

CAPILLARY PRESSURE SCANNING CURVES BY DIRECT MEASUREMENT OF SATURATION

by
E. A. Spinler and B. A. Baldwin
of Phillips Petroleum Co.

Abstract

There is relatively little literature reporting of water/oil capillary pressure data that include development of complete primary drainage, imbibition and secondary drainage curves. Major reasons for the lack of such data are the difficulty, expense and time to obtain them. Direct measurement of saturation profiles in plugs can significantly reduce these negatives. Saturation profiles for water/oil capillary pressures were established with the centrifuge and then locked in place by freezing the oil phase while still in the centrifuge. Magnetic resonance imaging was used to map the saturation profiles, obtaining over a hundred measurements per profile. Both positive and negative portions of the capillary pressure curve were obtained simultaneously by positioning the free water level along the length of the plug. Drainage or imbibition was achieved by centrifuging the plug under hydrocarbon or water by starting with the appropriate saturation state. The capillary pressure range was controlled by the speed of the centrifuge, the height above the free water level and the sample length. Comparisons of repeated measurements assured that an equilibrium saturation state was achieved. Measurements of bounding drainage and imbibition curves were made for two diverse rock types, Berea sandstone and reservoir chalk.

Introduction

Capillary pressure is a measure of the interaction between fluids and the rock pore surface. At given capillary pressure, the prior fluid saturation history and the direction of saturation change can significantly affect the saturation state in a porous rock at a given capillary pressure. Consequently, primary depletion methods and secondary or tertiary recovery methods applied to a reservoir have to consider the impact of prior and subsequent field operations on hydrocarbon recovery. Knowledge of the capillary pressure hysteresis, also called scanning curves, for a reservoir rock can be obtained in the laboratory. However, in practice this has been difficult, of uncertain accuracy, and/or very time consuming to experimentally determine.

Capillary pressure curves are typically determined by either mercury intrusion, porous plate/membrane or centrifuge methods. Mercury intrusion, although rapid and popular^{1,2}, provides questionable results due to the use of mercury in a vacuum to mimic water/oil behavior. It's methodology limits the technique to primary drainage and positive imbibition capillary pressures. Mercury derived scanning curves are therefore constrained to only highly water-wet behavior at positive capillary pressures. Porous plate/membrane methods can

generate all the capillary pressure hysteresis curves, but to reach an apparent equilibrium saturation can take days to months per pressure point for 5 to 8 data points per curve. This delays the availability of the results and limits the number of tests that can be made. However, Hammervold³ recently demonstrated a continuous change method using porous membranes to reduce the time to obtain quasi-equilibrium scanning loops. The centrifuge method is normally only used to determine drainage or negative imbibition curves and as a consequence scanning curves are not obtained. Torsaeter, et al^{4,5}, however, modified both the plug and the centrifuge to obtain positive imbibition. Use of the centrifuge is generally more rapid than porous plate/membrane methods, but since it measures a single point at a time, it also takes days to months to complete a test. A major limitation of typical centrifuge methods is that they can only provide an indirect, or assumed/calculated, measure of saturation at the inlet face of a rock plug based on the amount of fluid expelled from the rock. Numerous methods over the past 50 years⁶ have been proposed for approximating the inlet saturation from centrifuge effluent volumes, but in every case, the model can influence the resultant capillary pressure curves.

In 1996 and 1997, the authors introduced the methodology for direct measurement of capillary pressure curves using the centrifuge^{7,8}. Others^{9,10,11} have employed, to a limited extent, some of the techniques conceived in this methodology, but none have incorporated all of the features as described herein. The promise behind this direct measurement method is that it can reduce or eliminate many of the major limitations of other methods such as the length of time to obtain data, the proper shape to capillary pressure curves, questions on the impact of wettability/contact angle, saturation equilibrium and boundary conditions. Although all potential advantages will not be addressed in this paper, it is the authors intent to demonstrate that complete curves of positive and negative capillary pressures can be obtained for both drainage and imbibition allowing for the determination of bounding scanning curves. The method appears applicable to diverse rock types and furthermore, appears to be accurate, rapid and robust.

Equipment and Methodology

Equipment

For the work reported herein, saturation measurements were determined from Magnetic Resonance Imaging (MRI). Images were obtained with a Varian 85/310 CSI. It has a 31 cm bore, 2T superconducting magnet and operates at 85.55 Mhz for hydrogen protons. A 9 cm I.D. saddle coil was used as both transmitting and receiving coil. The amount of fluid hydrogen protons was obtained using the Hahn spin-echo sequence with a 4 ms echo time and a 2.0 second recovery time. One slice, approximately 4 mm thick, was obtained through the center axis of the plug. This orientation produces a rectangle which shows the saturation gradient aligned parallel to the centrifugal axis. The field of view was 9 cm x 8 cm with a pixel resolution of 0.35 x 0.62 mm. A total of 16 repetitive measurements averaged at each data point. The images consist of 255 levels of gray in a 256 x 256 display. The intensity of the individual pictures were adjusted to produce a presentable visual display, however, for quantitative determinations absolute intensities were used. The image spatial intensities were transferred to a spreadsheet where the saturations were calculated and capillary pressure curves developed.

