

Oil/Water Imbibition and Drainage Capillary Pressure Determined by MRI on a Wide Sampling of Rocks

Derrick P. Green¹, Josh R. Dick¹, Mike McAloon², P.F. de J. Cano-Barrita³, Jon Burger⁴,
and Bruce Balcom²

¹Green Imaging Technologies, 46 Dineen Drive, Fredericton, NB, Canada

²University of New Brunswick, Fredericton, NB, Canada

³CIIDIR IPN U. Oaxaca, Oaxaca, Mexico

⁴ChevronTexaco, Houston, TX, USA

This paper was prepared for presentation at the International Symposium of the Society of Core Analysts held in Abu Dhabi, UAE 29 October-2 November, 2008

ABSTRACT

This work investigates the results of using of a new magnetic resonance imaging (MRI) based method for acquiring primary drainage, imbibition and secondary drainage capillary pressure curves for oil/water systems. The technique uses a centrifuge to create a distribution of fluid and capillary pressures in a standard rock core plug and then uses a new quantitative MRI method to measure the saturation in the rock. This allows for many (approximately forty) capillary pressure/saturation points to be acquired for each centrifuge speed, dramatically decreasing measurement time. Also, the technique does not require the measurement of the expelled fluid as the fluid is directly measured in the rock using MRI. This allows for the oil/water contact to be placed half way up the rock enabling the measurement of both the positive and negative portions of the imbibition and secondary drainage curves. This work also presents information on how to integrate traditional T_2 cut-off measurements into MRI-based air/water capillary pressure measurements.

INTRODUCTION

Capillary pressure, P_c , is the difference in pressure across the interface between two immiscible fluids and is dependent on the interfacial tension, pore size, and wetting angle. Capillary pressure is the most fundamental rock-fluid property in multi-phase flow, just as porosity and permeability are for single phase flow in oil and gas reservoirs (Lake 1989). Capillary pressure curves directly determine the irreducible water saturation, residual oil saturation, rock wettability, and can be used to determine water oil contact point and approximate oil recovery. Water flood performance is also significantly affected by the capillary pressure of the rock (Masalmeh 2003).

Capillary pressure is typically measured in the laboratory by using mercury injection, porous plate, or centrifugation techniques (Dullien 1991). The porous plate method is considered the most direct and accurate method but takes a long time since each capillary pressure point requires an equilibrium time that can take weeks or months. The mercury injection method is fast 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 (Hassler and Brunner 1945). This

method uses reservoir fluids and decreases the equilibrium time by using high centrifugal forces.

We are using a new method (US Patent 7,352,179) that measures capillary pressure employing a centrifuge and a new quantitative magnetic resonance imaging (MRI) method for measuring fluid saturation. The capillary pressure is calculated from the Hassler and Brunner equation at each radial position in the rock. This, together with saturation as measured by MRI at each position, directly produces a capillary pressure curve with as few as one centrifuge equilibrium speed.

Previous investigations concentrated on air/brine capillary pressure systems (Green et al 2007 and 2008, Chen and Balcom 2005 and 2006). In the current investigation, we focus on oil/brine capillary pressure systems including primary drainage, imbibition and secondary drainage. A number of distinct advantages arise from the technique including the ability to measure both the positive and negative portions of the imbibition and secondary drainage capillary pressure curves. In addition, we show the advantages of integrating the air/brine capillary pressure measurement with traditional nuclear magnetic resonance (NMR) relaxometry.

Traditional Centrifuge Pc Measurement

Hassler and Brunner (1945) proposed a centrifuge method to determine capillary pressure saturation data from small core plugs. In this method, a fluid saturated core plug, confined in a special core-holder, is rotated at different rotational speeds as shown in Figure 1.

In the Figure, the relevant distances, denoted as r_2 , r_1 and r , are the distances from the rotational axis to the inlet face, the outlet face, and any point along the core length, respectively. The capillary pressure can be calculated at any position, r , along the rock using the Hassler-Brunner equation:

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

where $\Delta\rho$ is the density difference between wetting fluid and non-wetting fluid and ω is the angular rotation speed of the centrifuge.

In the traditional centrifuge method, a core-holder contains another fluid which displaces the fluid in the core plug. After reaching hydrostatic equilibrium, the amount of fluid expelled from the core plug is measured and therefore the average saturation is known. The inlet face saturation can be approximated using the average fluid saturation (Hassler and Brunner 1945, Ruth and Chen 1995, Forbes 1997, Rajan 1986). This saturation is plotted against the capillary pressure from equation (1) to produce one point on the capillary pressure curve. This procedure is repeated 7-10 times at different centrifuge speeds to fully define the capillary pressure curve.

For our method, we use MRI to directly measure the saturation in the core plug itself. This, combined with the known capillary pressure from equation (1), gives us many points on our capillary pressure curve at each centrifuge speed.

