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Positive imbibition capillary pressure curves using the centrifuge technique.

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Abstract

The standard centrifuge technique allows the determination of drainage and forced imbibition (negative) capillary pressure curves. We present an experimental set-up designed to measure, in addition, the positive imbibition capillary pressure curves for oil-water systems. The principle is to keep the fluid produced during drainage in contact with the sample while decreasing the speed of rotation, to allow that fluid to imbibe spontaneously step by step (positive imbibition). During the experiment, an oil-water interface is constantly maintained at the outlet face of the sample using an external (static) pump connected to the rotating core holder through a rotating fitting. The saturation of the sample is deduced from the volume pumped in and out of the core holder. Although the principle is simple and known, the system required development of the following: (i) a rotating fitting with a leak rate compatible with the porous volume and the duration of the experiment to obtain accurate saturation measurements and ii) a fluid level detector to locate the oil-water interface near the faces of the sample.

We present different tests and experiments demonstrating the different aspects of the device: leak rates and stability of the level position, accuracy of production measurements using a feed-back controller and drainage-imbibition Pc curves on a sandstone plug. Average saturation data obtained during the positive imbibition cycle are interpreted using existing software in a similar way as in drainage. Although technologically challenging, our device is simple to operate and has several practical advantages: (i) samples of length up to 8 cm can be analyzed and therefore low speed/large radius centrifuges (200-3000 rpm) are appropriate in most cases and (ii) the sample is not removed from the core holder during any capillary pressure cycle (from 1st to 2nd drainage).

Introduction

Imbibition capillary pressure curves are important data for reservoir simulations, in particular in fractured reservoirs where they can be critical for formation evaluation. However, they are very difficult to measure in the laboratory and the experiments are usually time consuming. Starting from the irreducible water saturation Swirr, the imbibition curve can be divided into two parts, the spontaneous imbibition or positive part and the forced imbibition or negative part (Figure 1) which is measurable with the standard centrifuge method. In most practical applications, the curvature of the positive part may not have a large impact on the kinetics of two phase flow. It is rather the saturation where

the capillary pressure is zero (Spo, Figure 1) and the forced imbibition curve which are more important. However, the value of Spo plays also an important role in the determination of wettability indices and an accurate determination of Spo is only possible if the water saturation is increased gradually, as in a capillary dominated displacement process. The standard centrifuge method does not provide a good estimation of Spo.

There are essentially three proven methods for measuring positive imbibition curves: the mercury injection method, the porous plate method and the semi-dynamic method. These methods have various domains of validity and various technical advantages and disadvantages, and their discussion is outside of the scope of this paper. For the centrifuge, there have been various attempts to extend the method to the determination of the positive part. Szabo (1974) proposed a method involving ceramic semi-permeable filters in a relatively complex core holder. More recently, Firoozabadi et al. (1988) proposed a method where oil is transferred into the centrifuge core holder through a rotating fitting to maintain at a fixed position the water level, and Torsaeter (1994) modified a standard centrifuge equipped with an optical level detection system to maintain the sample in contact with water. Baldwin and Spinler proposed to measure the saturation profile of a centrifuged sample to deduce the negative and positive capillary pressure curves.

The device described here is similar to the Firoozabadi et al. (1988) method. However, we used a new method to detect oil-water level while centrifuging, which has a high versatility and allows a very simple manipulation of the sample and interpretation of the data. This new method has been presented in a previous paper (Fleury et al., 1998) and applied to the determination of drainage and forced imbibition curves. Beside the positive imbibition curve, our motivation in this work is also strongly driven by the simultaneous determination of Pc and Kr curves during the full imbibition cycle.