Two different centrifuges were used to prepare the sample plugs for scanning curves, a Beckman L5-50p using a horizontal head and a Sorvall RC 26 plus with a swinging bucket. Both centrifuges had heating and cooling capability. Another centrifuge, a Sorvall ultracentrifuge OTD65B with an TI 45 angle head was used to obtain calibration data.

A large diameter porous disk was also used to obtain calibration data.

The fluids used were de-ionized water and octadecane, Aldrich 99% purity.

The selected porous rocks were a Berea sandstone plug and a extracted reservoir chalk plug. The Berea was selected because there are numerous capillary pressure studies cited in the literature for comparison. The chalk represents a vastly different porous rock to demonstrate the applicability of the technique to various rock types. The properties of these plugs are reported in Table 1. Both plugs were believed to be water-wet.

Methodology

Briefly, the method directly measures an oil/water saturation profile within a porous rock that is established under a known pressure gradient as measured from a free-water level established along the height of the rock plug (see Figure 1). A capillary pressure curve develops in porous rock as the fluid saturation changes with the varying pressure induced by the centrifuge and density difference between hydrocarbon and water. The difficulty has been in determining saturation information in the rock while it is centrifuging. Although some have tried to obtain the saturation profile in the porous plug while centrifuging¹², it is expensive and cumbersome to do.

To obtain the water saturation profile that exists while centrifuging, the authors preferred procedure was to freeze the oil phase while centrifuging and map the water in the rock via magnetic resonance imaging (MRI) outside the centrifuge. Other imaging methods are also applicable, but MRI was convenient and had high spatial resolution. Freezing the hydrocarbon prevented redistribution of the water in the rock. In a solid state, the relaxation time of the hydrocarbon was so short that it was not imaged. Thus, in this system the water was imaged and octadecane which froze at 27 degrees Celsius was not. Laboratory ambient temperature was typically about 23 degrees Celsius, so there was little risk of melting and fluid movement outside of the centrifuge. The plug was kept in a sealed plastic centrifuge bottle during handling and imaging to prevent evaporation of the water. The intensity map of water distribution was directly converted to water saturation via calibration curves. The small volume change (approximately 2%) created by the hydrocarbon contraction during freezing was corrected by adjusting the water saturation.

Calibration curves were measured for air-water images of the core plugs at varying water saturation states. Average MRI intensity was cross-plotted with average water saturation obtained by weight measurements (see Figures 2 and 3), once a relatively uniform saturation state in the plugs was obtained. The Berea plug was also desaturated by porous plate. The chalk plug was desaturated by centrifuge. Imbibition of the chalk plug provided another calibration point. The more rapid development of a calibration curve using the

centrifuge followed by imbibition was favored for future work. The broad porosity distribution of the Berea plug may be reflected in the logarithmic character of the MRI calibration curve. A near linear MRI calibration curve was obtained for the chalk.

Capillary pressure as a function of position in the sample and the achievable pressure range was determined by the speed of the centrifuge, distance from the free water level and the sample length. Positioning the free water level along the length of the plug enabled development of both the positive and negative portions of a capillary pressure scanning curve simultaneously in the sample rocks. The free water level was imaged with the plug which enabled an accurate calculation of capillary pressures in the plug without the questions on boundary conditions common to other centrifuge methods. The imaging also enabled non-uniform boundary conditions such as radial artifacts to be visually assessed and avoided or corrected. Movement of the free water level was minimized during imbibition and drainage by using a centrifuge cell with a bulk volume significantly larger than the pore volume of the plug.

The preparation of the plugs and the sequence of the centrifuge steps was important to control the direction of fluid flow and observe possible hysteresis effects. The sides of the plugs were sealed with a Teflon sleeve to further constrain the direction of flow to the axial direction. Only the end faces of the plugs were available for fluid to exit or enter. During centrifuging, the plugs were kept at all times immersed under water and/or octadecane.