Magnetic Resonance

NMR detects the amount of hydrogen (for proton NMR) in the sample or object under study. The lifetime of the detected NMR signal depends on the environment of the hydrogen. For example, signal detected from the hydrogen in oil decays faster than the hydrogen in free water. In oil field exploration, we can use this to distinguish between oil, water and gas in rocks but more importantly we can distinguish between clay bound (CBW), capillary bound (BVI) and free fluid (FFI) quantities in the reservoir. A common approach to determine these quantities is to use either T_2 or T_1 relaxation times. We know that the NMR relaxation parameters T_1 and T_2 follow the following equations:

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

$$\frac{1}{T_1} = \frac{1}{T_{1-Bulk}} + \rho \frac{S}{V} \quad (3)$$

Where ρ is the relaxometry constant, D_o is the free diffusion constant for the fluid, γ is the gyromagnetic ratio, G is the internal field gradient, T_E is the echo time (a measurement parameter), and S/V is the surface to volume ratio of the pores. The equations reduce to direct relationships to the surface to volume ratio due to the fact that, in rocks, the bulk relaxation times are much longer than the measured values and we select an echo time (T_E) such that the diffusion term can be ignored. The surface to volume ratio is a measure of the pore size distribution of the rock.

MRI spatially resolves the 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 main difficulty with NMR (or MRI) is that because the signals are dependent upon so many things (amount of hydrogen, pore size, fluid diffusion, etc), obtaining quantitative results can be difficult.

A centric scan strategy for SPRITE MRI (Balcom et al 1996 and Mastikhin 1999) removes the longitudinal steady state from the image intensity equation of standard SPRITE imaging, increases the inherent image intensity, and makes the detected signal only depend on the amount of hydrogen. The image signal intensity no longer depends on the spin-lattice relaxation time (T_1), making centric scan SPRITE an ideal method for quantitative imaging of sedimentary rocks with short relaxation times (Chen, Halse and Balcom 2005). A 1D double half k-space SPRITE (DHK) technique, also called 1D centric scan SPRITE, is illustrated in Figure 2.

Previous work (Green et al. 2008 and Chen et al. 2005) investigated typical T_2^* values in reservoir rock and its dependence on saturation level. It was found that T_2^* is single exponential and insensitive enough to saturation level to allow for quantitative results. Other MRI methods have a T_2 dependence which is known to be multi-exponential and depend heavily on the saturation level. This makes quantitative analysis nearly impossible with these MRI methods (Baldwin and Spinler 1998).

MRI-based Capillary Pressure

Capillary pressure theory combined with MRI-determined saturation profiles allow us to directly 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 which then can be quantified using MRI. The capillary pressure at each position down the rock is known from equation (1). The saturation at the corresponding positions is measured using MRI. The radial distance is determined at each profile point knowing that one edge is the distance to the outlet face, r_1 . Saturation profiles acquired after centrifugation at different speeds are plotted on the same curve expanding the range and resolution of the capillary pressure curve.

Rotational speed(s) for the centrifuge can be estimated by using the Leverett J function (Leverett 1941)

$$P_c(r) = \frac{1}{2} \Delta\rho \omega^2 (r_2^2 - r_1^2) \geq \frac{J(S_{wi}) \sigma \cos \theta}{\sqrt{\frac{k}{\phi}}} \quad (5)$$

where J is the Leverett value, σ is the normal interfacial tension, θ is the contact angle, k is the permeability and ϕ is the porosity for a given rock. The J value “normalizes” the speed using this function (Brown 1951). We have found that for primary drainage two centrifuge speeds with a J factor of 0.5 and 4.0 provides the best coverage of the capillary pressure curve. For imbibition and secondary drainage a single centrifuge speed is sufficient and can be chosen more accurately based on the primary drainage curve that is usually acquired first.

For these experiments, the centrifugation step was carried out on either a high speed Beckman J2-21M centrifuge or a desktop centrifuge modified for centrifuging rock core plugs. The MRI experiments were carried out on a GIT (Green Imaging Technologies, Inc., Fredericton, NB, Canada) modified 0.18 Tesla (6.2MHz) Bruker LF90 MiniSpec (Bruker Optics, Houston, TX). All processing was performed by commercial software from GIT. The benefits of using a low-field MRI instrument are that the instrument is relatively inexpensive, and the effective spin-spin relaxation time (T_2^*) is long enough for imaging. Most NMR instrumentation capable of diffusion measurements (i.e. 1D gradient set) are capable of performing these measurements.

The T_2 distributions were measured with a CPMG echo train (Howard et al 1993) with an echo time of 0.2 msec. The number of echoes was chosen to ensure that the echo train was long enough to be 5 times the maximum T_2 value in the rock.

The fluid content profiles along the length of the core before and after centrifugation were determined by 1D DHK SPRITE MRI with a phase encoding time of 50 μ s, field of view of 7cm (typically), flip angle of 15 degrees and a resolution of 64 points. The number of signal averages was varied to determine the required signal to noise ratio (SNR) for acceptable results. In general, SNR values of 100 or 200 were used. 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 but will require more signal averages to achieve the same SNR.

