

**A NOVEL METHOD FOR THE DETERMINATION OF
WATER/OIL CAPILLARY PRESSURES OF
MIXED WETTABILITY SAMPLES**

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ABSTRACT

Capillary pressure curves are generally obtained by the porous plate or the centrifuge methods. Both methods have their drawbacks, especially for imbibition curves and mixed wettability samples. We developed a method which easily provides the whole cycle of capillary pressure (drainage and imbibition for both positive and negative P_c) and for any type of wettability.

The principle of our method is to realise a steady flow in the sample and to deduce the capillary pressure P_c from the local balance between viscous and capillary forces. The set-up is a standard displacement cell with a pressure tap. The outlet face is washed by a continuous flow of the in-situ fluid to realise the condition of known uniform pressure and permit the invasion of this fluid. Ultrasonic transducers along the sample give the local saturation after calibration by using the effluent balance.

For the first time, whole pressure cycles are obtained for sandstone and sands of different wettabilities. The positive P_c qualitatively agree with results obtained by standard methods. The effect of wettability on the shape of the cycles can be interpreted by assuming the existence of an oil-wet and a water-wet network in the sample.

Compared to the centrifuge, the main advantage of this method is to give the positive imbibition curve (and negative drainage). Compared to the porous plate, there is no need for semi-permeable membranes. This technique can be used at high pressure and high temperature in a modified Hassler cell. In addition, it can be easily automated.

1. INTRODUCTION

It is now recognized that most of the reservoirs are not strongly water-wet (Cuiec¹, Anderson²). However the capillary pressure curves are still obtained by using techniques developed for strongly water-wet systems, such as the porous plate or mercury injection. This paper describes an improved method, called semi-dynamic, to measure the capillary pressure of mixed-wettability rocks.

For water-wet samples, the standard measurements of capillary pressure consist of a drainage and an imbibition, where the pressure in oil is always larger than the pressure in water. For mixed wettability samples, the roles of oil and water are symmetrical and the pressure in oil can be larger or smaller than the pressure in water during a displacement (positive and negative capillary pressure). Negative capillary pressures are measured either by the porous plate method with a membrane permeable to oil or by the centrifuge method with "imbibition" core-holders (Glotin et al.³).

Both methods are difficult to operate. In order to obtain the whole cycle of positive and negative drainage, the porous plate method requires two different experiments with different kinds of membranes. Centrifuge also needs different core holders for drainage and imbibition. In addition, positive imbibition and negative drainages cannot be obtained with standard equipment.

In this paper, we propose a novel approach which can easily give the whole cycle of positive and negative drainage and imbibition. We first recall the notations used in this paper and shared by most of the colleagues in petroleum engineering. Then the principle of the method is described and compared to the existing ones: the measurements are based on the balance between capillary pressure and viscous pressure drop. In the third part, preliminary results are presented. For the first time, cycles of positive and negative capillary pressures are obtained for sandstone and sands of different wettabilities. The effect of wettability on the P_c curves is then interpreted. In conclusion, we recall the advantages and the drawbacks of this technique.

2. NOTATIONS

We will first recall the notations which are commonly used in petroleum engineering. In fact they are derived from the water-wet case. Any other definition becomes very rapidly confusing for mixed or unknown wettability.

- *Capillary pressure.* Capillary pressure is defined as $P_c = P(\text{oil}) - P(\text{water})$. However, this difference has a physical meaning and can be used in the two-

phase Darcy's equations only if the fluids are continuous. We will discuss this point when presenting the experimental results.

- A *drainage* is a displacement where water saturation S_w is decreasing; an *imbibition* is a displacement where water saturation increases.
- A *primary* displacement occurs in a medium fully saturated with either oil or water. A *secondary* displacement follows the primary one: for instance, after a first drainage takes place a secondary imbibition.
- A displacement is *controlled* (rather than forced) when the pressure or the flow rate is fixed by the operator. Centrifuge and porous plate are *pressure controlled* displacements. The dynamic method described in the next section is a *rate controlled* displacement. The semi-dynamic technique described in this paper is rate controlled for one fluid and pressure controlled for the other fluid.
- A displacement is *spontaneous* if the external pressure difference is set to zero. Generally, the sample is immersed into one of the fluids. For mixed wettability samples, we can observe both spontaneous drainage and imbibition.
- A *Pc curve* is a smooth curve presenting the variations of P_c as a function of water saturation S_w . This curve is obtained by a succession of controlled displacements with small increments of pressures (or flow rates).