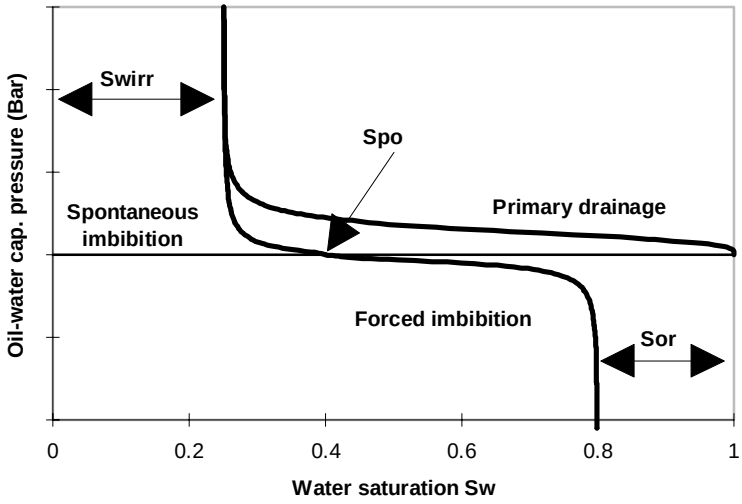

Figure 1: a typical capillary pressure curve for intermediate wet rock sample. With standard centrifuges, the spontaneous imbibition curve and the saturation Spo (where Pc=0) cannot be measured.

Methodology and experimental set-up

The key idea is to keep the fluid produced during a cycle (either oil or water) in contact with the sample while centrifuging so that it can "flow back" into the sample when the imposed pressure is decreasing. In the standard centrifuge method, the produced fluid is collected either at the bottom of the core holder (water during drainage) or at the top (oil during forced imbibition). In the proposed modified centrifuge, the oil-water interface is maintained in contact with the sample near its bottom or top face (larger or smaller radius) depending on the capillary pressure cycle.

For positive values of capillary pressure, the oil-water interface is maintained near the bottom end face of the sample (R=23 cm, Figure 2) and the speed of rotation is increased stepwise for drainage and then decreased stepwise for spontaneous imbibition. For negative values of capillary pressure, the oil-water interface is maintained near the top end face of the sample (smallest radius of rotation) and the speed of rotation is increased stepwise for forced imbibition.

We designed the experimental set-up shown in Figure 2. The Pumping While Centrifuging (PWC) system allows oil to be pumped in and out of the core holder while centrifuging. The excess water is expelled to a water reservoir located in the center of the rotor. A PID system allows the oil-water level to be maintained at a fixed position. When the level is set at the bottom face of the sample, water production is occurring for increasing speeds of rotation (drainage) and the oil-brine level has the tendency to move up; this is detected by the level analyzer and transmitted to the control system; the pump injects into the core holder the volume of oil necessary to keep the level at the pre-set position; the level in the brine reservoir moves up. For decreasing speeds of rotation (spontaneous imbibition), water flows back into the sample and the pump removes from the core holder the volume necessary to keep the position level at the bottom face; the level in the brine reservoir moves down.

Core holders

The core holder (Figure 2) is designed for samples of length up to 8 cm and diameter up to 4 cm. Although long samples will increase experimental time, the advantage of large pore volumes in terms of saturation measurements is more important. If relative permeability measurements are made, the sample is sleeved. The bottom face of the sample is located at a radius R of 23 cm, above a perforated (ceramic) semi-permeable filter of thickness 1 cm. The use of this ceramic is described later in the data analysis section. The oil-water level is always maintained at R=23 cm, except during a limited time between the end of drainage and the start of imbibition.

The level detector located on the side of the sample is a capacitance based system described in details in Fleury et al. (1998). The measured capacitance is proportional to the height of the oil-water interface in the core holder and this system proved to be of high

precision and reliability. At a given speed, we can detect a variation of height of 0.2 mm. For the lowest to the highest speed of rotation, there is a small drift of about 0.5 mm of the detector due to various mechanical factors.