The following generalized chronological sequence was used to produce complete capillary pressure scanning curves:

- 1) Prepare the plug to 100% saturation of the wetting fluid.
- 2) Centrifuge with a free water level contacting the plug to produce the primary drainage curve.
- 3) Invert and centrifuge the plug from Step 1 in the non-wetting fluid. Repeat and invert plug again to reach a relatively uniform saturation distribution throughout the plug at an initial water saturation (S_{wi}) to prepare the plug for imbibition.
- 4) Centrifuge the plug from Step 4 with a free water level in contact with the plug. The free water level should be adjusted to achieve the range of positive and negative curves desired for producing a primary imbibition curve.
- 5) Invert and centrifuge the plug in the wetting fluid. Invert the plug and repeat to reach a relatively uniform saturation distribution throughout the plug at residual oil saturation (S_{or}) to prepare the plug for secondary drainage.
- 6) Centrifuge the plug from Step 5 with a free water level contacting the plug. The free water level should be adjusted to achieve the range of positive and negative curves desired for producing the secondary drainage curve.
- 7) Steps 3 to 6 could be repeated to evaluate additional hysteresis of the capillary pressure curves.

The experimental work with each plug was done separately in different centrifuges. Images of the plugs were taken at 1 day intervals for the Berea and 3 day intervals for the chalk during the steps listed above. The plugs were not changed to the next step until the image intensity profile measured for each plug was observed to be stabilized.

Results

Images of the 100 percent water saturated plugs and a corresponding intensity map for each plug is shown in Figures 4 through 7. The intensity map was constructed from the image area bound by the dashed rectangle for all images and represented the average intensity along the length of the plug. There were 133 averaged measurements for the Berea plug and 105 averaged measurements for the chalk plug. The larger variation in the map for the Berea reflects the less uniform porosity distribution found in the sandstone as compared to the chalk and not a higher noise level. Since the image represented only a 4 millimeter slice through each plug, averaging or imaging more of the plug by MRI can further smooth the capillary pressure curves.

An image of the Berea plug with a primary drainage saturation distribution is shown in figure 8. The image includes a reference standard to its left and is imaged with free water which is the bright area near the bottom of the plug. An arrow indicates a line where the free water level was located along the plug. Dark areas represent either solid octadecane or the sample plug holder. Although, this plug was desaturated using a swinging bucket at 1500 rpm, the free water level was not vertical, probably due to gravity. The impact of this sloping free water level was considered in the calculation of capillary pressures and would play an increasing role at lower centrifuge speeds. Since the image was obtained as a 4 mm slice parallel to the centrifuge axis, radial effects can be neglected.

The primary drainage capillary pressure data corresponding to figure 8 are shown in figure 9. Some scatter in the data results from porosity variations in the plug, but a capillary pressure curve could be easily drawn through the data over the 4 pound pressure range. The threshold pressure at approximately one half of a pound and the shape of the curve is typical of Berea plugs of similar properties reported in the literature^{13,14}.

The imbibition capillary pressure data for the Berea plug are shown in figure 10. Positive and negative data were obtained by placing the free water level near the center of the plug. The data crosses the free water level ($P_c = 0$) at S_w equal to 62 percent and form the basis for a bounding capillary pressure curve.

The secondary drainage capillary pressure data for the Berea plug are shown in figure 11. The free water level was placed near the outlet end of the plug anticipating that the data would cross the free water level at the same saturation as for imbibition since Berea is highly water-wet. However, the crossing point for secondary drainage was at S_w equal to 69 percent suggesting that the wettability was slightly less water-wet. The secondary drainage data form the basis for the upper bounding capillary pressure curve.

An image of the chalk plug with a primary drainage saturation distribution is shown in figure 12. The free water was removed prior to imaging and only residual water remains

on the boundary. An arrow indicates a line where the free water level was along the plug. This plug was desaturated using a horizontal centrifuge head at 9000 rpm. The free water level was close to vertical and no adjustments were made for gravity. The tilt to the image merely reflects that the plug was not parallel to the axis of the MRI when imaged. Since the image was obtained as a 4 mm slice parallel to the centrifuge axis, radial effects were neglected.

The primary drainage capillary pressure data corresponding to figure 12 are shown in figure 13. There is little scatter in the data since the porosity variations in the plug were small. A capillary pressure curve could be easily drawn through the data over the 100 pound pressure range. The threshold pressure at approximately 22 pounds and the shape of the curve is similar to chalk plugs of similar properties reported in the literature¹⁵.

The imbibition capillary pressure data for the chalk plug are shown in figure 14. Positive and negative data were obtained by placing the free water level along the side of the plug. The data cross the free water level ($P_c = 0$) at S_w equal to 72 percent and forms the basis for a bounding scanning capillary pressure curve.

The secondary drainage capillary pressure data for the chalk plug are shown in figure 15. The crossing point for secondary drainage was at S_w equal to 72 percent confirming that the plug was highly water-wet. The secondary drainage data form the basis for the upper bounding capillary pressure curve.