This set of definitions leads to the different parts of the *Pc curves* presented in figure 1. Initially the sample is saturated with water:

- 1) Primary drainage ($P_c > 0$): oil is displacing water
- 2) Positive (secondary) imbibition
- 3) Negative (secondary) imbibition.
- 4) Negative (secondary) drainage
- 5) Positive (secondary) drainage.

In addition, we can represent the end points of spontaneous displacements, for instance a spontaneous (secondary) imbibition, when the pressure is released at end of drainage and the sample immersed in water (2').

A primary imbibition curve (3') corresponds to the injection of water in a medium fully saturated with oil.

3. PRINCIPLE OF THE SEMI-DYNAMIC METHOD

In this method, the capillary pressure is balanced by the viscous pressure drop. The experimental set-up is described in figure 2. It consists of a standard core-holder without any semi-permeable membranes and a separator used to measure the fluid production. The saturations are measured at different sections of the sample by using an ultrasonic method. Any other technique could be used (conductivity, X-ray, γ -ray, etc.). We will only describe the measurement for positive capillary pressures. For negative P_c , the role of water and oil are permuted.

The valve V1 is closed and V2 is open (fig. 2). Oil is injected through the sample and water is only "washing" the outlet face of the sample in order to realize the continuity between water inside the sample and the outlet. The first role of this circulation is to impose the pressure P_w in water to a known value (atmospheric pressure, or any fixed back-pressure, corrected by the buoyancy in the tubings). The second role is to permit the invasion of water into the sample when the pressure in oil is decreased (imbibition).

An increase of oil flow rate displaces water and the level of the meniscus in the fluid separator rises. When the steady state is reached, water is no longer produced and therefore, there is no pressure gradient in the water phase. The water pressure P_w is uniform along the sample. The pressure P_o in oil is measured through a hole in the coating or in the rubber sleeve of the core-holder. Through this hole, we measure the pressure in the fluid which would flow through the opening; i. e. the injected fluid. For positive P_c , the measured pressure is the oil pressure. For negative P_c , when water is injected, the pressure will be the water pressure. From the values of P_w and P_o , we can calculate the capillary pressure defined as $P_c = P_o - P_w$.

The method also requires the measurement of the saturation S_w at the same section as the pressure to obtain one point $P_c(S_w)$ of the capillary pressure curve. The primary drainage curve is obtained by increasing the oil flow rate from zero in a sample initially saturated with water. Imbibition (curve 2, fig.1) is obtained by decreasing the oil flow rate. During all these displacements, the water flow rate is kept constant at a low value to not perturb the flow in the sample (negligible pressure drop) but high enough to wash the oil in the outlet.

The negative part of the P_c curve is obtained by permuting the roles of oil and water (V1 is open and V2 is closed). The negative imbibition (3) (Fig. 1) is obtained by increasing the water flow rate, and the secondary drainage (4) by decreasing this flow rate. The secondary drainage (5) is obtained by injecting oil again.

To summarize our method: the local saturation and oil and water pressures are measured during a displacement. But we overcome the need for semi-permeable membranes by an original flow set-up.

4. COMPARISON WITH OTHER METHODS

Our method can be seen as an intermediate between the porous plate and the dynamic method introduced by Brown⁴. Let us describe briefly the different steady-state methods used for measuring capillary pressures. The principles of these methods are shown in figure 3, together with the diagram of the pressures in oil and water along the sample (from inlet A to outlet B) .