The following expanded Brooks-Corey equation is used to fit the data:

$$P_C = p_{we} \left(\frac{S_w - S_{wi}}{1 - S_{wi}} \right)^{-\frac{1}{\lambda_w}} + p_{oe} \left(\frac{1 - S_w - S_{wi}}{1 - S_{or}} \right)^{-\frac{1}{\lambda_o}} \quad (6)$$

Where the “w” and “o” subscripts represent the entry pressure and lamda for the water and oil phase.

Air/Brine Capillary Pressure

For air/brine systems the fully saturated profile gives us the 100% saturation level. We know that the 0% saturation level (i.e. 100% air) will yield no MRI signal as there is no measurable hydrogen present. The saturation level is calculated by dividing the centrifuged profile by the 100% saturated profile. The MRI equipment can be calibrated such that the measured profiles directly determine saturation not requiring the 100% saturated profile assuming the rock is uniform. Figure 6 is an example of saturation distribution. The figure shows a series of centrifuged profiles and highlights the redistribution of fluids following centrifugation.

At the higher centrifuge speed most of the rock is at the irreducible water saturation. Therefore, it makes sense to create a protocol that not only performs a capillary pressure measurement but also a T_2 or T_1 relaxation measurement. This requires the addition of T_2 (or T_1) measurement when the rock is fully saturated and again when it is fully de-saturated which is usually the last centrifuge speed used for the capillary pressure measurement.

Air/Brine Combined Pc and T_2 Experimental Procedure

General experimental procedures for air/brine primary drainage capillary pressure curve and T_2 NMR measurements follow:

1. The core plugs were cleaned and dried.
2. Helium grain volume, pore volume, and permeability were measured.
3. The cores were vacuum saturated with 2% KCl brine. Porosity was calculated using the saturation method and compared to step 2 to ensure full saturation.
4. An MRI 1D DHK SPRITE MRI profile of the fully saturated (ambient conditions) core was measured. This was used later to determine water saturation after centrifugation.
5. A T_2 NMR measurement was made of the fully brine saturated rock.
6. Using the Leverett J function of equation (5), the rotational speed was determined for a variety of J values for each rock. To study the new method several J values were selected (0.5 and 3-4 were determined to be the best).
7. The core was placed in the centrifuge and spun for 24 hours at the first speed.
8. The core was removed from the centrifuge and placed in the NMR machine and a one dimensional profile on the centrifuged core using the same MRI sequence from step 4 was performed.
9. The capillary pressure curve for primary drainage was created using the method described above.
10. Repeat steps 7 through 9 for various centrifuge speeds.

11. At the final centrifuge speed a T_2 NMR measurement was made of the fully de-saturated rock plug.
12. All centrifuge speed data were combined into one capillary pressure curve and the results were modeled using a Brooks-Corey model.
13. A T_2 NMR distribution was produced and FFI, BVI, CBW and T_2 cutoff were calculated.

Oil/Brine Capillary Pressure

The simplest way to perform oil/brine capillary pressure measurements is to have one of the two fluids be NMR “invisible” by choosing one of the fluids such that it contain no hydrogen atoms. One solution is to use fluorinated oil and brine but the best solution is to use deuterium oxide, D_2O , brine and regular oil. Deuterium is an isotope of hydrogen and deuterium oxide has all the same chemical properties as water (although it does have a slightly higher density). The starting point for a deuterium-oxide/oil capillary pressure measurement is to have the rock fully saturated with the deuterium oxide brine which will have no NMR signal. To acquire the needed fully saturated measurement of the rock, a simple oil like decane can be used then evaporated out of the rock in an oven before saturating the rock with D_2O brine (or the saturated profile can be taken at the end of the test). When measuring the oil, the water saturation is calculated by subtracting the measured oil profile from the fully saturated profile (accounting for any hydrogen index differences).

Unlike traditional centrifuge techniques, both the negative and positive capillary pressures can be measured for the imbibition and secondary drainage curves by placing the oil/brine contact point half way up the rock. This moves the 0 psi point to the mid-point of the rock with negative and positive capillary pressures present in opposite ends of the rock.

Oil/Brine Experimental Procedure

1. The core plugs were cleaned and dried.
2. Helium grain volume, pore volume, and permeability were measured.
3. The cores were vacuum saturated with decane. Porosity was calculated using the saturation method and compared to given helium porosity to ensure full saturation.
4. A MRI 1D DHK SPRITE MRI profile measurement of fully saturated core was measured. This was used later to determine water saturation after centrifugation.
5. The core plugs were vacuum dried to remove the decane. The cores were vacuum saturated with 2% KCl deuterium brine (or formation brine).
6. Using the Leverett J function of equation (57), the rotational speed was determined for using Leverett J values of approximately 0.5 and 4 for each rock.
7. The core was placed in the centrifuge and spun for 24 hours (unless otherwise noted) at the first speed.
8. An MRI profile on the centrifuged core using the same MRI sequence from step 4 was performed and the capillary pressure curve for primary drainage was created using the method described above.
9. Repeat steps 6 through 7 for the other centrifuge speed(s).