Overlays of the scanning curves are shown in figures 16 and 17. The Berea data have been smoothed and it is apparent that closure to bounding curves was not obtained. By placing the free water level to obtain more of the negative portion of the P_c curve, it should be possible to achieve closure. There was no smoothing to the chalk data and it can be seen that the imbibition and secondary drainage curves overlay. The more uniform porosity distribution in chalk and a high degree of connectivity between pores (high coordination number) may account for the lack of hysteresis between the imbibition and secondary drainage curves for the chalk.

The time needed to complete each step of the above test depended on how rapidly the fluids reached their equilibrium saturation in the centrifuge. This was largely determined by the permeability of the rock. This was especially true for low permeability chalk which had to be centrifuged up to 12 days to obtain and verify a single stabilized saturation profile compared to 2 days for the Berea sandstone. Once a calibration curve is available, the total experimental time to generate a set of equilibrium primary drainage, primary imbibition and secondary capillary pressure curves would take approximately 16 days for a Berea sandstone plug and approximately 1.5 months for a low permeability chalk plug. However, with a centrifuge head capable of holding more than one plug, the average time for 3 to 4 plugs could be similar to that for one plug.

Conclusions

1. Direct measurement of the saturation distribution in centrifuged rock plugs by MRI or other means eliminates the need for assumed or indirect determination of fluid saturations.
2. Placement of the free water level along the length of rock plugs produces simultaneous development of both the positive and negative capillary pressures for drainage and imbibition.
3. Freezing of the oil phase during centrifuging permits handling and determination of the water saturation in rock plugs without fluid redistribution.
4. The use of a centrifuge for pressure development coupled with direct measurement of the resulting saturation distribution appears to provide a rapid, accurate and robust laboratory method for determining capillary pressure scanning curves in porous media.

Acknowledgment

The authors thank Phillips Petroleum Co. for permission to publish this extended abstract and thank D. M. Chancellor for his mechanical assistance in building equipment for this work, J. C. Stevens for performing the centrifuging and R. L. King for assistance with MRI

References Cited

1. Wardlaw, N. C. and Taylor, R. P., "Mercury Capillary Pressure Curves and the Interpretation of Pore Structure and Capillary Behavior in Reservoir Rocks," *Bulletin of Canadian Petroleum Geology*, (1976) vol. 24, no. 2, pp. 225-262.
2. Purcell, W. R., "Capillary Pressures - their Measurement using Mercury and the Calculation of Permeability therefrom," *Petroleum Transactions, AIME* (Febr. 1949) pp. 39-48.
3. Hammervold, W. L., Knutsen, O., Iversen, J. E., and Skjaeveland, S. M., "Capillary Pressure Scanning Curves by the Micropore Membrane Technique," *The 4th International Symposium on Evaluation of Reservoir Wettability and its Effect on Oil Recovery proceedings* (1996)
4. Torsaeter, O., Determination of Positive Imbibition Capillary Pressure Curves by Centrifuging," *The 3rd International Symposium on Evaluation of Reservoir Wettability and its Effect on Oil Recovery proceedings* (1994)
5. Bolas, T. and Torsaeter, O., "Theoretical and Experimental Study of the Positive Imbibition Capillary Pressure Curves Obtained from Centrifuge Data," *1995 International Symposium of the Society of Core Analysts proceedings* (1995)
6. Ruth, D. and Chen, Z., " Measurement and Interpretation of Centrifuge Capillary Pressure Curves - The SCA survey Data," *The Log Analyst*, (Sept.-Oct 1995) pp. 21-33
7. Baldwin, B. A. and Spinler, E. A., "A Direct Method for Simultaneously Determining Positive and Negative Capillary Pressure Curves in Reservoir Rock," *4th International Symposium on Evaluation of Reservoir Wettability and Its Effect on Oil Recovery proceedings*. (1996)

8. Spinler, E. A. and B. A. Baldwin, "A Direct Method for Determining Complete Positive and Negative Capillary Pressure Curves for Reservoir Rock using the Centrifuge," *4th International Reservoir Characterization Technical Conference proceedings*. (1997)
9. Baldwin, B. A. and Yananashi, W. S., "Capillary-Pressure Determinations from NMR Images of Centrifuged Core Plugs: Berea Sandstone," *The Log Analyst* (Sept.-Oct.1991) pp. 550-556.
10. Baardsen, H., Nilsen, V., Leknes, J., and Hove, A., "Quantifying Saturation Distribution and Capillary Pressures using Centrifuge and Computer Tomography," *Reservoir Characterization II NIPER/DOE proceedings*. (1989)
11. O'Meara, D. J. Jr., Hirasaki, G. J. and Rohan, J. A., "Centrifuge Measurements of Capillary Pressure: Part 1 - Outflow Boundary Condition," *SPE Reservoir Engineering* (Feb.1992) pp. 132-142
12. Chardaire-Riviere, C., Forbes, P., Zhang, J. F., Chavent, G., and Lenormand, R., "Improving the Centrifuge Technique by Measuring Local Saturations," *SPE 24882* (1992)
13. Dullien, Lai, and Mac Donald, Jan., "Hydraulic Continuity of Residual Wetting Phase in Porous Media," *Journal of Colloid and Interface Science* (1996) vol 109, no.1
14. Hermansen, H., Eliassen, O., Guo, Y., and Skjæveland, S. M., "Capillary Pressure from Centrifuge - a New, Direct Method: Advances in Core Evaluation II," *EuroCAS*. (1991)
15. Norgaard, J. V. , Olsen, D., Springer, N., and Reffstrup, J., "Capillary Pressure Curves for Low Permeability Chalk by NMR Imaging of Core Saturation Profiles," *SPE 30605* (1995)