- *Porous plate*: The capillary pressure is balanced by the *static* pressure difference between the fluids. In principle, the fluids are injected through two semi-permeable membranes to measure the positive and negative part of the capillary pressure curve. The displacements are controlled by the difference of pressure between the fluids which is increased step by step during drainage (and decreased during imbibition). With the two semi-permeable membranes, this difference can take any positive or negative values and the whole controlled P_c cycle can be measured (curves 1 to 5, fig. 1). During such a displacement, the saturation is uniform along the sample and its value can be deduced from the effluent production. To our knowledge, there is no published result using this method.
- *Centrifuge*: (see for instance O'Meara et al⁵). The capillary pressure is balanced by the difference of hydrostatic pressure due to centrifugation. The pressure profile in each fluid can be related to the rotation speed and the fluid density. In drainage, for instance, the sample is immersed in oil and P_c is assumed to be zero at the outlet B (fig. 3). At any section of the sample, the pressure is known, but the saturation must be calculated from the effluent production by using the general flow equations. This difficulty can be overcome, by directly measuring the saturation while centrifuging (Chardaire et al⁶, Forbes et al⁷).
- *Dynamic method*: In the experiments described by Brown⁴, the two fluids are simultaneously injected, like in the measurement of a steady-state relative permeability. The non-wetting fluid (gas in Brown's experiments) is injected and produced behind a semi-permeable membrane. The flow rates are adjusted in order to have the same difference at the entrance and the exit of the sample (equal to P_c). This condition leads to a uniform capillary pressure along the sample and therefore to a uniform saturation. This saturation is deduced from the effluent balance. This experiment was proposed as a validation of the porous plate method: the static and dynamic P_c were proved to be identical.

- *Semi-dynamic method* (this paper). As shown on the pressure diagram, the pressure in water is constant like in the static porous plate method (for $P_c > 0$). Pressure in oil follows the pressure drop as in the dynamic method. For these reasons, this method can be called “semi-dynamic”.

In addition to these steady-state methods, a transient method has been developed by Kalaydjian⁸. Pressures and saturation are measured in the same section of the sample. Oil and water pressures are measured by using semi-permeable membranes and saturation by using an ultrasonic method. These three parameters are recorded during a displacement, like during an unsteady-state relative permeability experiment. The main conclusion was a strong effect of the flow rate on the P_c curve.

Ramakrishnan and Capiello⁹ also proposed the principle of a method for measuring the relative permeabilities and the capillary pressure by injecting only one fluid at various flow rates. However, in their set-up, there is no washing of the outlet and the pressure in the in-situ fluid is unknown. In addition the parameters are obtained from the derivation of the injection pressure, a method less accurate than the direct measurements proposed in our method. On the other hand, their experimental set-up is much simpler.

5. RESULTS

The purpose of these experiments was essentially to test the feasibility of the method for samples of various wettabilities. In this chapter, we present the experimental set-up, the results obtained with sand and sandstone, and discuss the accuracy of the experiment. The interpretations of the curve are detailed in the next chapter.

5.1 Experimental set-up

This simple set-up is suitable only for pressures up to 10 bar and room temperature. A modified version of a Hassler cell will be used for further experiments.

The sand is packed in a plastic core holder, and the sandstone sample is coated with epoxy resin. The length of each sample is around 11 cm and the diameter 3.5 cm. The fluids for washing the outlet faces flow through grooves on the end pieces and the rate is set to 20 cc/h. The maximum rate for the injected fluid is 500 cc/h. The steps are 5, 10, 20, 50, 100, 200, 300, 400 and 500 cc/h. Effluent production is measured in a burette with an accuracy of 0.2 cc. The pore volume is of the order of 50 cc. The dead volumes in the tubings are of the order of 5 cc.

The local saturation is measured by an array of 6 ultrasonic transducers separated by a distance of 1.4 cm and at 2 cm from the ends of the sample. The calibration uses the effluent production either with a linear (for sand) or a parabolic law (sandstone) between relative sound velocity and saturation (Deflandre and Lenormand¹⁰) In addition, the effect of pressure on sound velocity is corrected for sandstone. For this purpose, the pressure effect was studied for the sample saturated with 100% water and 100% oil and a linear law was assumed for any intermediate saturation.

