CALILLARY PRESSURE CURVE MEASUREMENT USING A SINGLE-MODERATE-SPEED CENTRIFUGE AND QUANTITATIVE MAGNETIC RESONANCE IMAGING

Quan Chen and Bruce J. Balcom

MRI Centre, Department of Physics, University of New Brunswick, P. O. Box 4400, Fredericton, NB, E3B 5A3, Canada

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ABSTRACT

For traditional centrifugal capillary pressure curve measurements, a short core plug is often employed. The capillary pressure is assumed to be zero at the outflow boundary of the core. The traditional experiments are time consuming. Measurement of the full capillary pressure curve often requires 7 to10 different centrifuge speeds, thus requiring one day to several days for measurement. Different approximate solutions may be employed to deduce the capillary pressure curve from experimental data of average saturation and the corresponding rotational speed.

In the current work, we propose a new method to measure the capillary pressure curve using a single-moderate-speed centrifuge experiment and one dimensional centric scan SPRITE (Single-Point Ramped Imaging with T_1 Enhancement) magnetic resonance imaging (MRI) technique for long rock samples. As a pure phase encoding quantitative MRI technique, centric scan SPRITE offers unique advantages in the measurement of spatially resolved fluid saturation in rocks.

The proposed method is rapid and accurate. Because the new method uses a long core to directly measure a large range of saturation distributions along the length of the core, at a single moderate centrifuge speed, this process will be much faster than a traditional measurement. There is no requirement for an approximate solution; the capillary pressure curve is directly determined by the measured saturation profile. Long cores can be employed, which leads to a smaller radial effect than short core plugs. Since a moderate centrifuge speed is employed, the effect of gravity will be very small and the outlet boundary condition of the core plug can be satisfied. A comparison of capillary pressure curves obtained by a single-speed centrifuge and SPRITE MRI with mercury intrusion methods yields remarkably consistent results.

INTRODUCTION

Capillary pressure results from the interaction between a wetting fluid, and a non-wetting fluid, as well as their bounding solid matrix. Capillary pressure critically influences the initial reservoir fluid distribution and dynamic processes of oil recovery. Capillary pressure is the most fundamental rock-fluid characteristic in multi-phase flows, just as porosity and permeability are the most fundamental properties in single-phase flow in porous media [1].

In evaluating hydrocarbon reservoirs, laboratory capillary pressure curve measurements on reservoir cores are directly applied to determine many basic petrophysical properties, for example: pore size distribution, irreducible water saturation, residual oil saturation, and wettability of reservoir rocks. In addition, they are used to determine the initial water and oil saturation as a function of height above the free water level, an approximate oil recovery efficiency, and to calculate the relative permeability [2-4]. Capillary pressure can also have a significant impact on water flood performance [5].

In the laboratory, the capillary pressure curve can be determined with either mercury intrusion porosimetry, porous diaphragm, or centrifuge methods, based on hydrostatic equilibrium [6]. The porous diaphragm method is a direct and accurate technique, but the measurement is extremely time-consuming, since the equilibration time can be weeks or months for each individual pressure point. The mercury intrusion method is rapid, but destructive. In addition, the mercury intrusion measurement does not provide information on reservoir wettability, and mercury is a health hazard.

The centrifuge method for capillary pressure curve measurement was first introduced by Hassler and Brunner [7] in 1945. This method involves rotating fluid bearing rock cores at variable speeds in a specially modified centrifuge. Sample rotation yields a centrifugal force which will counterbalance the capillary pressure when hydrostatic equilibrium is reached. Collecting and measuring the expelled fluid as a function of increasing the rotational speed permits a quantification of the capillary pressure as a function of fluid saturation [8-10]. This technique has been extensively investigated, and is commonly used in the petroleum industry.

A full capillary pressure curve determined with traditional centrifuge methods requires 7 to 10 different centrifuge speeds, thus requiring one day to several days for measurement. The experiment requires a very expensive ultracentrifuge with precise speed control over a wide range of speeds.