10. The rocks were rotated and respun at the highest centrifuge speed to ensure the entire rock is at the irreducible water saturation.
11. The plugs were then spun at the last primary drainage centrifuge speed with the decane/brine boundary half way up the rock. A one dimensional saturation distribution was measured. A GIT-CAP primary imbibition P_c was determined.
12. The rocks were rotated and respun at the highest centrifuge speed to ensure the entire rock plug was at the irreducible oil saturation.
13. The plugs were then spun at the imbibition centrifuge speed with the Decane/brine boundary half way up the rock. A one dimensional saturation distribution was measured. A GIT-CAP secondary drainage P_c was determined.
14. All centrifuge speed data were combined into one capillary pressure curve and the results were modeled using a Brooks-Corey model.

Results and Discussion

Following the air/brine test protocol above, both a primary drainage air/brine capillary pressure curve, Figure 4, and a NMR T_2 cutoff, Figure 5, measurements were acquired. The two centrifuge speeds used correspond to a Leverett J value of 0.73 and 4.59. The curve shows excellent correlation with a traditionally acquired porous plate measurement. We know the distribution of fluid created in the centrifuge will eventually change after removal from the centrifuge. Figure 6 shows the water distribution in the rock as a function of time. Even in this high permeability rock the errors introduced by the redistribution of fluid are undetectable until 1 hour has passed. We have found from previous work that the redistribution of fluid is function of the permeability and is faster in higher permeability rocks. Figure 5 shows the T_2 distribution of the same rock fully saturated and fully de-saturated. The FFI, BVI and T_2 cutoff were found to be 23.3%, 4.54%, and 63ms. These T_2 distributions were acquired during the process of acquiring the primary drainage curve. The total time for acquiring both the primary drainage capillary pressure curve and the T_2 measurements was 3 days including sample preparation with 24 hour centrifuge equilibrium time.

Figure 7 shows oil/brine primary drainage, imbibition and secondary drainage curves for a sandstone. Kerosene was used as the oil and D_2O 2% KCL brine were used as the two fluids for this measurement. The imbibition and secondary drainage curves were acquired with only one fluid present in the centrifuge. When measured in this way, the positive capillary pressure for the imbibition and the negative pressures for the secondary drainage are not present. The USBM wettability is 0.28. The drainage and imbibition curves can be modeled by using equation (6) with water term zeroed for imbibition and the oil term zeroed for drainage. The fitted values are shown in the Figure.

Figure 8 and Figure 9 show the oil/brine primary drainage, imbibition and secondary drainage curves for 2 chalk samples. Decane and D_2O reservoir brine were used as the two fluids for these measurements. The oil/water contact point for the imbibition and secondary drainage curves was placed half way up the rock. This allows for the measurement of both the positive and negative pressures in the imbibition and secondary drainage curves. The fitted values from equation (6) are shown on the Figure. For these samples attempts were made to preserve the reservoir wettability and the rocks are shown

to be weakly water wet with an Amott-Harvey Index of 0.069 and 0.297 and a USBM wettability of 0.028 and 0.302, respectively

The MRI SNR will directly affect the accuracy of the capillary pressure measurement obtained. This study followed the results obtained from previous work and acquired the fully saturated profiles with an SNR of 200 and the centrifuged profiles with an SNR of 100. The total MRI scan times are listed on the Figures.

CONCLUSION

The new MRI-based capillary pressure measurement technique (GIT-CAP) has the capability to directly measure the fluids in the rock much more quickly and accurately than traditional techniques. The technique is 7 to 10 time faster for some Pc curves as only a single centrifuge equilibrium speed is required. The fluid saturations are directly measured in the rock eliminating the need for an approximate solution to the Hassler-Brunner equation. In addition, it is possible to acquire both the positive and negative portions of the imbibition and secondary drainage curve.

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, the University of New Brunswick for conducting the measurements, and Chevron, ConocoPhillips, Shell, and ExxonMobil for providing rock cores and comparison capillary pressure data.

