Valid Capillary Pressure Data at Low Wetting-Phase Saturations

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Summary. Air/brine capillary pressure data obtained by three different techniques are reported as a function of saturation for several Berea core-plug samples. The capillary pressure values ranged from about 0.67 to 9.5 MPa [97 to 1,380 psi]. The corresponding brine saturations ranged from 10 to 3% PV. The techniques used were high-speed centrifuge, porous plate, and water-vapor desorption (WVD). The results show that a plot of log P_c vs. log S_w is linear in the saturation range studied. These data are believed to be the first valid capillary pressure data reported for Berea samples in the high-pressure, low-saturation region. Also, no comparable data appear to have been published for any sedimentary rock in the permeability range above 1 md.

Introduction

Capillary pressure data are used in reservoir engineering to determine the initial distribution of brine, oil, and gas in the reservoir. Such a determination is particularly important in reservoirs with very large oil columns where low brine saturations and high capillary pressures are encountered. In the early published work on this problem, however, the available experimental techniques were quite limited in the range of capillary pressures that could be attained.[1] In most instances, only the mercury-injection technique had the capacity of reducing the wetting-phase saturation to values below about 15%. For this and other reasons, the concept arose that reservoir rocks are characterized by a minimum or irreducible water saturation value.

Today, each of the traditional methods used to measure capillary pressure is capable of reaching a much higher range of capillary pressures.[1] As a result of advances in experimental technique, it is now clear that the concept of an irreducible water saturation is not valid. It is also recognized that the porous-plate and centrifuge techniques often have been used in such a way that the data obtained do not correspond to true equilibrium conditions.[2]

In this paper, air/brine capillary pressure data are reported for three matched samples of Berea sandstone. The data were obtained in the capillary pressure range from about 0.67 to 9.5 MPa [97 to 1,380 psi]. The corresponding brine saturations ranged from 10 to 3% PV. Three different experimental techniques were used: (1) high-speed centrifuge, (2) porous plate, and (3) WVD. To the best of my knowledge, no previous work has been reported in which three different methods were used with a set of matched samples. Also, the data obtained in this work are believed to be the first high-pressure data reported for sandstone samples with permeabilities above 1 md. Portions of the work that have been previously presented[1,3] are not covered in detail here.

Core Data and Methods

Table 1 gives the initial dry-core data for the three matched Berea plug samples. These samples were nominally 3.8 cm [1.5 in.] in diameter and 5.1 cm [2 in.] in length. Dry weights averaged 117 g, while the average PV was 11.0 cm^3. Gas permeabilities were about 200 md, and the average porosity was 20.0%. After cleaning by extraction with a boiling methanol/toluene mixture, all three samples were saturated with a 2.0 wt% NaCl solution. Because air was the displacing phase in the capillary pressure experiments with these samples, a zero contact-angle value was believed to be maintained in all cases.

The following sequence of air/brine capillary pressure experiments was followed. First, the three plugs were simultaneously desaturated by the porous-plate method at a single capillary pressure level of 2.24 MPa [325 psi]. The plugs were then extracted, dried, reweighed, and resaturated with the same NaCl solution. Next, a high-speed-centrifuge desaturation run was carried out on all three samples at five centrifuge speeds from 4,260 to 13,680 rev/min. Following this experiment, the plugs were again extracted, dried, reweighed, and resaturated. The final experiment first involved centrifuge desaturation at about 10,000 rev/min. During this step, one of the centrifuge cells did not seal properly. For this reason, the sample held in this cell was not used in the subsequent WVD experiment. A detailed description of this last experiment and the results obtained are given in a previous paper.[3]

Fig. 1 is a schematic illustrating the porous-plate method. A commercially available pressure-membrane apparatus was used.[4] The membrane furnished with this apparatus was of the cellulose type. No special means were used either to prepare the ends of the plug samples or to ensure contact with the membrane. The maximum capillary pressure at which the equipment was rated, with this particular membrane, was about 6.5 MPa [1,000 psi]. It was anticipated that localized drying of the membrane might occur during a desaturation run of 1 month or more because of its large lateral area. This would lead to gas breakthrough at the point at which drying occurred. To avoid this possibility, a small open beaker of brine was placed in the pressure chamber. No fluid production was detected after 38 days. Saturation was determined gravimetrically. Temperature was maintained at 23±0.5°C [73±0.9°F].