For sand experiments, the pressure is measured near the outlet A (fig. 2), at the same section than the 2nd transducer. That leads to a small pressure when water is injected (this section is then near the exit). For the sandstone sample, we used another pressure tap for negative P_c , near the water injection (5th transducer) to have higher pressures.

5.2 Sand

In order to study the effect of wettability on the capillary pressure, we used mixtures of oil-wet and water-wet sand. The wettability is changed by a silane treatment, a technique which is efficient and stable (Lombard and Lenormand¹¹). The fraction of oil-wet sand is noted f . We used three samples: water-wet ($f=0$), mixed-wet ($f=0.5$) and oil-wet ($f=1$). The sand permeability is around $4.6 D$ ($10^{-12} m^2$).

- **$f=0$** (Figure 4): The cycle starts with a secondary imbibition (there was no measurements during the first drainage), with its positive and negative parts (2 and 3 refereeing to our notation), followed by a negative (4) and positive (5) secondary drainage. Hysteresis on the positive P_c is similar to standard porous plate measurements. The plateau value around 40 mbar agrees with centrifuge measurements. The negative imbibition and drainage take place without any variation of saturation (vertical line).
- **$f=1$** (Figure 5): The cycle consists of a primary drainage (1) followed by an imbibition (2 and 3) and a secondary negative drainage (4). The shape of the P_c curve is symetrical to the one for $f=0$. The plateau is also around 40 mbar (in absolute value). The negative pressures are limited to -60 mbar, because of the position of the pressure tap near the exit.
- **$f=0.5$** (Figure 6): a primary drainage followed by imbibition and secondary negative drainage. There is no vertical part in the curve and the positive and negative parts are more symmetrical.

5.3 Sandstone

We performed two different experiments with the same Berea sample (permeability around 130 mD):

- 1) The P_c curve consists of a first drainage (1) followed by a secondary imbibition with positive P_c (2). Before drainage, the sample is 100% saturated with water (fig. 7, curves 1 and 2),
- 2) The P_c curve consist of a first imbibition (3') followed by a secondary drainage with negative P_c (4). At the beginning, the sample is 100% saturated with oil (fig. 7, curves 3' and 4).

For the positive curve we used the non-linear calibration and the correction for pressure effects. For the negative part, we used directly the effluent production since the response of the 6 transducers was identical (the saturation can be assumed to be uniform along the sample).

The positive part agrees with porous plate determinations on similar samples. However, the maximum flow rate of 500 cc/h is not high enough to reach the irreducible water saturation. This problem could be easily solved by using another pump.

The negative part shows a sharp decrease similar to the water-wet sand. However, there is a production of oil when the pressure is above -5 bars. We will explain this effect in the next chapter.

5.4 Discussion (experimental set-up)

The accuracy of the measurements can be improved by reducing the dead volumes for effluent production and by using a non-linear calibration for all saturation measurements. In addition, for sand experiments, the level of pressure is very low and the errors due to buoyancy and even meniscus in the tubing are important. Therefore, the results presented here should be considered rather qualitative, especially for the saturation values. For instance the difference between the S_{or} for $f=0$ (fig. 4) and S_{wi} for $f=1$ (fig. 5) can be due to the poor accuracy of the measurements. A more accurate set-up has been built and quantitative comparisons with other methods are in progress.

6. INTERPRETATION

It is clear from the results that the shape of the pressure cycle strongly depends on the wettability of the sample. This property is used in the USBM wettability test which measures the ratio of the areas under the negative imbibition 3 and positive secondary drainage 5 are measured (Donalson and Thomas¹²): $WI = \log (A5/A3)$

For the sand experiments, although the second drainages have not been performed for $f=0.5$ and $f=1$, we can estimate the USBM wettability index. Since one of the curves is a vertical line, WI is respectively plus and minus infinity for $f=0$ and $f=1$. For the intermediate case ($f=0.5$), the positive and negative curves are roughly symmetrical and therefore $WI=0$ (the ratio of areas is roughly equal to 1).

