_1}^{r_2} S(r) dr$$  \hspace{1cm} (4)

Equation (4) may be rewritten by changing the integration variable $P_c(r_2)=0$ and $P_c(r_1)=P_{cL}$, with additional mathematical manipulation, which yields the Hassler-Brunner integral equation

$$\bar{S}P_{cL} = \frac{P_c}{2r_2} \int_0^{P_c} \frac{S(P_c)}{\sqrt{1 - \frac{P_c}{P_{cL}} (1 - \frac{r_1}{r_2})}} dP_c$$  \hspace{1cm} (5)

Equation (5) cannot be directly solved for the unknown function S. Assuming short cores (i.e. $r_1/r_2 \approx 1$), then equation (5) is reduced to

$$\bar{S}P_{cL} = \frac{P_c}{2r_2} \int_0^{P_c} S(P_c) dP_c$$  \hspace{1cm} (6)

which in differential form is

$$S_L = \frac{d(\bar{S}P_{cL})}{dP_{cL}}$$  \hspace{1cm} (7)

The value of $P_{cL}$ is then calculated at each rotational speed based on equation (3), and the value of the saturation at the inlet face, $S_L$, is obtained according to equation (7). A plot of these two values, $P_{cL}$ and $S_L$, at different rotation speeds yields the capillary pressure curve. Note that equation (7) is an approximate solution proposed by Hassler and Brunner. Based on equation (5), a number of other approximate solutions have been

**Magnetic Resonance Imaging**

Nuclear Magnetic Resonance (NMR) detects the amount of hydrogen (for proton NMR) in the sample or object under study. The hydrogen atoms are excited by using radio frequency (RF) energy at the NMR resonance frequency of the hydrogen. The lifetime of the signals detected after excitation depends on the environment of the hydrogen. For example, signal detected from the hydrogen in oil decays away quicker than the hydrogen in free water. Magnetic resonance imaging (MRI) spatially resolves the nuclear magnetic resonance (NMR) signal. Spatially resolving the MRI signal is achieved by linearly altering the magnetic field creating a magnetic field gradient. Both the field of view and the resolution are limited by the linear region and strength of the magnetic field gradient. A wide variety of different pulse sequences (combinations of gradient, excitation, and detection schemes) are available.

The standard SPRITE MRI [Balcom et al. (2003, 1996)] technique has proven, over the last 10 years, to be a very robust and flexible method for the study of a wide range of systems with short Magnetic Resonance (MR) relaxation times. As a pure phase encoding technique, SPRITE is largely immune to image distortions due to susceptibility variation, chemical shift, and paramagnetic impurities. Repetitive excitation and data acquisition are performed in the presence of ramped phase encoding gradients, which enable systems with $T_2^*$ lifetimes as short as tens of microseconds to be successfully visualized.

A centric scan strategy for SPRITE MRI [Mastikhin (1999)] removes the longitudinal steady state from the image intensity equation of standard SPRITE imaging, and increases the inherent image intensity. The image signal intensity no longer depends on the spin-lattice relaxation time ($T_1$) and the repetition time. These features ensure that centric scan SPRITE is an ideal method for quantitative imaging of sedimentary rocks with short relaxation times.

A 1D double half k-space SPRITE technique, also called 1D centric scan SPRITE, is illustrated in Figure 2. In this method the k-space data are acquired sequentially from '0' to '-kz', corresponding to a gradient change from 0 to minus maximum gradient (-Gmax), after a delay of 5 times $T_1$, the other half of the k-space data is collecting from '0' to '+kz', corresponding to a gradient change from 0 to a maximum gradient (Gmax). Fourier transformation of the k-space data yields a real space image. In the centric scan SPRITE method, the image signal intensity ($S$) is given by:

$$S = M_0 \exp\left(-\frac{t_p}{T_2^*}\right) \sin \alpha$$

(8)

where $M_0$ is the equilibrium magnetization, $\alpha$ is the RF flip angle, $t_p$ is the phase encoding time, $T_2^*$ is the effective spin-spin relaxation time. $M_0$ is directly proportional to
the local fluid content. Centric scan SPRITE methods are naturally fluid content weighted. In this sequence the field of view is governed by

\[ FOV = \frac{2\pi}{\gamma \Delta G t_p} \]  

(9)

where \( \Delta G \) is the incremental magnetic field gradient step. The resolution of the resultant profile is

\[ \text{Resolution} = \frac{FOV}{N} = \frac{\pi}{\gamma G_{\text{max}} t_p} \]  

(10)

Where \( G_{\text{max}} \) is the maximum magnetic field gradient and \( N \) is the number of gradient steps.