The centrifuge capillary pressure experiment was carried out with a Beckman L5-50P Rock Core Ultracentrifuge™.[5] A three-place rotor (PIR-16.5), designed for plug samples with the nominal dimensions specified above, was used. The overall run time for the five speeds used was 21 days. The laboratory temperature was maintained, as in the porous-plate experiment, at 23±0.5°C [73±0.9°C].

A major concern in high-speed-centrifuge runs over an extended time period is the possible failure of the so-called Hassler boundary condition.[6,7] This failure arises when the displacement pressure for a particular sample is exceeded in the pores leading from the interior of the plug to the boundary at the bottom end-face. In the present work, the sample permeability appears not to have been sufficiently high for this problem to occur. Fig. 2 shows the capillary pressure value at which the Hassler boundary condition is expected to fail as a function of permeability.[6] If the sample permeabilities in the present work had exceeded about 400 md, failure of the Hassler boundary condition might have occurred. A more detailed discussion of the boundary-condition problem has been presented by O'Meara *et al.*[8]

All methods of measuring capillary pressure exert stresses on the samples undergoing testing. Obviously, errors would occur if these stresses caused a significant change in the internal pore structure. The possibility of a permanent, in contrast to a purely elastic, change was monitored by redetermining the sample weights and PV's after each of the first two methods was applied. The combined effect of the porous-plate measurement (46 days) and the high-speed-centrifuge run (21 days) was to decrease the sample weight by about 0.2% and to increase the measured PV by about 0.3%. These observations indicate that the increase in porosity was only about 0.4% of the initial porosity.

As already suggested, each method of measuring capillary pressure has an inherent limitation with respect to the maximum value of the capillary pressure that can be reached. For the porous-plate method, this limitation is determined by the size of the largest pores in the membrane material used. For the centrifuge method, the limitation is a result of the failure of the Hassler boundary condition,[6]

TABLE 1—DRY CORE DATA FOR 2-in. BEREA PLUGS*

Sample	Dry Weight (g)	PV (cm^3)	Porosity (%)
B-22-5	116.0895	11.008	20.10
B-22-6	113.2993	10.765	20.15
B-323-1	122.0242	11.310	19.76

*Gas permeability ~ 200 md, grain density = 2.655 g/cm^3, average length = 4.80 cm.

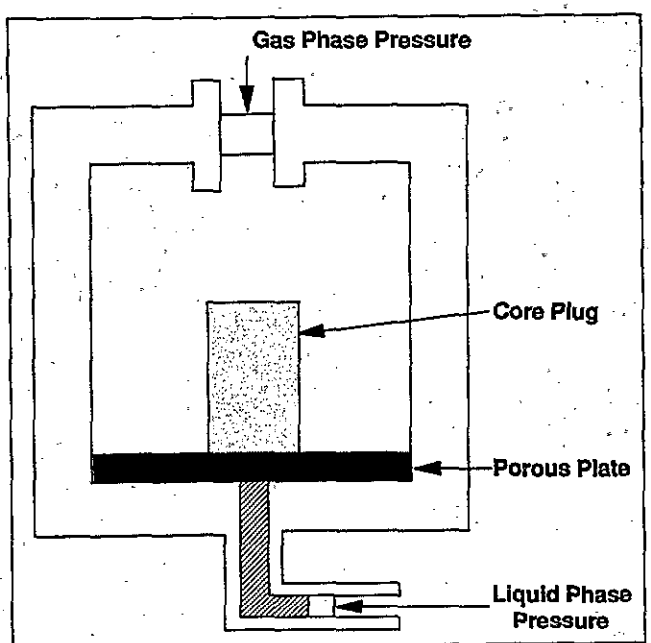

Fig. 1—Schematic, capillary pressure measurement by porous-plate method.