We can also interpret the different parts of these P_c curves and explain their relationship with the wettability. We follow the interpretations proposed by Dullien and Fleury¹³ for centrifuge, but with different notations.

- *water-wet samples* ($f=0$ and sandstone)

The positive curve is a standard drainage and imbibition.

At the end of positive imbibition, the non-wetting oil phase is disconnected, otherwise it would continue to flow. During negative P_c measurements, water is flowing through a sample containing oil ganglia. It has been experimentally shown that there is no ganglia displacement below a threshold flow rate, or capillary number (Lenormand and Zarcone¹⁴). For sand, the flow rate is not high enough to displace the trapped ganglia. On the other hand, for sandstone, the production of oil for pressures below -5 bars can be interpreted by this mechanism of ganglia displacement.

During the negative drainage, there is no variation of saturation (vertical straight line). Oil cannot invade the sample because its pressure is below the pressure in water.

- *oil-wet sample* ($f=1$): The case is not exactly symmetrical to the water-wet case because the initial filling fluid is still water. However, the mechanisms of trapping and ganglion displacement are analogous.

During the first displacement, oil is injected at a very low flow rate (drainage) It invades the sample and the water phase gets discontinuous. Any further increase of the oil flow rate cannot displace the water ganglia (perhaps a small fraction above 50 mbar). When the rate of oil injection is reduced, the non-

wetting water phase cannot flow back into the sample and the saturation remains constant (curve 2).

For negative P_c , water can enter the sample only if the pressure in water exceeds the pressure in oil. The threshold value, around 40 mbar (curve 3), has the same absolute value as for $f=0$ (this value is linked to the grain size). The curves 3 and 4 correspond to the standard injection and withdrawal of a non-wetting fluid.

- *mixed wettability*: The curves can be interpreted by assuming the existence of two distinct networks formed by the sand grains of different wettabilities (Lombard and Lenormand¹¹).

The first drainage (curve 1 in fig. 6) presents a first part with low capillary pressure which corresponds to the invasion of the oil-wet network. Then the pressure increases to invade the water-wet network. During positive imbibition (curve 2), the oil phase is displaced from the water-wet network.

For negative pressure, water is invading the oil-wet network during imbibition (3). During drainage (4), water is removed from the oil-wet network.

The pressure measurements are not very accurate. However, it seems that the plateau values are smaller than the 40 mbar observed for $f=0$ and $f=1$. That can be explained by looking at the pore-size mechanisms. Even if the necks or thresholds between three grains have the same shape and size in all the samples, there is a low probability, for $F=0.5$, to have three grains with the same wettability. Therefore, the capillary threshold is smaller when one of the grains is wetted by the injected fluid.

CONCLUSION

We have described a novel technique for measuring the capillary pressure of a sample of any kind of wettability. The first pressure cycles have been obtained for sandstone and three sands of different wettabilities. These preliminary results are in qualitative agreement with results obtained by other methods and they can be interpreted by simple physics of flow in porous media.

This new method presents the following advantages:

- All the different parts of the P_c cycle can be obtained with the same set-up: positive and negative drainages and imbibitions. The porous plate method needs two different set-up to obtain these cycles, and standard centrifuge measures only the positive drainage and negative imbibition.

- There is no need for semi-permeable membranes, which are always difficult to operate with mixed wettability samples.
- The measurements can be performed at high pressure and high temperature,
- Relative permeabilities and resistivity index can be measured on the same set-up, without displacing the sample.

The main drawback is the need for local saturation. We used an ultrasonic method and we are testing electrical conductivity.