In the current work, we propose a method to measure the capillary pressure curve of a long rock core using a single-moderate-speed centrifuge experiment and one dimensional quantitative magnetic resonance imaging (MRI) to determine the fluid saturation distribution, S(r), along the length of the sample, instead of non-quantitative spin echo based MRI [11], and nuclear tracer imaging technique [12]. A full capillary pressure

curve can be determined by the relation of S(r) and capillary pressure distribution, Pc(r), along the length of the core. This technique is a "single-shot" method [13].

MRI is a powerful, non-destructive, measurement method, which, with quantitative imaging techniques such SPRITE (single-point ramped imaging with T_1 enhancement) [14], offers unique advantages in the measurement of spatially resolved fluid saturation in rocks [15, 16].

The proposed method is rapid, precise, and relatively inexpensive. It is rapid because only one centrifuge speed is required. With a one dimensional saturation profile determined by SPRITE MRI, a capillary pressure curve, with approximately 40 data points is obtained. The proposed measurement is precise, because the fluid saturation distribution along the length of the core plug is directly measured. Since a single moderate centrifuge speed is employed, the effect of gravity is much smaller than the centrifugal force, and the outlet boundary condition of the rock core can be maintained. In addition, some friable and unconsolidated rock cores may be applicable to this method, since extreme high rotational speeds are not necessary. The new measurement will ultimately require only a desktop centrifuge and a desktop permanent magnet based one-dimensional MRI instrument.

CAPILLARY PRESSURE THEORY
Capillary Pressure
Two immiscible fluids (non-wetting phase and wetting phase) in contact in the pore networks of a porous medium, produce a discontinuity in pressure across the interface [17]. The pressure difference across the interface is called the capillary pressure, P_c, which is defined by the following equation

$$P_c = P_{non\text{-}wetting} - P_{wetting} \tag{1}$$

The capillary pressure is a result of the curvature of fluid interfaces, and the interfacial (or surface) tension, according to the Young–Laplace equation [5]:

$$P_c = \sigma\left(\frac{1}{R_1} + \frac{1}{R_2}\right) \tag{2}$$

where σ is the interfacial (or surface) tension between the two fluid phases and R_1 and R_2 are principal radii of curvature.

If the pore throat shape of a porous medium can be described as a cylindrical capillary tube, equation (2) becomes

$$P_c = 2\sigma\cos\theta / R \tag{3}$$

where R is the pore throat radius, and θ is the contact angle.

The capillary pressure can be converted directly into a pore throat size according to equation (3).

Traditional Centrifuge Methods
In 1945, Hassler and Brunner [7] proposed a centrifuge method to determine capillary pressure-saturation data from small core plugs. They also proposed an approximate solution to the basic equation relating capillary pressure and average saturation by neglecting the gravity effect and assuming the length of the core was negligible compared to the radius of rotation.

In a centrifuge capillary pressure experiment, a fluid saturated core plug, confined in a special core-holder, is rotated at different rotational speeds. The relevant distances are denoted as r_1, r, and r_2, as illustrated in Figure 1, where r_1, r_2, 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 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 with a stroboscope [10].

The basic concepts for capillary pressure measurement with a centrifuge are outlined below.

When a cylindrical core is placed in a centrifuge, a centrifugal acceleration $a_c = -\omega^2 r$, is generated, where ω 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

$$\frac{dP_c}{dr} + \Delta\rho\omega^2 r = 0 \tag{4}$$

where $\Delta\rho$ is the density difference between wetting fluid and non-wetting fluid.
The differential equation can be solved by simple integration

$$P_c(r) = \frac{1}{2}\Delta\rho\omega^2(r_2^2 - r^2) + P_{c2} \tag{5}$$

If we apply the Hassler-Brunner boundary condition, i.e., the capillary pressure at the outlet face of the core is assumed to be zero, i.e., $P_{c2} = 0$, so

$$P_c(r) = \frac{1}{2}\Delta\rho\omega^2(r_2^2 - r^2) \tag{6}$$

and for a continuous phase, the capillary pressure at the inner face of the core is

$$P_{cL}(r) = P_c(r_1) = \frac{1}{2}\Delta\rho\omega^2(r_2^2 - r_1^2) \tag{7}$$

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, ω, and the average fluid saturation, $\overline{S}$, within the core.