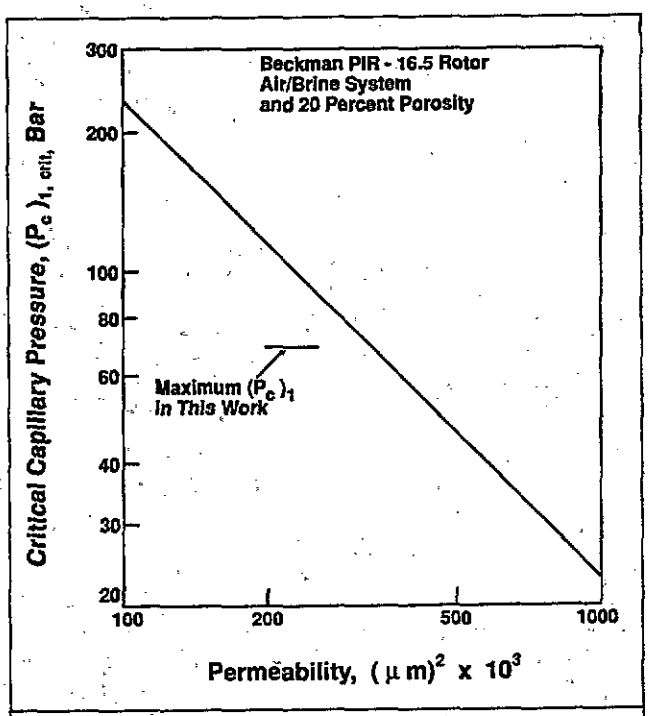

Fig. 2—Limiting capillary pressure with high-speed-centrifuge method.

TABLE 2—AIR/BRINE POROUS-PLATE CAPILLARY PRESSURE DATA*

Sample	P_c (MPa)	Equilibrium Time** (days)	S_w (%)
B-22-5	2.24	38	6.29
B-22-6	2.24	38	6.35
B-323-1	2.24	38	6.13

*Temperature = 23 ± 0.5°C, 2.0 wt% NaCl brine.
**Total run time = 46 days.

as indicated previously. For the WVD method, the limitation arises when the tensile strength of the liquid phase is reached. The available experimental evidence relating to this limitation has been reviewed previously.[3] This evidence indicates that the limit is at least 27.5 MPa [4,000 psi]. In the capillary pressure range above about 12 MPa [1,740 psi], however, corrections to the classical form of the Kelvin equation may be required. Thus, in a previous review[1] of the limitations of each capillary pressure method, the value of the limiting air/brine capillary pressure indicated was 12 MPa [1,740 psi]. It should be emphasized that this very conservative estimate of the limiting value refers only to the use of the uncorrected form of the Kelvin equation. When the more general form of this equation[3] is used, the WVD method is applicable over a much larger range of capillary pressure values.

Results

Table 2 records the air/brine capillary pressure data obtained by the porous-plate method at a single pressure level. The average brine saturation for the three plugs was 6.26%, with a standard deviation of 0.09 saturation %, or about 1.5% of the average value. This deviation indicates excellent agreement among the three samples. The long equilibration time of 38 days is obviously a serious disadvantage in the routine use of this method. A somewhat shorter time might have been required had the samples been partially desaturated by another method before being placed in the pressure chamber of the equipment used. On the other hand, this procedure probably would have resulted in a larger standard deviation because additional sources of error would be involved.

In Table 3, the primary data obtained from the high-speed-centrifuge capillary pressure measurement are given. A run time of 6 days was used at each of the three highest speeds. In all cases, equilibrium appeared to be reached at a considerably shorter time than the run time adopted. The agreement among the average saturation values at a given speed was less satisfactory than for the porous-plate experiment. On average, the standard deviation, as a percent of the average saturation value, was about three times larger than in the porous-plate case. This decrease in data precision no doubt results because the saturation determined by a volumetric method is less accurate than that determined by a gravimetric method. The deviations in the centrifuge speed values over the course of a run at any one speed were about ±10 rev/min.