A wide range of experimental results [Chen et al. (2005)] show that the overall Free Induction Decay (FID) decay rate (1/ \( T_2^* \)) in fluid saturated sedimentary rocks is dominated by an internal field distribution (\( \Delta B' \)) induced by the large susceptibility difference (\( \Delta \chi \)) between the pore filling fluid and solid matrix due to paramagnetic impurities in the solid matrix. The decay rate of the FID and the corresponding MR linewidth (\( \Delta \nu=1/\pi T_2^* \)) for fluid saturated sedimentary rocks may be estimated by [Chen et al. (2005)],

\[ \frac{1}{\pi T_2^*} = \Delta \nu \approx \frac{\gamma \Delta B'}{2\pi} = \frac{C \Delta \chi B_0}{2\pi} \]  

(11)

where \( \gamma \) is the gyromagnetic ratio, and \( B_0 \) is the applied magnetic field strength, while \( C \) is a dimensionless constant.

Equation (11) predicts a single exponential \( T_2^* \) decay: this prediction has been confirmed by a wide range of MR experiments for sedimentary rocks [Chen et al. (2005)]. Single exponential \( T_2^* \) decay is anticipated for a wide variety of sedimentary rock systems, but is not a universal result. Equation (11) also predicts that effective transverse relaxation time (\( T_2^* \)) is inversely proportional to the applied magnetic field strength (\( B_0 \)). It suggests that low field MR will lead to a longer MR signal lifetime. Figure 3 shows a semi-logarithmical FID decay after a 90 degree RF excitation pulse for a Berea sandstone. The data was fit to the equation:

\[ S = M_0 \exp \left( -\frac{t}{T_2^*} \right) \]  

(12)

where \( S \) is the MR signal intensity and \( t \) is the acquisition time. The fit \( T_2^* \) was 127 µs. We have observed for many sedimentary rocks that \( T_2^* \) is largely insensitive to water saturation with a single exponential FID. These features ensure that Centric Scan SPRITE methods permit quantitative imaging. Quantitative images are frequently impossible with spin echo based MRI methods, due to multi-exponential \( T_2 \) decay in porous rocks [Baldwin and Spinler (1998)].
**MRI-based Capillary Pressure**

Capillary pressure theory combined with MRI determined saturation profiles allow us to obtain capillary pressure curves. With this technique, the centrifuge is used to create a distribution of fluid in the rock core plug dependent on capillary pressure and then quantified using MRI. The capillary pressure at each position down the rock at hydrodynamic equilibrium is known from equation (2). The saturation at the corresponding positions is measured using MRI. The fully saturated profile gives us the 100% saturation level. We know that the 0% saturation level will yield no MRI signal as there is no hydrogen present. Therefore, dividing the centrifuged measured profile by the 100% saturated profile gives a quantitative saturation level versus position. In fact, the MRI equipment can be calibrated such that the measured profiles directly determine saturation. Figure 4 shows a fully saturated and a series of centrifuged profiles. The radial distance is determined at each profile point knowing that one edge is the distance \( r_2 \). The capillary pressure is then computed using equation (13) at each point and plotted with the saturation percent to create a capillary pressure curve. Saturation profiles acquired after centrifugation at different speeds are plotted on the same curve expanding the range and resolution of the capillary pressure curve.

**Procedure**

In this case study we focused on primary drainage air/water capillary pressure tests. Experimental procedures for primary drainage capillary pressure curves follow:

1. Examine the core and inspect for visual damage. Exclude damaged or vuggy core from study.
2. Clean the core using Soxhlet extraction with toluene and methanol to remove any oil and salts.
3. Dry the core at 240 degrees Fahrenheit for 24 hours or until a stable weight is reached.
4. Measure helium grain volume, pore volume, and permeability. Compute porosity and compare to available data. Any discrepancies must be explained or the core plug should not be used for this study.
5. Place the sample under vacuum for 24 hours and saturate with 4% KCl brine under 2500psi pressure for 24 hours. Compare the gravimetric saturated pore volume with the helium pore volume determined in step 4. Any discrepancies must be explained or the core plug should not be used for this study.
6. Run a baseline proton (1H) MRI one-dimensional double half k-space SPRITE MRI profile measurement of fully saturated core and store the results. This is used later to determine water saturation after centrifugation.
7. Using porosity, permeability, fluid densities, and sample length, determine the rotational speed using equation (14). To study the new method several speeds were selected based on \( J \) values of 0.5, 1, 2, 4, and 8.
8. Place the core in the centrifuge and spin for 24 hours.
9. Obtain MRI profile on the centrifuged core using the same MRI sequence and acquisition parameters from step 6.
10. Determine the capillary pressure curve for primary drainage using the method described above.