The average fluid saturation of the core after centrifugation can be expressed as

$$\overline{S} = \frac{1}{r_2 - r_1} \int_{r_1}^{r_2} S(r)dr \qquad (8)$$

Equation (8) 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

$$\overline{S}P_{cL} = \frac{r_1 + r_2}{2r_2} \int_0^{P_{cL}} \frac{S(P_c)}{\sqrt{1 - \frac{P_c}{P_{cL}}(1 - \frac{r_1^2}{r_2^2})}} dP_c \qquad (9)$$

Equation (9) cannot be directly solved for the unknown function S. As pointed out by Hassler and Brunner [7], for small values of α (short core), assuming $r_1/r_2 \approx 1$, then equation (9) is reduced to

$$\overline{S}P_{cL} = \int_0^{P_{cL}} S(P_c)dP_c \qquad (10)$$

whose differential form is

$$S_L = \frac{d}{dP_{cL}}(\overline{S}P_{cL}) \qquad (11)$$

The value of P_{cL} for each rotational speed is then calculated with Eq. (7), and the value of saturation at inlet face, S_L, is obtained according to Eq. (11). The relation of values of P_{cL} and S_L, at different rotational speeds, yields the capillary pressure curve. Equation (11) is an approximate solution introduced by Hassler and Brunner [7]. Based on equation (9), a number of other approximate solutions have been developed and used to determine capillary pressure curves [8, 18].

SPRITE IMAGING

Standard SPRITE Imaging
The standard SPRITE imaging [14] technique has proven, over the last 9 years, to be a very robust and flexible method for the study of a wide range of systems with short 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.

Centric scan SPRITE Imaging
A centric scan strategy for SPRITE imaging [19] 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 longitudinal relaxation time 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 centric scan SPRITE technique is illustrated in figure 2, where the k-space data are acquired sequentially from '0' to '$-k_z$', corresponding to a gradient change from 0 to minus maximum gradient (-Gmax), after a delay of 5 times T_1, the other of half k-space data is collecting from '0' to '$+k_z$', corresponding to a gradient change from 0 to a maximum gradient (Gmax). Fourier transform of the k-space data yields a real space image. In the centric scan SPRITE method, the image signal intensity (S) is given by:

$$S = M_0 \exp(-\frac{t_p}{T_2^*}) \sin \alpha \tag{12}$$

where M_0 is the equilibrium magnetization, α is the RF flip angle, t_p is the phase encoding time, T_2^* is the effective transverse relaxation time. M_o is proportional to the local fluid content. Centric scan SPRITE methods are naturally spin-density weighted.

Spin Density Imaging with SPRITE
A wide range of experimental results [16] show that the overall FID (free induction decay) decay rate ($1/T_2^*$) in sedimentary rocks is dominated by an internal field distribution (ΔB^i) induced by the large susceptibility difference ($\Delta \chi$) between the pore fluid and solid matrix due to paramagnetic impurities in the solid matrix. The decay rate of the FID and the corresponding NMR linewidth ($\Delta v = 1/\pi T_2^*$) for fluid saturated sedimentary rocks may be estimated by [16],

$$\frac{1}{\pi T_2^*} = \Delta v \approx \frac{\gamma \Delta B^i}{2\pi} = \frac{C \Delta \chi \gamma B_0}{2\pi} \tag{13}$$

where γ is the gyromagnetic ratio, and B_0 is the applied magnetic field strength, while C is a dimensionless constant.

Equation (13) predicts a single exponential T_2^* decay, this prediction has been confirmed by a wide range of NMR experiments for sedimentary rocks [16]. Single exponential T_2^* decay is anticipated for a wide variety of sedimentary rock systems, but is not a universal result.

Figure 3 shows a semi-logarithmical FID decay after a 90 degree RF excitation pulse. The data was fit to the equation:

$$S = M_0 \exp(-t/T_2^*) \tag{14}$$

where S is the NMR signal intensity, 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 images are essentially spin density images. Spin density images may not be obtained by spin echo based MRI methods, due to multi-exponential T_2 decay in rocks [11].