The centrifuge speed data and the average saturation data were converted to the corresponding top endface P_c and S_w values with the usual equations used in the interpretation of primary centrifuge data.[6] Table 4 gives the averages of the top endface capillary pressure and saturation values for the three plugs. The usual Hassler approximation was used because it involves only a small error in the low-saturation range.[6] Fig. 3 is a plot, on logarithmic scales, of the endface capillary pressure as a function of the average saturation in the core plug. The slope of this function is plotted vs. the endface capillary pressure in Fig. 4. The values of the slope shown in Fig. 4 and given in Table 4 were used in the calculation of the Hassler endface saturation.

In Fig. 5, the results of the centrifuge method are shown as a plot of log P_c vs. log S_w. Also shown is the porous-plate result obtained at a single pressure level. The line defined by the three

TABLE 3—PRIMARY DATA, AIR/BRINE CENTRIFUGE RUN*

Centrifuge Speed (rev/min)	Run Time (days)	$\langle S_w \rangle$ (%) B-22-5	B-22-6	B-323-1
4,620	1	17.7	16.5	15.9
6,080	2**	13.2	13.7	13.3
7,840	6	9.8	11.4	11.1
11,130	6	8.6	7.9	7.8
13,680	6	6.3	6.8	6.7

*Temperature = 23 ± 0.5°C, 2.0 wt% NaCl brine.
**Temporary shutdown caused by power outage that occurred after 1 day at this speed.

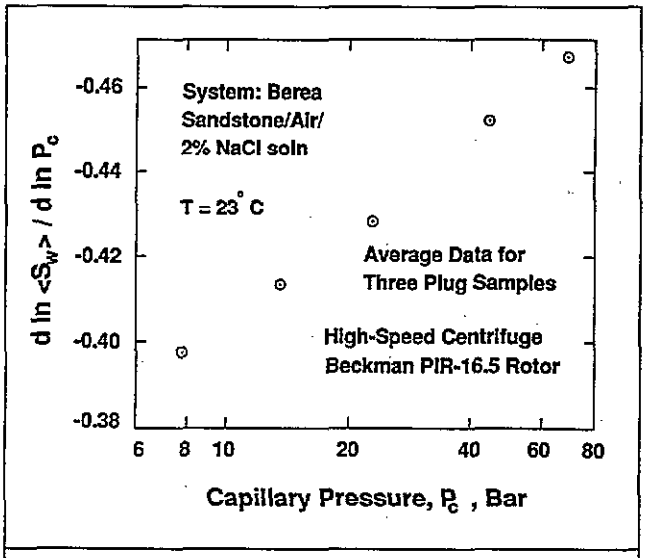

Fig. 4—Slope of log P_c vs. log $\langle S_w \rangle$ as a function of P_c.

TABLE 4—AIR/BRINE HIGH-SPEED-CENTRIFUGE CAPILLARY PRESSURE DATA*

P_c (MPa)	$\langle S_w \rangle$ (%)	Slope** $-d \ln \langle S_w \rangle / d \ln P_c$	$(S_w)_H$† (%)
0.79	16.7	0.398	10.1
1.37	13.4	0.413	7.9
2.28	10.8	0.427	6.2
4.60	8.1	0.452	4.4
6.95	6.6	0.466	3.5

*Average data for three plug samples.
**Determined from smoothed plot of ln P_c vs. ln $\langle S_w \rangle$.
†Top endface saturation, Hassler approximation.