11. Repeat steps 7 through 10 for various centrifuge speeds.

12. Combine all centrifuge speed data into one capillary pressure curve.

The centrifuge experiment was carried out with a high speed centrifuge modified for centrifuging rock core plugs. The MRI experiments were carried out on a 0.18 Tesla Bruker LF90 MiniSpec (Bruker Optics, Houston, TX) and proprietary software and hardware from Green Imaging Technologies (GIT, Fredericton, NB, Canada). The benefits of using a low-field MRI instrument are that 1) the instrument is relatively inexpensive, and 2) the effective spin-spin relaxation time ($T_2^*$) is much longer than the phase encoding time ($t_p$). This ensures the SPRITE MRI profile is a simple fluid saturation with no other NMR relaxation dependencies.

The fluid content profiles along the length of the core before and after centrifugation were determined by one-dimensional double half k-space SPRITE MRI with a phase encoding time of 50 $\mu$s, field of view of 7cm, flip angle of 15 degrees and a resolution of 64 points. The number of signal averages was varied to achieve the desired signal to noise ratio of 100. More data points along the length of the core can easily be obtained by increasing the image matrix size and/or decreasing the field of view of the image. This results in more data points on the capillary pressure curve.

Rotational speed for the centrifuge was determined by using the following equation, known as the Leverett J function [Leverett, 1941]

$$P_c(r) = \frac{1}{2} \Delta \rho \omega^2 (r_2^2 - r_1^2) \geq \frac{J(S_{wi}) \sigma \cos \theta}{\sqrt{k/\phi}}$$

where $J$ is the Leverett value, $\sigma$ is the normal interfacial tension, $\theta$ is the contact angle, $k$ is the permeability and $\phi$ is the porosity for a given rock. The $J$ value “normalizes” the speed using this function [Brown, (1951)].

**RESULTS**

Results for 4 different rocks are included in this paper. The basic rock properties for each sample are included in Table 1. Although it is theoretically possible to obtain a complete capillary pressure curve from a single centrifuge speed, we choose to obtain results after five equilibrium speeds. This allows us to fully investigate the method and determine how many equilibrium steps should be used. Table 2 shows the centrifuge speeds and Leverett $J$ values used for each rock.

A typical set of MRI profiles is shown in Figure 4. In this figure a fully saturated profile is shown along with the saturation profile after each of the five centrifuge speeds for sample #3. Note that the fully saturated profile is uniform or flat. If any inhomogeneity exists in the rock the fully saturated profile will show its effects. The capillary pressure
curves for samples 1 through 4 are shown in Figures 5 through 8, respectively. The plots also include a separately performed capillary pressure curve (line) determined by traditional centrifuge techniques. The traditional centrifuge capillary pressure curve was determined using a volumetric reading of the expelled fluid and the Forbes method for determining the inlet face saturation from the average saturation. There is a very good correlation between the two techniques.

Note that as few as three speeds can be used to fully define the capillary pressure curve. Figure 9 shows sample #2 with only three centrifuge speeds. Utilizing longer cores, as few as two centrifuge speeds may be required to fully define the capillary pressure curve.

DISCUSSION

This capillary pressure measurement technique directly measures the water saturation in the rock core plug. Traditional centrifuge techniques measure the expelled water and require a calculation to determine the inlet face saturation which requires simplifications and assumptions. The capillary pressure using MRI is calculated using equation (13) and requires only that the outlet boundary condition be met (i.e. 100% saturation at the outlet). The assumption that the core plug length is negligible compared to the radius of rotation is not required in this new MRI technique. In fact, we rely on the capillary pressure gradient and the subsequent saturation gradient in the core.

This technique requires the rock core plug to be moved from the centrifuge to the MRI machine. Much care and investigation has been given to the question of the redistribution of fluids between these two steps. Fluid redistribution is minimized by acquiring the MRI profiles directly after centrifugation. The MRI measurement time is typically between a few minutes to a couple of hours. In the rocks currently under study, the redistribution of fluids is insignificant in the time required to acquire the MRI profile. Figure 10 shows a MRI profile acquired after centrifugation repeated at different time intervals. In this case, the fluid still has not fully redistributed even after 5 days.

Although it would be ideal to acquire the complete capillary pressure curve in one centrifuge speed, operational and resolution restrictions may prevent this. In order to acquire a complete capillary pressure curve the centrifuge speed must be selected such that the irreducible water saturation is achieved at the inlet face of the rock. This can be estimated by equation (14) but cannot be assured for all rocks. Assuming that the outlet is at 100% saturation we would have all the capillary pressures required for a complete curve. We may, however, not have enough resolution, depending on the MRI resolution, at the lower capillary pressures to fully define the curve at this point. In addition, it would appear that the 100% saturation at the outlet may be a very thin layer which cannot be resolved at the MRI resolutions currently used. In the end, it seems apparent that at least two centrifuge speeds will be required.