SINGLE-SHOT CAPILLARY PRESSURE CURVE MEASUREMENT

A cylindrical Berea sandstone was employed for the measurements. The dimensions of the core were measured to determine the bulk volume of the rock sample. The sample was dried at 80°C until a constant weight was reached, and the weight of the dried sample was determined. The sample was kept under vacuum conditions for more than 24 hours, and then saturated with distilled water under vacuum conditions until no bubbles came from the core. The porosity and permeability of the core were 0.19, and 0.2 Darcy, respectively.

The centrifuge experiment was carried out with a Beckman TJ6R tabletop centrifuge, at 1500 RPM for 140 minutes, the temperature in the centrifuge was controlled to be 4°C to avoid water evaporation during the centrifuge experiment. The duration of centrifugation is shorter than the duration typically used in crude/brine systems, since the equilibrium time for water/air system is reduced. In addition, the equilibrium time for high permeability rocks is less than that for low-permeability rocks. The centrifuge radius to the bottom of the rock core was 138 mm. A perforated Teflon end piece was employed to support the core and ensure full water saturation at outlet face. The cylindrical surface of the core sample was covered with a heat-shrink Teflon tube to maintain longitudinal fluid flow within the core.

All NMR experiments were carried out in a 2.4 Tesla horizontal bore superconducting magnet (Nalorac Cryogenics Inc., Martinez, CA) with an Apollo console (Tecmag Inc., Houston, TX). A proton-free 4.7 cm inner diameter eight-rung quadrature birdcage probe (Morris Instruments, Ottawa, ON) was employed. The core samples were wrapped with Teflon tape to decrease the evaporation of water within the samples during NMR measurements.

The water content profiles along the length of the core before and after centrifugation, were determined by 1D centric scan SPRITE MRI with a phase encoding time of 30μs, flip angle α of 6 degrees, for a field of view of 9 cm, with an image matrix size of 64 points. Four signal averages were acquired for a total acquisition time of 25 seconds. 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 image, which results in more data points on the capillary pressure curve.

Figures 4 and 5 show the 1D water content distribution along the length of the core before and after centrifugation, respectively. The left side and right side of the water content profile correspond to the inlet face and the outlet face of the core, respectively.

After centrifugation, the average water saturation was 46.3%. Figure 6 shows the 1D water saturation distribution along the length of the core. The water saturation at the outlet face is 100%, which shows that the outlet boundary condition is satisfied. Thus, MRI can be used to check the boundary condition for centrifuge capillary pressure measurements.

The capillary pressure distribution can be calculated with equation (6). Therefore, the relationship between capillary pressure and the corresponding water saturation can be determined very straightforwardly. The capillary pressure curve is shown in Figure 7.

The capillary pressure curve obtained with water and air can be converted to a capillary pressure curve under mercury intrusion conditions according to the following equation.

$$\frac{P_c^{Hg}}{P_c^{w-a}} = \frac{(\sigma \cos \theta)_{Hg}}{(\sigma \cos \theta)_{w-a}} \tag{15}$$

where P_c^{Hg} and P_c^{w-a} are the capillary pressures under mercury intrusion condition and water-air condition, respectively; $(\sigma \cos \theta)_{Hg}$ and $(\sigma \cos \theta)_{w-a}$ are the products of the interfacial tension and cosine of the contact angle under mercury intrusion conditions and water-air condition, respectively.

For mercury intrusion, $\sigma = 480$ mN/m and $\theta = 140$ degree; for a water and air system, $\sigma = 72$ mN/m and $\theta = 0$ degree. A comparison of the capillary pressure curves obtained by the proposed single-shot method and by mercury intrusion porosimetry are shown in Figure 8. The two methods yield remarkably consistent results.

GENERALITY OF THE SINGLE-SHOT METHOD

There are two ways to increase the range of capillary pressure distribution, i.e., increase the length of the sample and increase the rotational speed. The rotational speed chosen for the single-shot measurement should be sufficiently large to ensure that an irreducible water saturation is reached at the inlet face of the core. An estimate of the capillary pressure for irreducible water saturation may be obtained by the Leverett J function suggested by Leverett [20] as follows:

$$J = \frac{P_c}{\sigma \cos \theta} \sqrt{\frac{k}{\phi}} \tag{16}$$

where k is permeability, and ϕ is porosity.