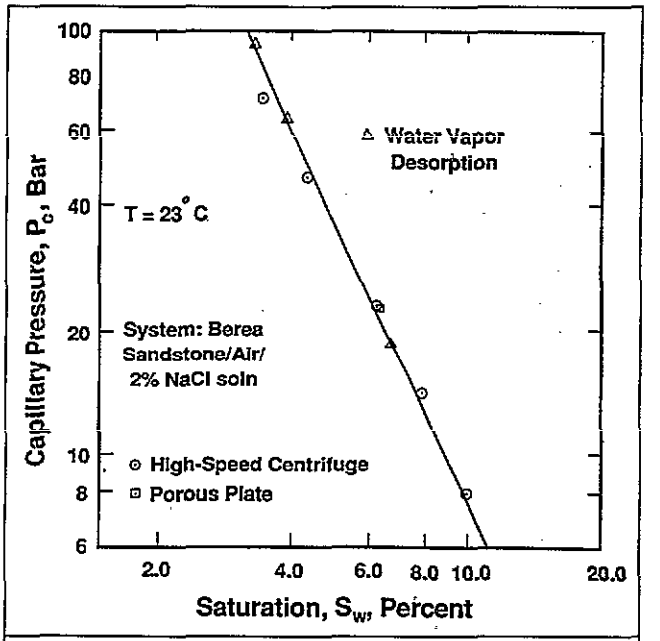

Fig. 5—Endface capillary pressure vs. endface saturation.

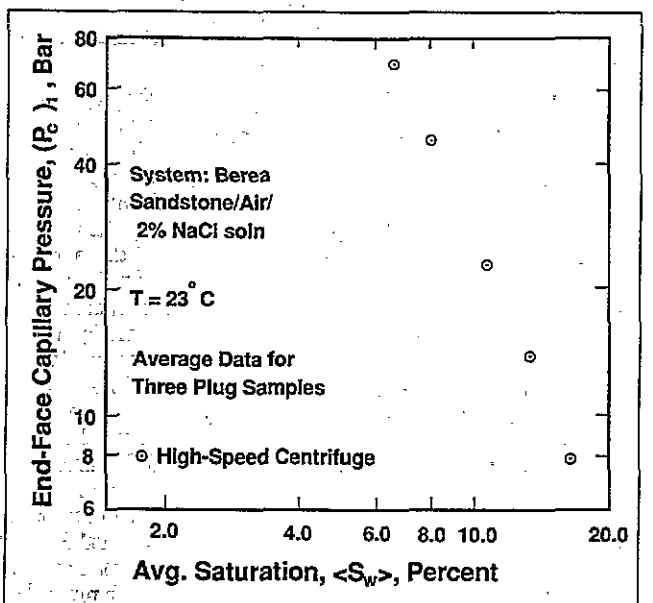

Fig. 3—Endface capillary pressure vs. average core saturation.

TABLE 5—COMPARISONS OF AIR/BRINE CAPILLARY PRESSURES BY DIFFERENT METHODS

P_c (MPa)	S_w (%) Centrifuge	Porous Plate	WVD
0.79	10.1	—	—
1.37	7.9	—	—
1.84	—	—	6.7
2.24	—	6.3	—
2.28	6.2	—	—
4.60	4.4	—	—
6.37	—	—	3.9
6.95	3.5	—	—
9.36	—	—	3.3

WVD values reported previously[3] is included in Fig. 5 for comparison. The excellent agreement between the porous-plate result and the high-speed-centrifuge data is evident.

Table 5 summarizes the data obtained by all three methods. Also shown in Fig. 5 are the individual data points obtained by the WVD method. Note that the WVD data represent the average results for two samples, whereas the porous-plate and centrifuge data are averages for three samples. The agreement among all three methods, along with the small standard deviations of the values for individual samples when tested by any one of the methods, provides strong evidence that the data are valid.

Discussion

Previous studies of reservoir-rock samples in the high-pressure, low-saturation region of the capillary pressure curve have been quite limited in scope. In the first such study, by Calhoun *et al.*[9] in 1949, WVD data were obtained on four samples of artificially consolidated sandstone. Obviously, clay minerals were not present in these samples. On the other hand, the consolidating material may have been sufficiently porous to affect the results. Porous-plate capillary pressure data were reported, but these data were restricted to the low-capillary-pressure range—i.e., below 400 kPa [58 psi]. The WVD data were restricted to the range above 7 MPa [1,000 psi]. Consequently, verification of the WVD data by comparison with a second method was not possible.