Table 1 - Petrophysical Properties

Plug #	Type	Length (cm)	Diameter (cm)	Porosity (fraction)	Permeability (md)
bb	berea	4.78	2.52	0.20	612
1052b	chalk	3.94	2.54	0.33	~ 5

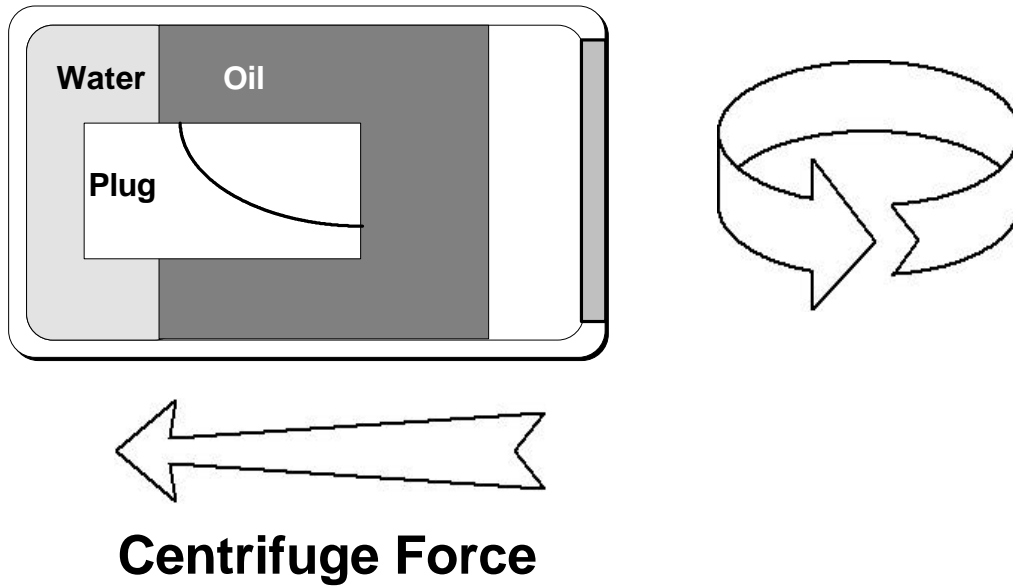


Figure 1. Simplified drawing of plug with free water level in a large bulk volume centrifuge cell with a saturation profile.

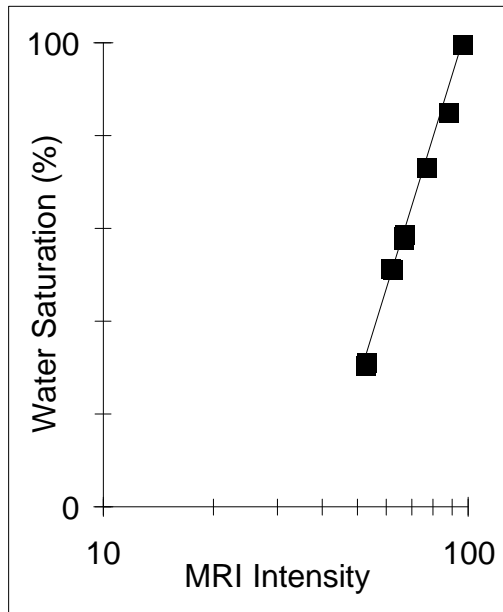


Figure 2. Berea calibration curve from air-water porous plate desaturation.