The rotational speed for reaching the irreducible water saturation may be estimated by

$$P_c(r_1) = \frac{1}{2}\Delta\rho\omega^2(r_2^2 - r_1^2) \geq J(S_{wi})\sigma\cos\theta / \sqrt{k/\phi} \qquad (17)$$

$$\text{or} \qquad \omega^2 \geq \frac{2J(S_{wi})\sigma\cos\theta}{\Delta\rho(r_2^2 - r_1^2)\sqrt{k/\phi}} \qquad (18)$$

Reasonable Leverett J values at irreducible water saturation [21] are $J(S_{wi}) \approx 3\text{-}4$.

The rock sample for centrifuge capillary pressure measurement is required to be homogeneous, MRI can of course be employed to check the homogeneity of the core.

In this paper, we have introduced a method to determine the primary drainage capillary pressure curve using a single-speed centrifuge, with air displacing water. It is a logical extension of these ideas to measure the primary drainage, imbibition, and secondary drainage capillary pressure curves for the water/oil system at reservoir temperatures. In these experiments, it is necessary to distinguish between oil and water phases by MRI. This may be undertaken several different ways, for example, employing D_2O for the water phase.

CONCLUSIONS

In the current work, a new "single-shot" method to determine the capillary pressure curve is introduced. This method combines a single-moderate-speed centrifuge experiment for long rock cores and a quantitative SPRITE MRI technique to directly determine the fluid saturation distribution (from an irreducible water saturation at the inlet face to 100% water saturation at the outlet face) along the length of the core. The proposed method for determining capillary pressure curve is rapid, cheap, and precise. The capillary pressure curve can be obtained straightforwardly with about 40 spatial image data points, more data can be obtained simply by increase the spatial resolution of the MRI. The duration of the experiment is much less than the traditional method. Since only one single moderate rotational speed is employed, the outlet boundary condition can be maintained, the effect of gravity can be neglected, and there is no effect of centrifuge speed variations on pore structure of rocks [22]. In addition, a long rock core can be employed for the single-shot method result in a relatively small radial effect compared with a short sample [23]. A comparison of the capillary pressure curves obtained with the "single-shot" method and with the mercury intrusion porosimetry, yields remarkably consistent results.

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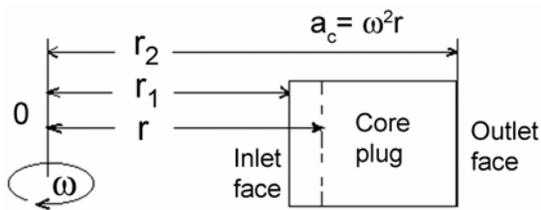
Figure 1. Schematic of a rock core plug spinning in a centrifuge.
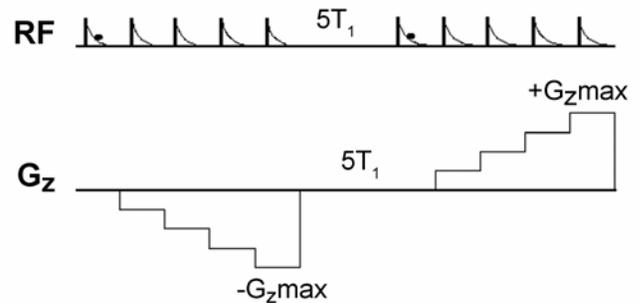
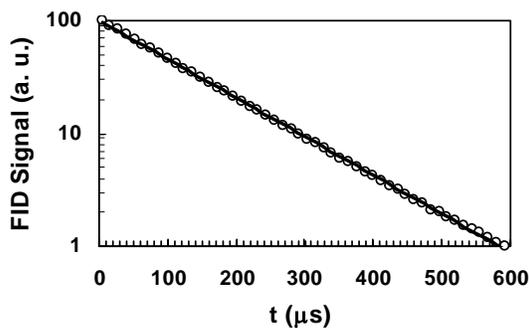Figure 2. 1D centric scan SPRITE imaging method.