A second study that used water-vapor adsorption as well as desorption was reported by Hsieh and Ramey[10] in 1983. The Berea sandstone samples used in that work were similar to those used in the present study. However, the capillary pressures computed from the Kelvin equation were in excess of 40 MPa [5,800 psi]. In this range, a method based on vapor adsorption/desorption is clearly the only method applicable. As pointed out previously, however, in this range, the general rather than the approximate form of the Kelvin equation must be used.

A more recent study by Ward and Morrow[11] appears to be the first in which WVD data were actually compared with data obtained by a second method. It was found that over the range of air/brine capillary pressures from 1.4 to 5.5 MPa [200 to 800 psi], independent high-speed-centrifuge data were in approximate agreement with WVD data. These data were obtained on a set of five different core plugs taken from various low-permeability (<0.1-md) gas sands. The range of overlapping capillary pressures generally corresponded to brine saturations ranging from 25 to 45%.

A final study relevant to this work is that of Dullien *et al.*[2] With a calcined Berea sandstone sample, an oil/brine porous-plate capillary pressure of 100 kPa [14.5 psi] was found to reduce the brine saturation to about 10%. This capillary pressure is approximately equivalent to an air/brine capillary pressure of 200 kPa [29 psi]. Thus, at equivalent capillary pressures, the saturation reported by Dullien *et al.* is significantly less than would be expected from an extrapolation of the data obtained in the present work. This difference in saturation, however, can be attributed[3] to the destruction of surface area and microporosity by the procedure used to ensure water-wet conditions. This procedure involved heating the samples at 600°C [1,112°F] for 2 days. Despite the lack of agreement in the observed saturation, the work of Dullien *et al.*[2] clearly shows that the concept of a limiting or irreducible wetting-phase saturation in sandstones is not supported by carefully conducted experiments.

Another method of obtaining capillary pressure data in the high-pressure region is mercury injection into an evacuated sample. This method was not attempted in this study for two reasons. First, a significant uncertainty exists about the proper method of scaling the effect of differing contact-angle values. Because the pore walls in the low-saturation region are predominantly converging instead of parallel—i.e., cylindrical or slit-shaped—the factor $\cos\theta$ clearly is not an appropriate scaling factor. If this factor is adopted to scale the capillary pressure coordinate, an additive correction to the saturation measured by mercury injection is required. Model calculations show that this converging pore-wall correction is far from negligible.[12] Second, mercury-injection data indicate saturations in the high-pressure region that are too low because of the absence of a contribution to the saturation from wetting films. Model calculations again show that this is by no means a negligible contribution.[12]

An important point to emphasize is that each method of obtaining capillary pressure data has its own advantages, limitations, and special problems relating to the interpretation of the primary data obtained. In attempting to extend a particular method into the high-pressure region, all these factors obviously require careful consideration. However, the limitations and special problems encountered with a given method are not always clearly defined and self-evident. Therefore, the only way of establishing the validity of the data obtained is to use more than one independent method, which is the approach taken in this work. The good agreement among the three techniques used greatly enhances the probability that the results are valid. Each of the techniques used appears to be free from significant systematic errors over the capillary pressure range studied.

A final point relates to the form of the capillary pressure curve in the high-pressure, low-saturation region. The data in Fig. 5 indicate that $\log P_c$ is linearly related to $\log S_w$ in the region in question. This linear relationship may be a characteristic feature of the microporosity associated with the assemblage of clay minerals present in Berea sandstone. Further studies of the high-capillary-pressure region in other sandstones are clearly needed.