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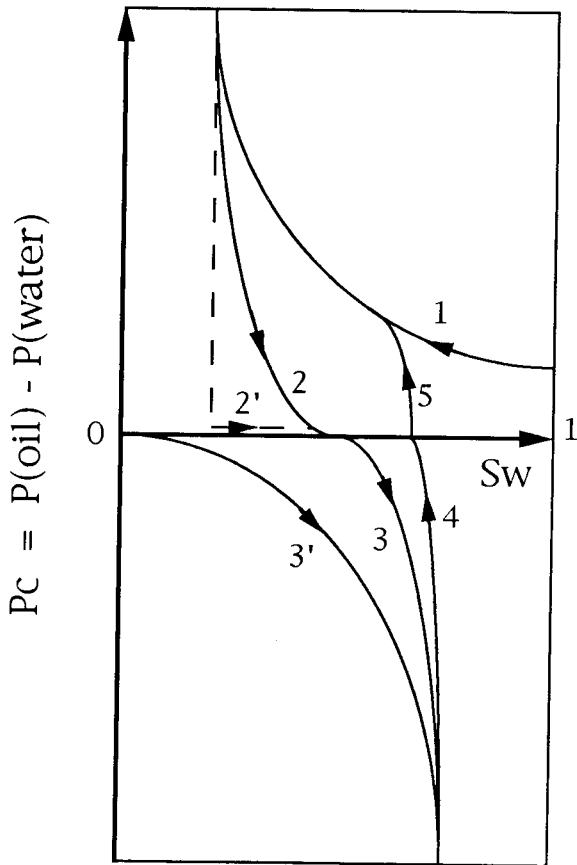


Fig.1. Different types of capillary pressure curves:

- 1) primary drainage,
- 2) secondary imbibition $P_c > 0$
- 3) secondary imbibition $P_c < 0$
- 4) secondary drainage $P_c < 0$
- 5) secondary drainage $P_c > 0$
- 2') spontaneous imbibition
- 3') primary imbibition

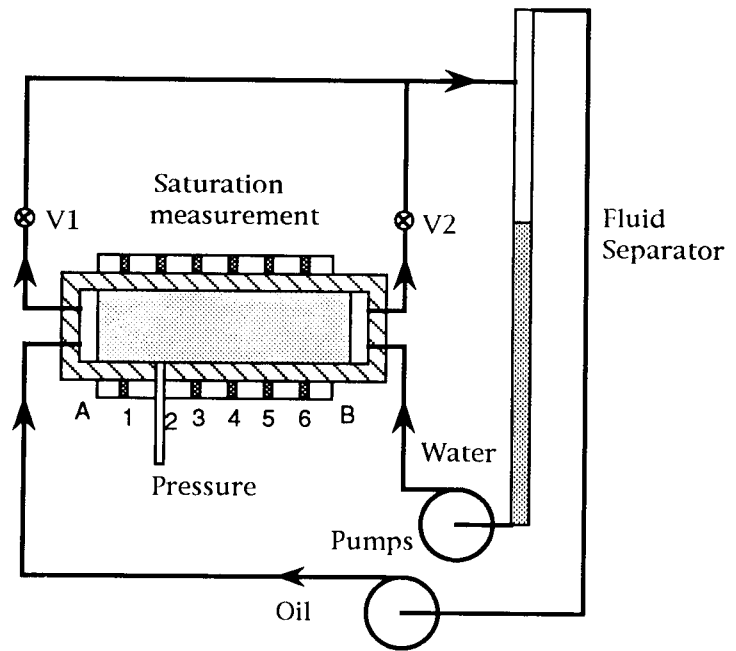


Fig. 2. Principle of the semi-dynamic method for measuring capillary pressures.