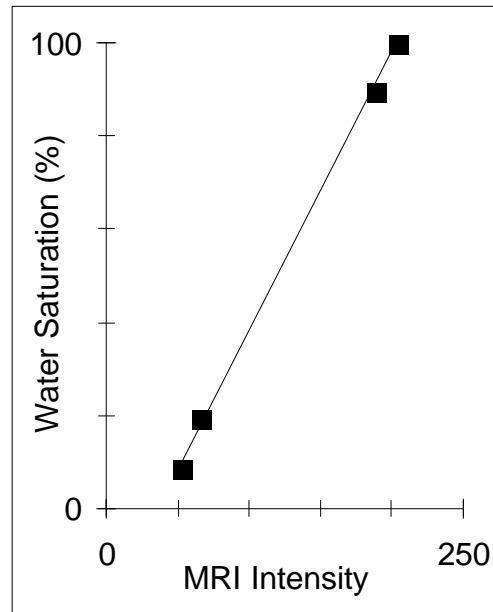


Figure 3. Chalk calibration curve from air-water centrifuge desaturation and imbibition.

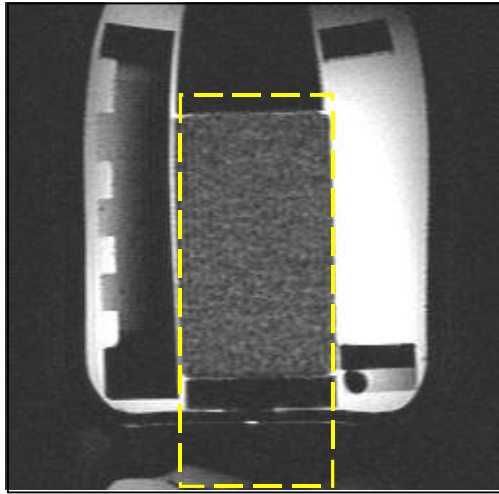


Figure 4. MRI image of Berea plug at 100% water saturation. Dashed rectangle encloses image area.

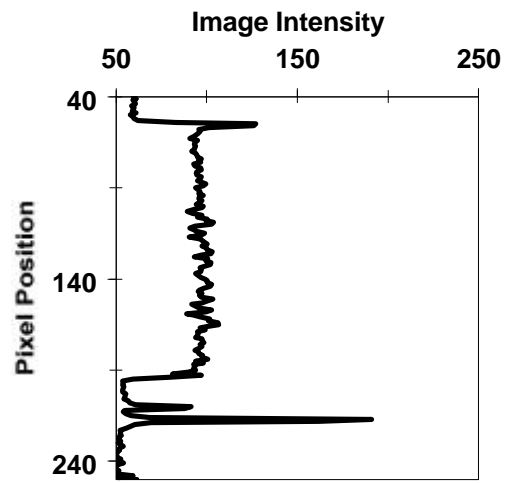


Figure 5. Image intensity map for the boxed area of Figure 4.

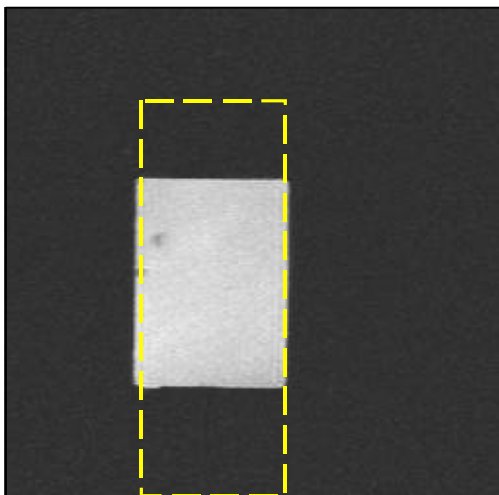


Figure 6. MRI image of chalk plug at 100% water saturation. Dashed rectangle encloses image area.

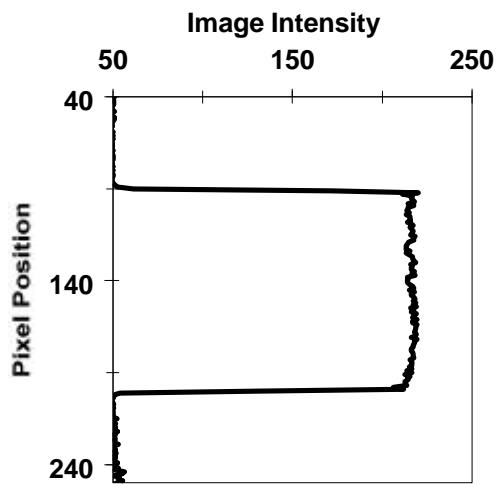


Figure 7. Image intensity map for the boxed area of Figure 6.

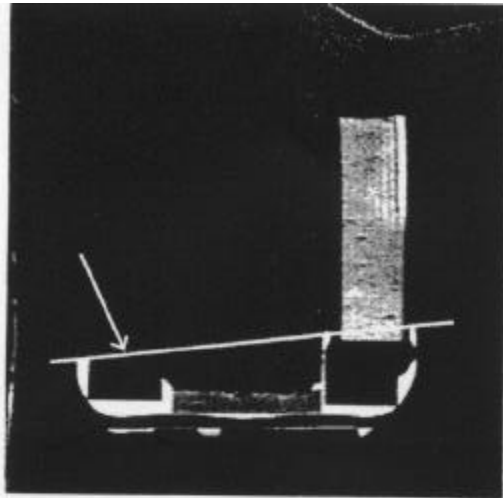


Figure 8. MRI image of Berea plug at primary drainage. Arrow indicates marked free water level.