The MRI signal to noise ratio will directly affect the accuracy of the capillary pressure measurement obtained. During this study we attempted to define and relate the signal to
noise ratio to acceptable accuracy in the capillary pressure results. We used a signal to noise ratio of 100 for the results reported here.

A number of additional benefits can be exploited using this technique. As briefly mentioned, the fully saturated profiles can yield inhomogeneities in the rocks as well as pore volumes. If the homogeneities are only in a portion of the rock, capillary pressure curves can still be obtained by only using the homogeneous section. Another benefit is the ability to use longer rock core plugs to increase the maximum capillary pressure obtainable. Longer rocks will not only increase the maximum capillary pressure but also increase the MRI signal to noise ratio and increase resolution.

CONCLUSIONS
In this work, we have compared a new MRI based capillary pressure measurement technique with traditional centrifuge techniques. The technique requires far fewer centrifuge equilibrium steps (as few as two) and thus decreases the measurement time for a capillary pressure curve by a factor of 3-5 times. In addition, it is believed that this measurement is inherently more accurate because the water saturation is directly measured in the rock. Future work includes expanding this work to include oil/water systems and adding overburden pressure.

ACKNOWLEDGEMENTS
DPG thanks NSERC of Canada, Atlantic Canadian Opportunities Agency, National Research Council, and Petroleum Research Atlantic Canada for their financial support. DPG would also like to thank Bruker Optics for equipment support and CoreLab for conducting the on-site trial.

REFERENCES


US patent application number 11/262,658, “Methods and apparatus for measuring capillary pressure in a sample”.
Table 1 – Rock properties for Sample #1

<table>
<thead>
<tr>
<th>Property</th>
<th>#1</th>
<th>#2</th>
<th>#3</th>
<th>#4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Porosity</td>
<td>12.8%</td>
<td>14.5%</td>
<td>14.5%</td>
<td>12.6%</td>
</tr>
<tr>
<td>Permeability</td>
<td>2.32mD</td>
<td>2.28mD</td>
<td>0.908mD</td>
<td>1.08mD</td>
</tr>
<tr>
<td>Pore Volume</td>
<td>5.51mL</td>
<td>5.65mL</td>
<td>5.47mL</td>
<td>5.38mL</td>
</tr>
<tr>
<td>Core Diameter</td>
<td>3.77cm</td>
<td>3.79cm</td>
<td>3.83cm</td>
<td>3.79cm</td>
</tr>
<tr>
<td>Core Length</td>
<td>3.84cm</td>
<td>3.76cm</td>
<td>3.84cm</td>
<td>3.76cm</td>
</tr>
</tbody>
</table>

Table 2 – Centrifuge RPM used

<table>
<thead>
<tr>
<th>J value</th>
<th>#1</th>
<th>#2</th>
<th>#3</th>
<th>#4</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.5</td>
<td>2700</td>
<td>2800</td>
<td>3500</td>
<td>3300</td>
</tr>
<tr>
<td>1.0</td>
<td>3800</td>
<td>4000</td>
<td>4900</td>
<td>4600</td>
</tr>
<tr>
<td>2.0</td>
<td>5300</td>
<td>5600</td>
<td>7000</td>
<td>6500</td>
</tr>
<tr>
<td>4.0</td>
<td>7600</td>
<td>7900</td>
<td>9900</td>
<td>9200</td>
</tr>
<tr>
<td>8.0</td>
<td>10700</td>
<td>15000 (J=16)</td>
<td>14000</td>
<td>13000</td>
</tr>
</tbody>
</table>

Figure 1 – (a) Schematic of a rock core plug spinning in a centrifuge. (b) Capillary pressure distribution along the length of the core. (c) Water saturation distribution along the length of the core.

Figure 2 – The double half k-space version of the SPRITE MRI method.

Figure 3 – A semi-logarithmical plot of the NMR free induction decay (FID) of the water saturated Brea sandstone.

Figure 4 – MRI profiles of sample #3. The fully saturated profile is the uniform profile showing the homogeneity of the rock. The remaining profiles are acquired after successively higher centrifuge speeds.
Figure 5 – MRI determined capillary pressure curve for sample #1 (all points). The line is the fitted line from a traditional capillary pressure centrifuge measurement.

Figure 6 – MRI determined capillary pressure curve for sample #2.

Figure 7 – MRI determined capillary pressure curve for sample #3.

Figure 8 – MRI determined capillary pressure curve for sample #4.

Figure 9 – MRI determined capillary pressure curve for sample #2 using three centrifuge speeds.

Figure 10 – A series of MRI profiles taken after centrifugation (units are hours). Successive profiles show the fluid slowly returning to equilibrium.