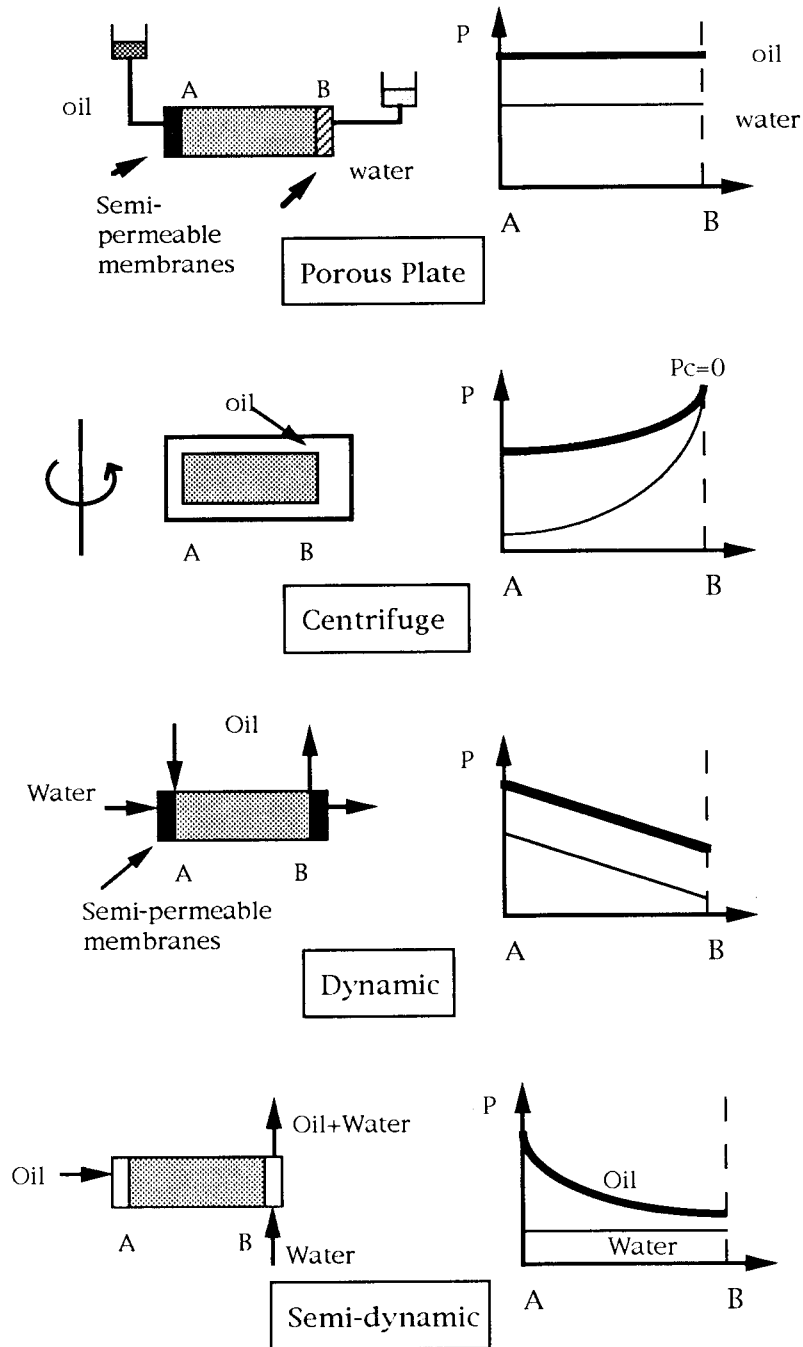


Fig. 3 Various methods for measuring capillary pressure curves: principle of the set-up and pressure diagram.

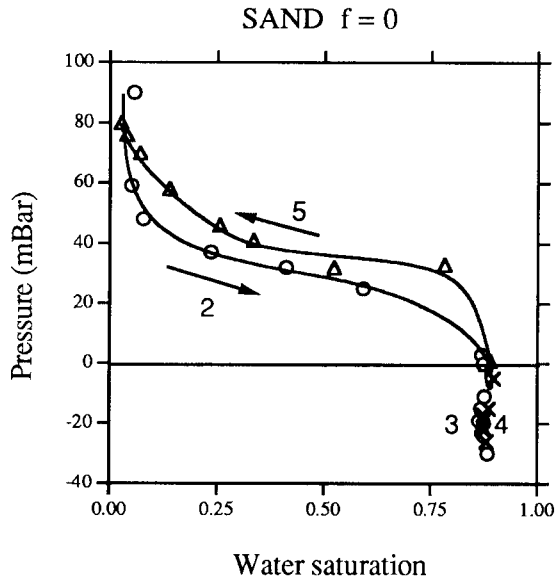


Fig. 4 Capillary pressure cycle for water-wet sand:
 2) and 3) secondary imbibition
 4) secondary drainage $P_c < 0$
 5) secondary drainage $P_c > 0$