REFERENCES

- Balcom B., MacGregor R., Beyea S., Green D., Armstrong R., Bremner T., "Single Point Ramped Imaging with T₁ Enhancement (SPRITE)", *J. Magn. Reson.* (1996), A123, 131.
- Baldwin B. and Spinler E., "A direct method for simultaneously determining positive and negative capillary pressure curves in reservoir rock, *J. Pet. Sci. Eng.* (1998), 20, 161.
- Brown H. W., "Capillary pressure investigations", *Trans. AIME*, (1951), **192**, 67.
- Chen Q. and Balcom B., "Capillary Pressure Curve Measurement Using a Single-Moderate-Speed Centrifuge and Magnetic Resonance Imaging", SCA2005-44, the Intl. Symp. of the SCA, Toronto, Canada, 2005
- Chen A., Hickey H. and Balcom B., "A Single-shot Method for Determining Drainage and Imbibition Capillary Pressure Curves", SCA Annual Conference, SCA2006-12, Trondheim, Norway, 2006
- Chen Q., Halse M. and Balcom B.J., "Centric scan SPRITE for spin density imaging of short relaxation time porous materials", *Magnetic Resonance Imaging*, (2005), 23, 263.
- Dullien F., *Porous Media: fluid transport and pore structure*, Academic Press, New York, (1991), 139-176.
- Green D.P., Dick J. R., Gardner J., Balcom B.J., and Zhou B. Comparison Study of Capillary Pressure Curves Obtained using Traditional Centrifuge and Magnetic Resonance

Imaging Techniques, SCA 2007-30, the Intl. Symp. of the SCA, Sept. 10-13, Calgary, Canada, 2007

Green D.P., Gardner J., Balcom B.J., McAloon M.J., and Cano-Barrita P.F. de J.. Comparison Study of Capillary Pressure Curves Obtained using Traditional Centrifuge and Magnetic Resonance Imaging Techniques, SPE 110518, SPE-IOR Conference, Apr. 19-23, Tulsa, OK, USA, 2008

Forbes P., "Centrifuge data analysis techniques, an SCA survey on the calculation of drainage capillary pressure curves from centrifuge measurements", SCA-9714, the Intl. Symp. of the SCA, Calgary, Sept. 8-10, (1997).

Hassler G. and Brunner E., "Measurement of capillary pressures in small core samples", Trans. AIME, (1945), 160, 114.

Howard, J.J., Kenyon, W.E., and Straley, C., "Proton Magnetic Resonance and Pore Size Variations in Reservoir Sandstones", SPE\$ Formation Evaluation, 194 (1993)

Masalmeh S., X. Jing, W. van Vark, S. Christiansen, J. van der Weerd and J. van Dorp, Impact of SCAL on carbonate reservoirs: how capillary forces can affect field performance predictions, SCA 2003-36, the Intl. Symp. of the SCA, Pau, France, Sept. 21-24, (2003).

Mastikhin I., Balcom B.J., Prado P. and Kennedy C., "SPRITE MRI with Prepared Magnetization and Centric k space Sampling", J. Magn. Reson. (1999), 136, 159.

Rajan R., "Theoretically Correct Analytical Solution for Calculating Capillary Pressure-Saturation from Centrifuge Experiments", paper J in SPWLA 27th Annual Logging Symposium Transactions, Houston, Texas, June 9-13 (1986).

Ruth D. and Chen Z., "On the measurement and interpretation of centrifuge capillary pressure curves: The SCA survey data", The Log Analyst, (1995), 36, 21.

US patent application number 11/262,658, "Methods and apparatus for measuring capillary pressure in a sample".

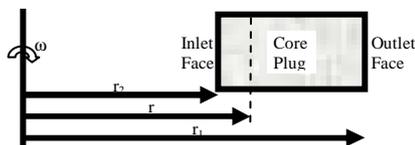


Figure 1 - Schematic of a rock core plug spinning in a centrifuge.

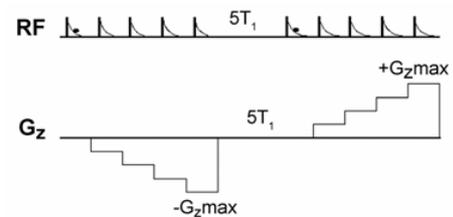


Figure 2 - The double half k-space version of the SPRITE MRI method.

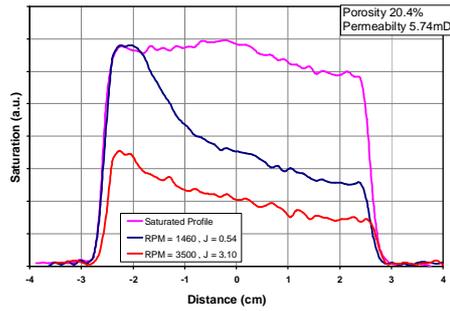


Figure 3 - MRI profiles of 1.5” diameter rock core plug during a air/Brine P_c measurement. The fully saturated profile is the relatively uniform profile showing the homogeneity of the rock. The remaining profiles are acquired after successively higher centrifuge speeds.