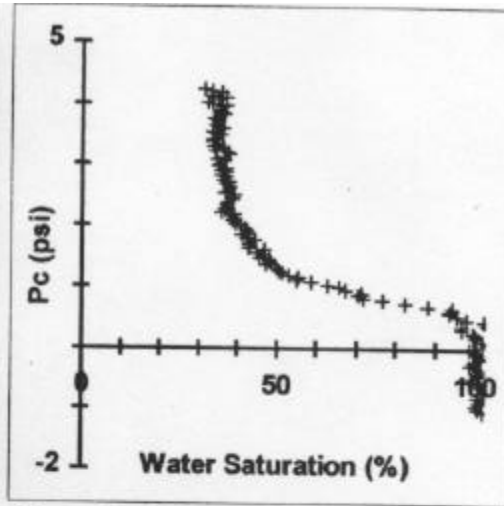


Figure 9. Primary drainage Pc for Berea plug from MRI in Figure 8.

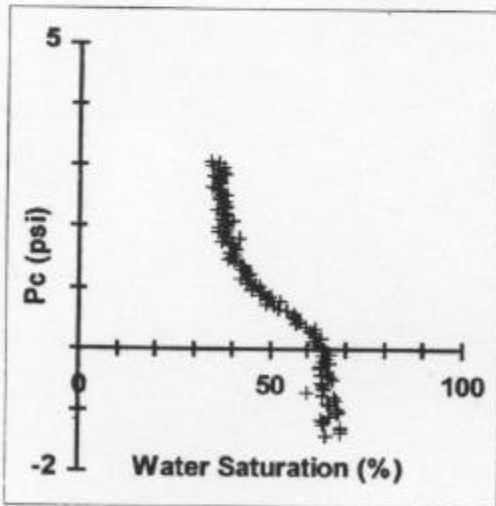


Figure 10. Imbibition Pc for Berea plug.

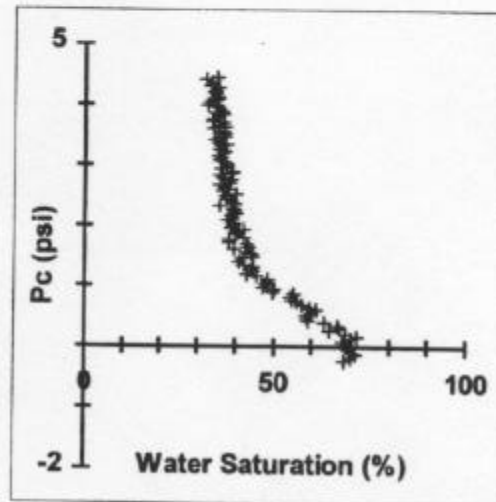


Figure 11. Secondary drainage Pc for Berea plug.

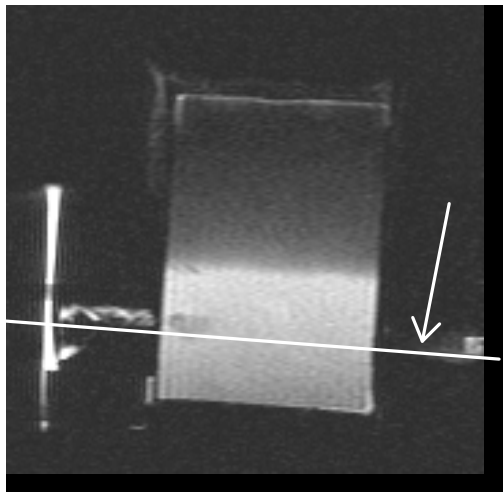


Figure 12. MRI image of chalk plug at primary drainage. Arrow indicates marked free water level.

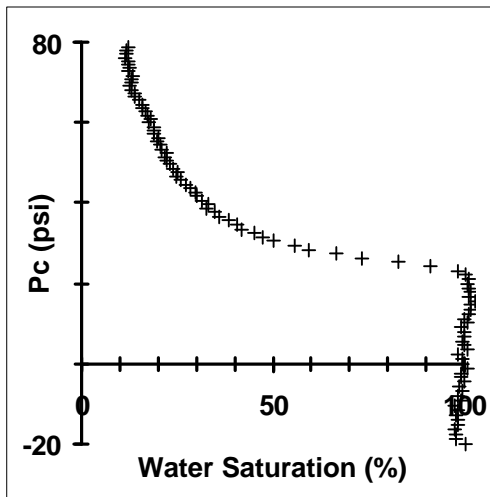


Figure 13. Primary drainage Pc for chalk plug from MRI in Figure 12.