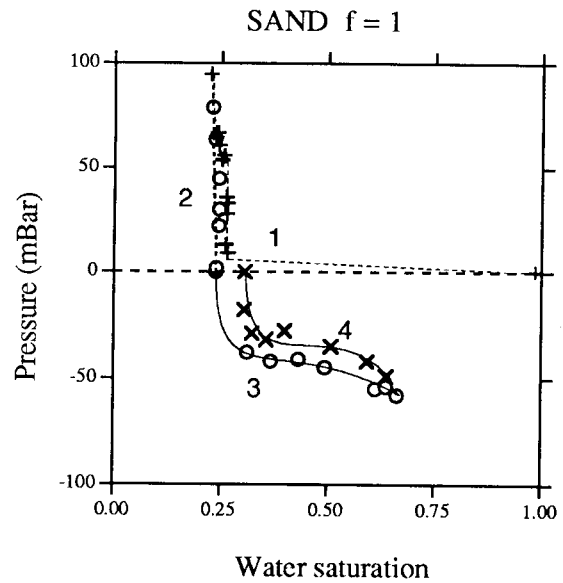


Fig. 5 Capillary pressure cycle for oil-wet sand:
 1) primary drainage
 2) and 3) secondary imbibition
 4) secondary drainage $P_c < 0$

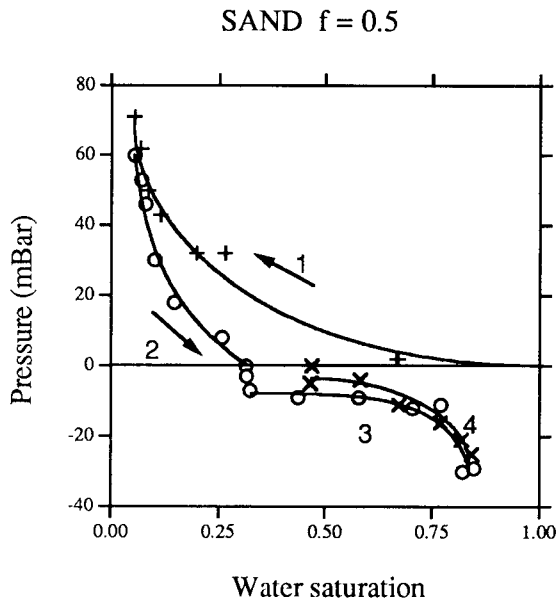


Fig. 6 Capillary pressure cycle for mixed-wettability sand:
 1) primary drainage
 2) and 3) secondary imbibition
 4) secondary drainage $P_c < 0$

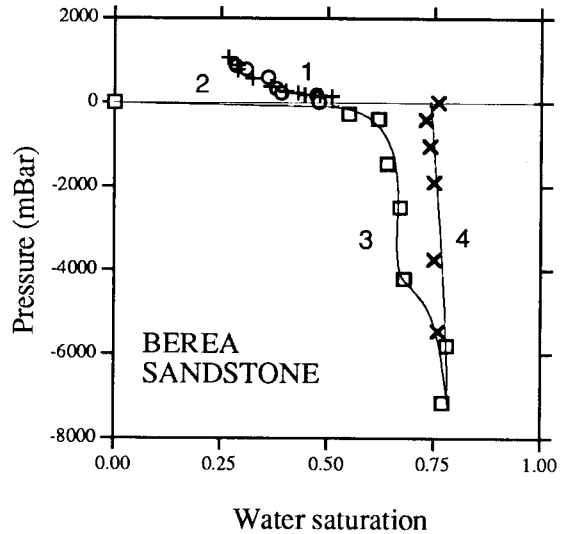


Fig. 7 Capillary pressure cycle for Berea sandstone:
 1) primary drainage
 2) secondary imbibition
 3) primary imbibition
 4) secondary drainage $P_c < 0$