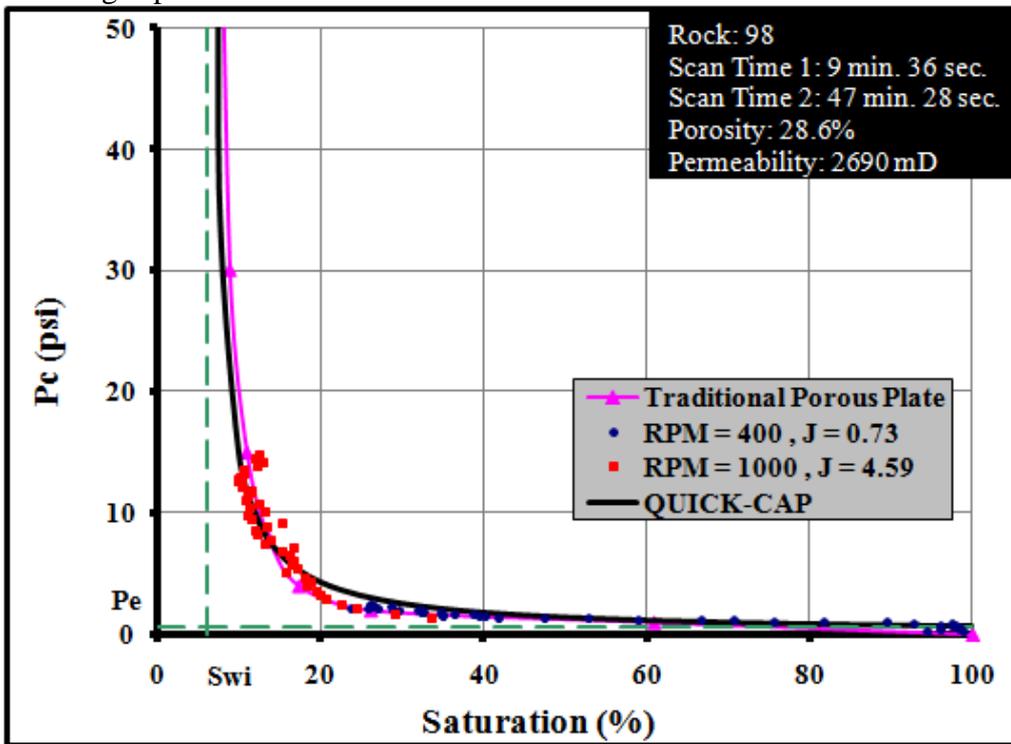


Figure 4 - Rock 98 Air/Brine Capillary pressure curve of a high permeability sandstone