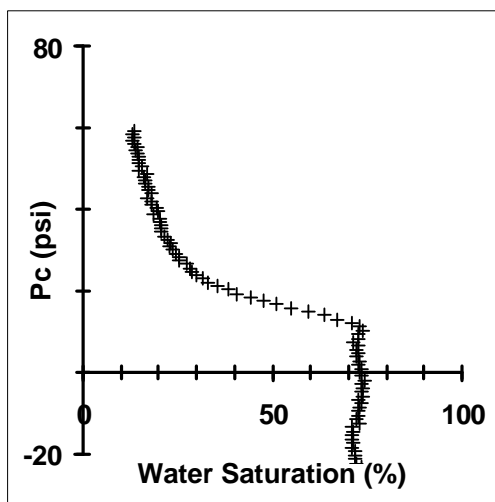


Figure 14. Imbibition Pc for chalk plug.

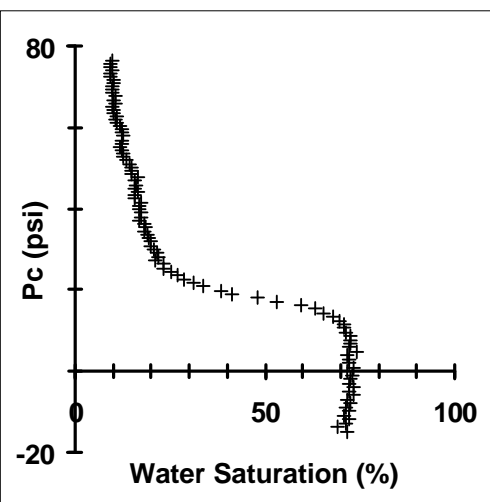


Figure 15. Secondary drainage Pc for chalk plug.

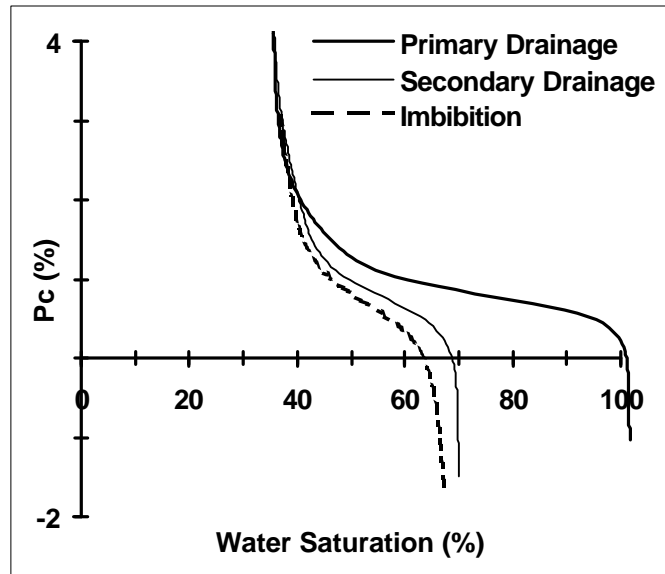


Figure 16. Smooth capillary pressure curves for Berea plug showing details of the bounding curves.

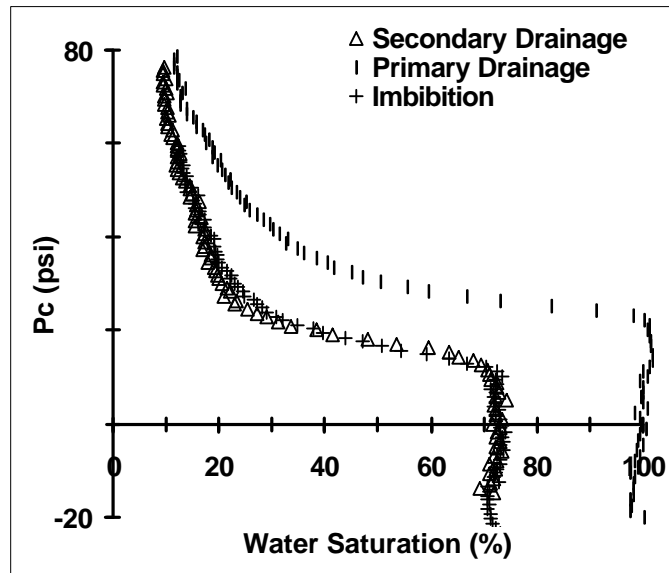


Figure 17. Capillary pressure curves for chalk plug showing no hysteresis of the bounding curves.