Figure 3. A semi-logarithmical plot of the NMR FID (free induction decay) of the water saturated Berea sandstone. The best fit line is single exponential with a decay time constant T_2^* of 127μs.
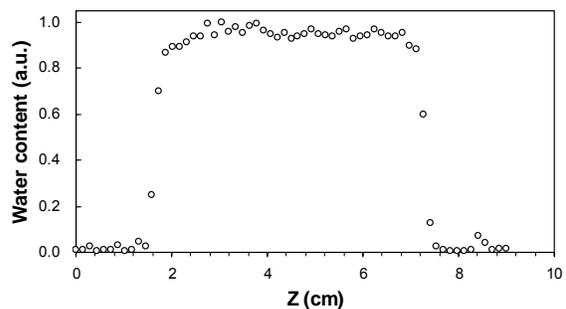

Figure 4. One dimensional water content distribution along the length of the fully water saturated rock core, determined with 1D centric scan SPRITE imaging.
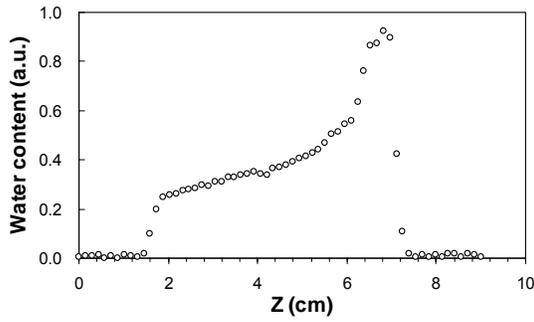
Figure 5. One dimensional water content distribution along the length of the rock core after centrifugation at 1500 rpm for 140 minutes, determined with 1D centric scan SPRITE imaging.
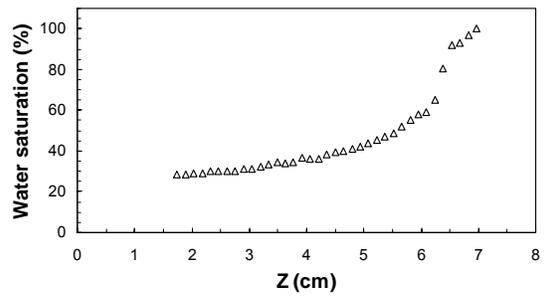

Figure 6. One dimensional water saturation distribution along the length of the Berea sandstone core after centrifugation at 1500 rpm for 140 minutes, determined with 1D centric scan SPRITE imaging. The water saturation is 100% at the outlet face of the core.
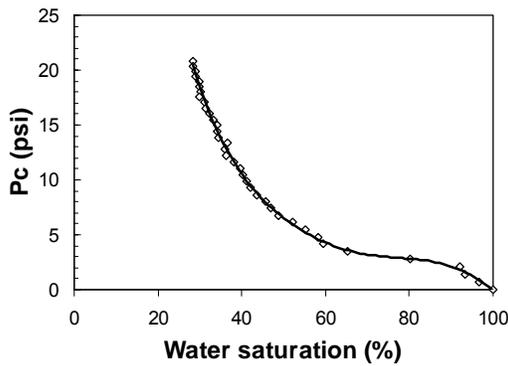

Figure 7. A drainage capillary pressure curve of the Berea sandstone core determined with a single-shot measurement method. The solid line is an interpolation through the experimental data points.
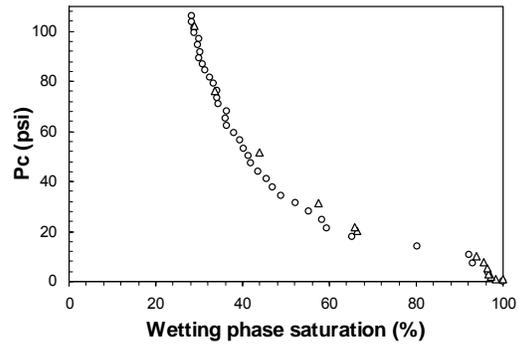

Figure 8. A comparison of drainage capillary pressure curves of the Berea sandstone determined by mercury intrusion porosimetry (Δ) and by a single-shot measurement ($\circ$).