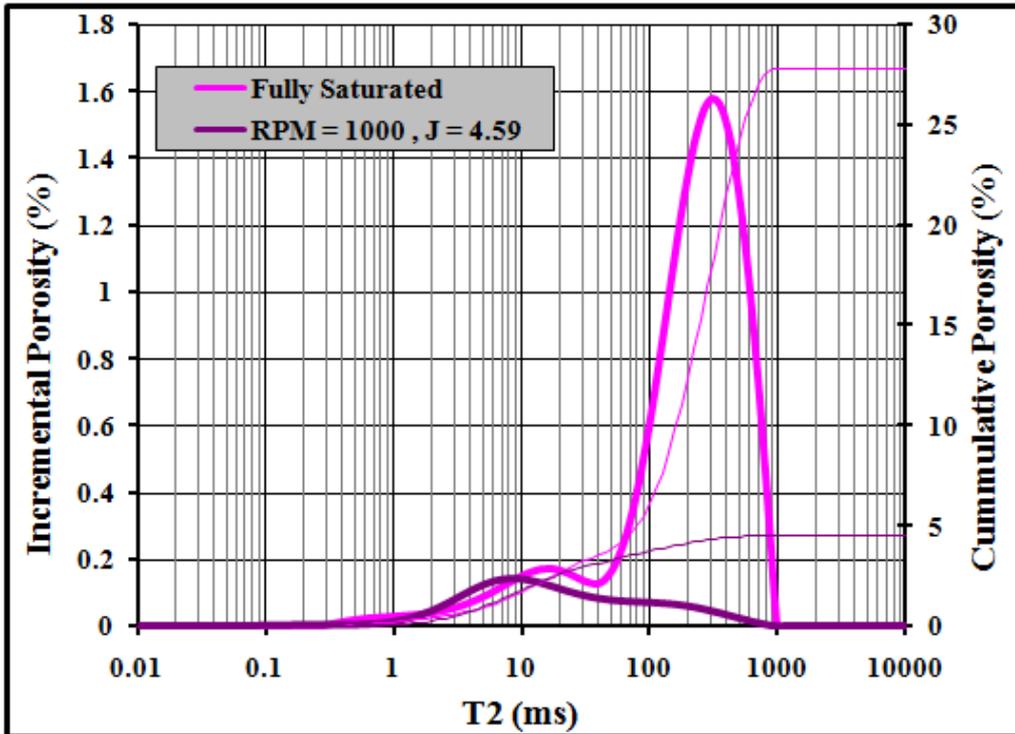


Figure 5 - Rock 98 T₂ cutoff measurement

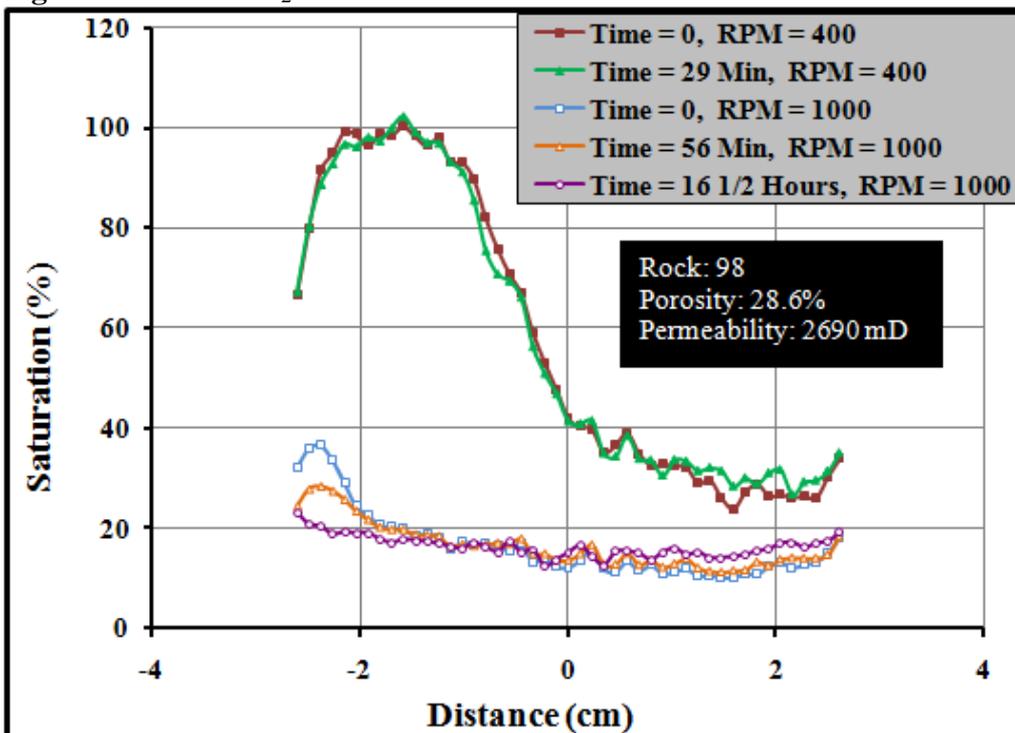


Figure 6 - Rock 98 redistribution of fluid following centrifugation

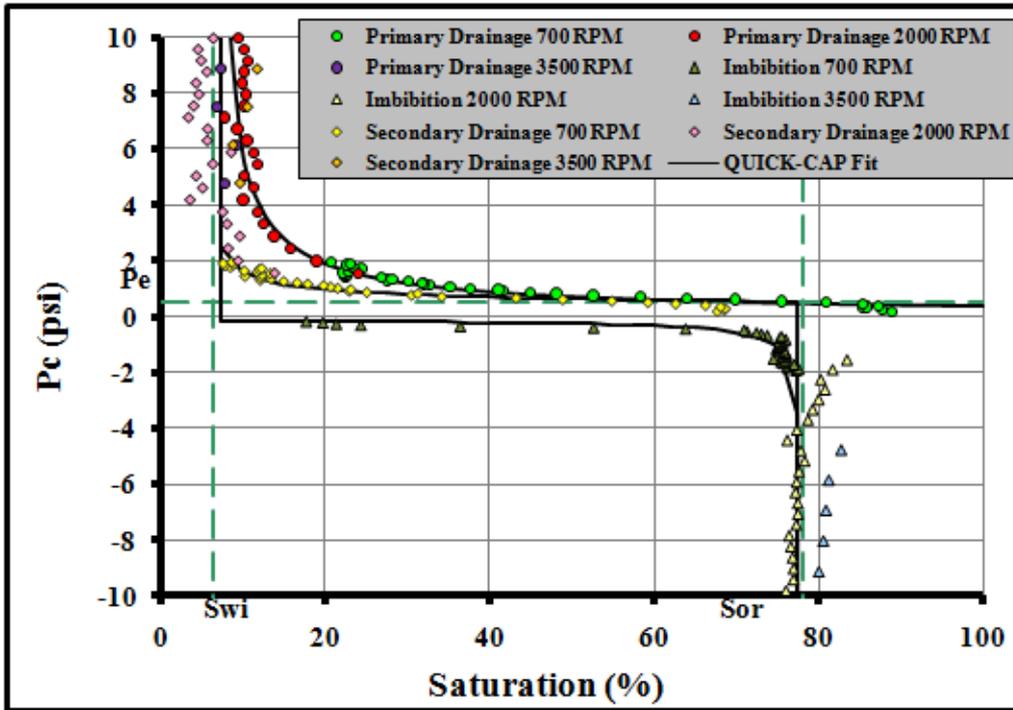