Conclusions

1. New experimental capabilities have extended the traditional limits for measuring capillary pressures on reservoir-rock core-plug samples.
2. In the low-water-saturation region, the porous-plate, high-speed-centrifuge, and WVD measurements all give concordant results when properly used.
3. The use of more than one independent method appears to be the only way to establish the validity of results obtained in the high-capillary-pressure range because the experimental difficulties arising in this range are not always easily recognized.
4. For Berea samples with permeabilities of about 200 md, the plot of $\log P_c$ vs. $\log S_w$ is linear in the low-saturation region; no evidence for a minimum or irreducible saturation is found.
5. At the 9.5-MPa [1,380-psi] air/brine capillary pressure level, the brine saturation for Berea samples was found to be about 3% PV; at 670 kPa [97 psi], the saturation was about 10%.

Nomenclature

P_c = capillary pressure, MPa [psi]
$(P_c)_H$ = capillary pressure calculated with Hassler approximation, MPa [psi]
$(P_c)_1$ = top endface capillary pressure, MPa [psi]
$(P_c)_{1,crit}$ = critical value of $(P_c)_1$, MPa [psi]
S_w = brine saturation
T = temperature, °C [°F]
θ = contact angle, degrees

Acknowledgments

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SI Metric Conversion Factors

bar	× 1.0*	E+05	= Pa
°F	(°F−32)/1.8		= °C
in.	× 2.54*	E+00	= cm
md	× 9.869 233	E−04	= μm^2
psi	× 6.894 757	E+00	= kPa

*Conversion factor is exact.

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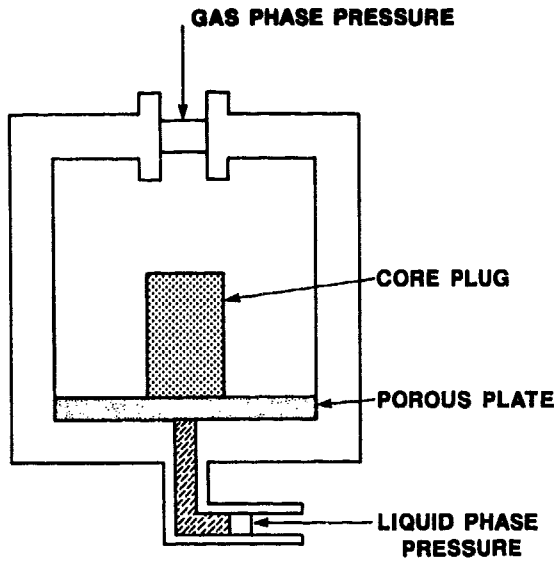

Fig. 1—Schematic diagram, capillary pressure measurement by porous plate method.

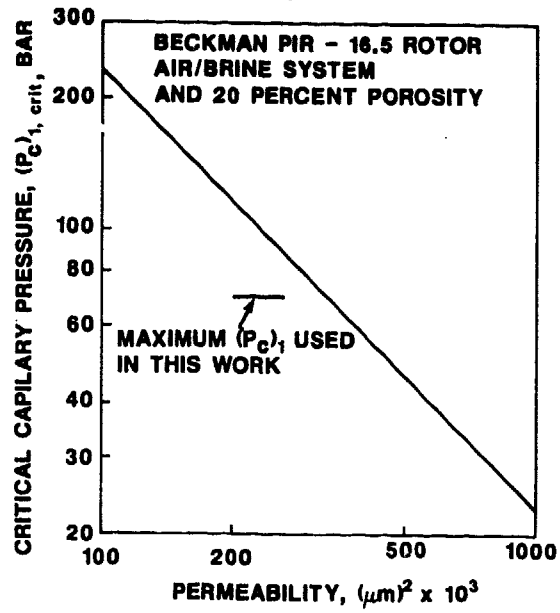

Fig. 2—Limiting capillary pressure using high-speed centrifuge method.