Figure 7 - Rock 42S oil/brine capillary pressure curves

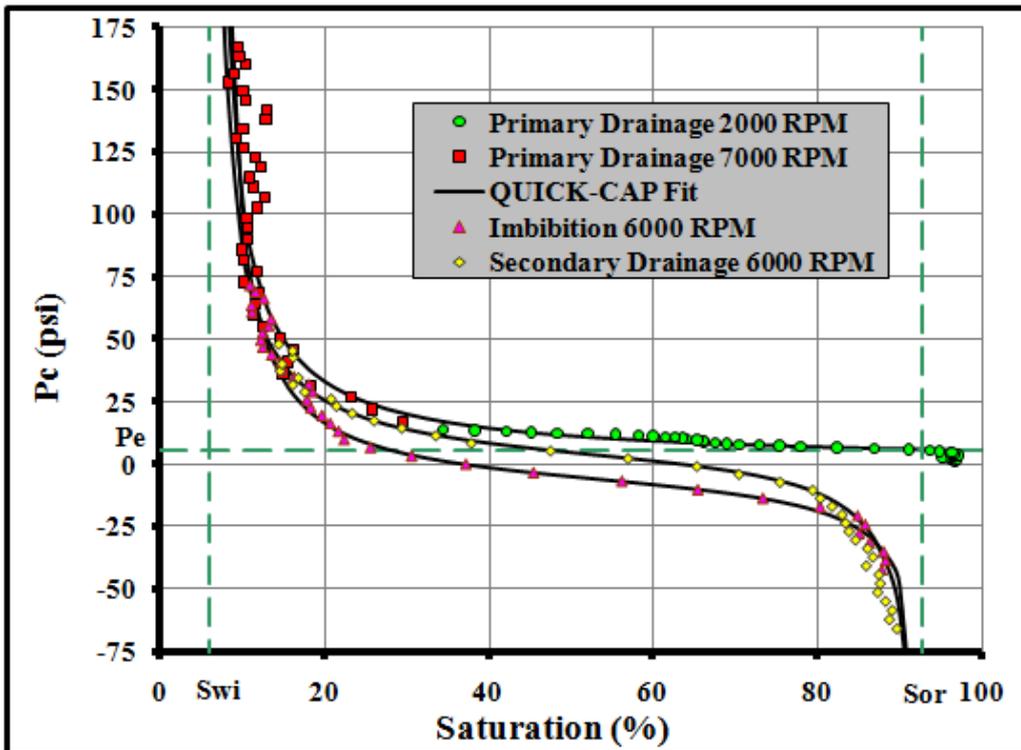


Figure 8 - Rock MT1 oil/brine capillary pressure curves

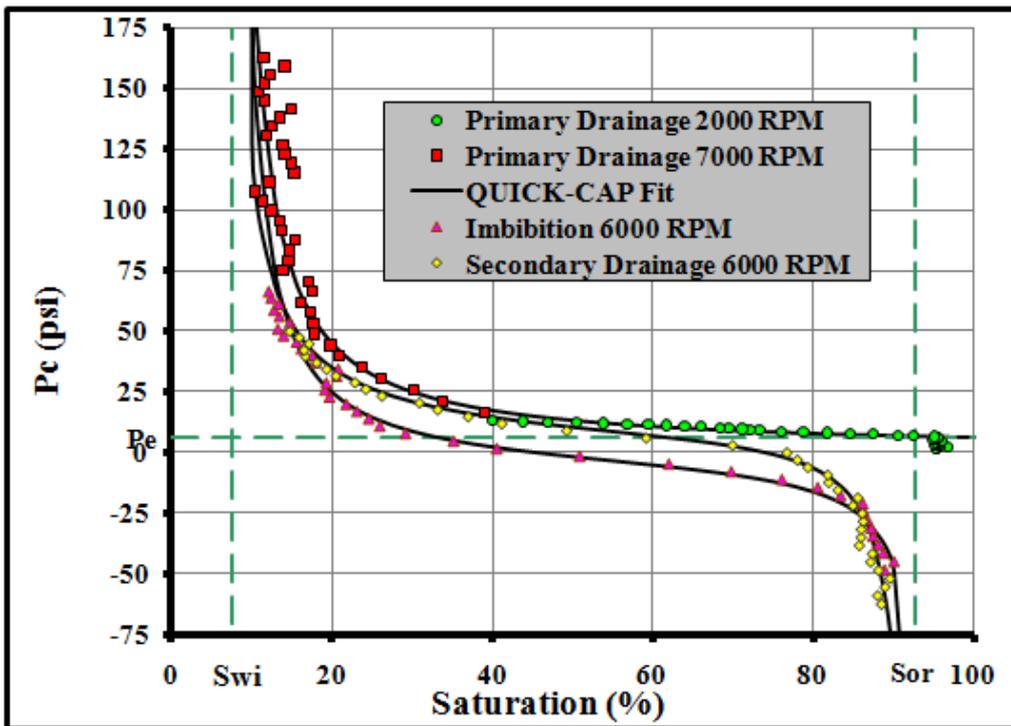


Figure 9 - Rock MT2 oil/brine capillary pressure curves