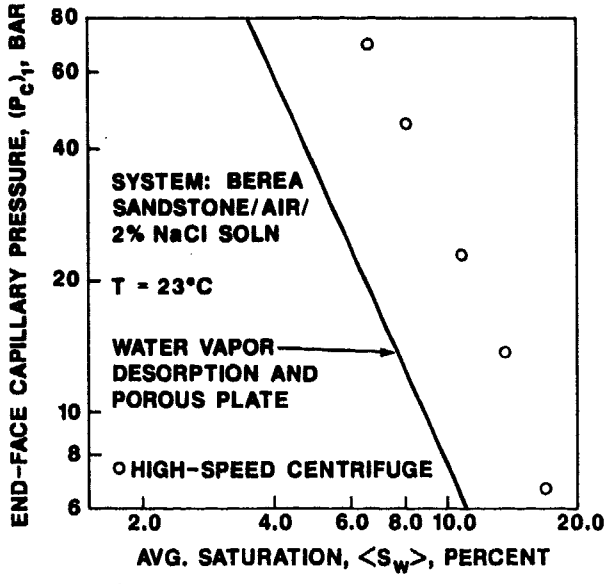

Fig. 3—End-face capillary pressure vs. average core saturation.

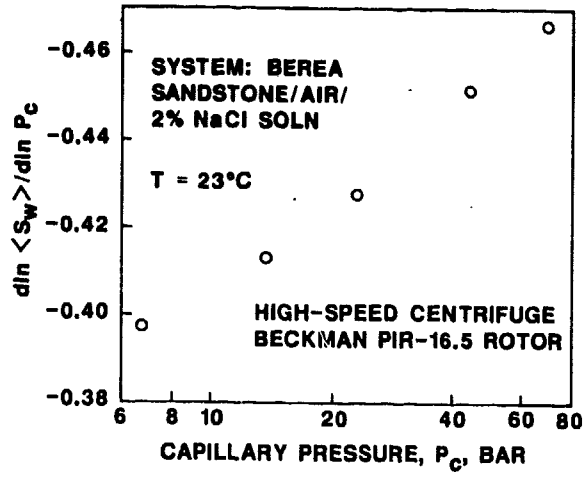

Fig. 4—Slope of Log Pc vs. Log $\langle S_w \rangle$ as a function of Pc.

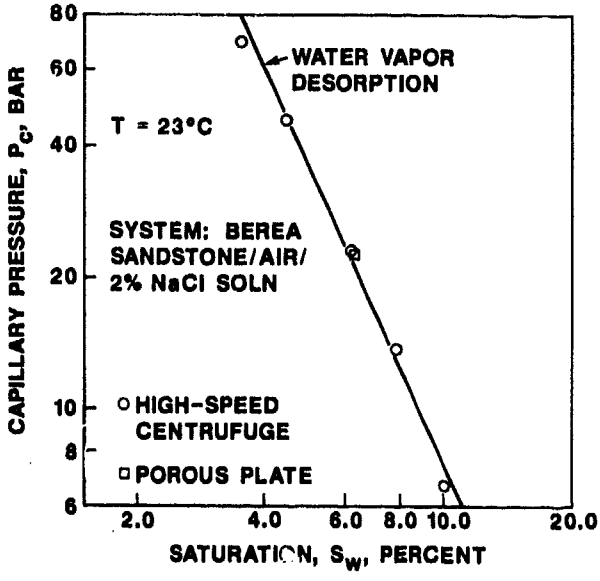

Fig. 5—End-face capillary pressure vs. end-face saturation.

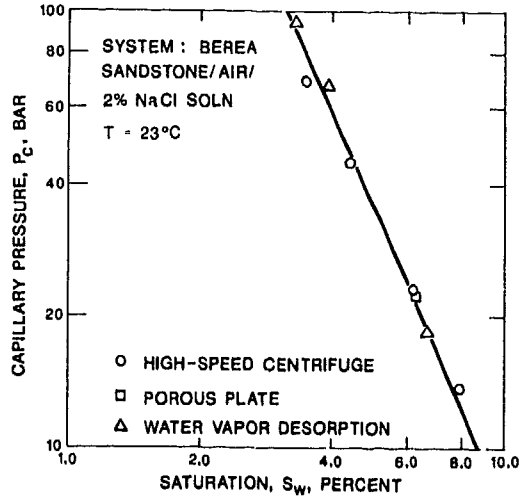

Fig. 6—Capillary pressure data by three different methods.