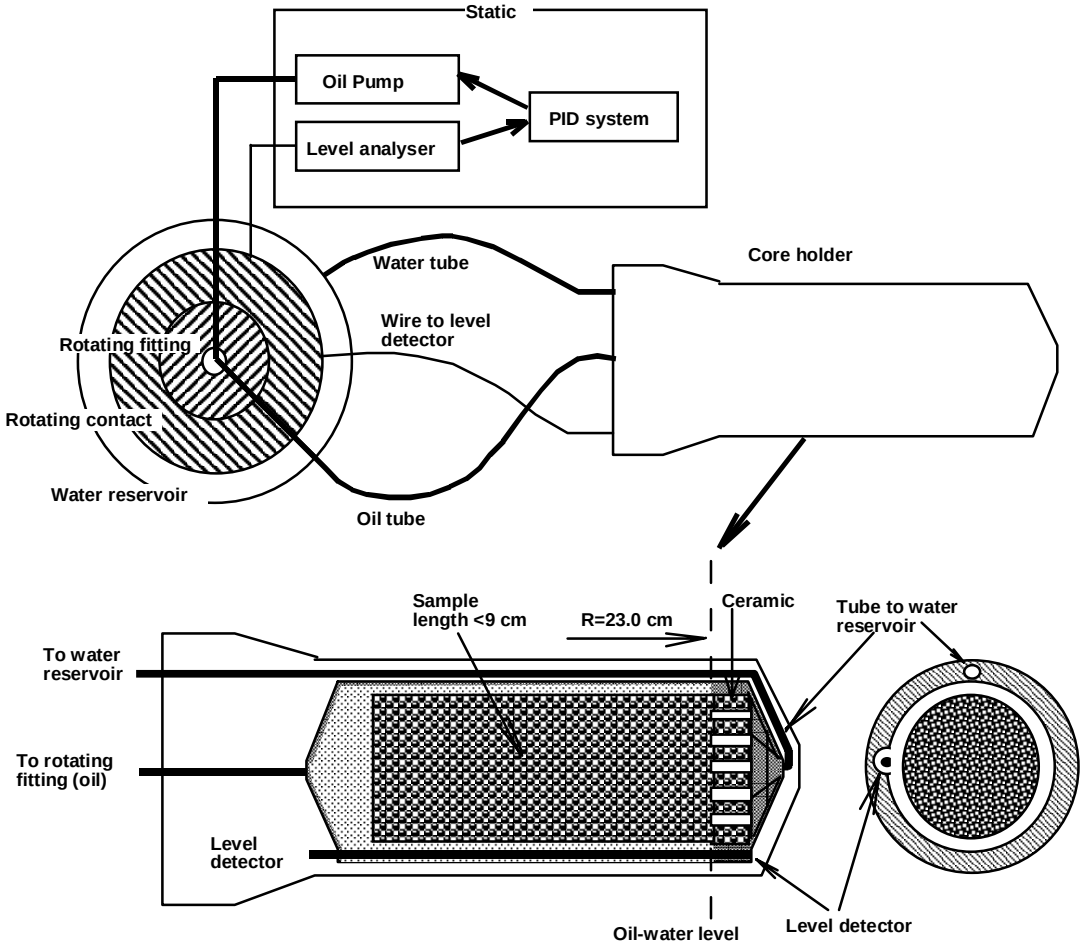

Figure 2: Schematic view of the Pumping While Centrifuging (PWC) system. A water reservoir, a rotating contact and a rotating fitting are positioned at the center of the centrifuge rotor. The core holder is always horizontal to allow tubes to be connected to the rotating fitting (oil), and to the water reservoir. The detector located on the side of the sample can follow the oil-water level from the top to the bottom.

Average saturation measurements

The average saturation of the sample is deduced from the volume of oil pumped in or out of the core holder while centrifuging. The motion of the oil-water level is permanently compensated by the pump and this leads to up and down motions of the oil-water level. Using the same detector in a tube with a comparable volume sensitivity (1cc/cm, Figure 3 left), we tested the accuracy of the volume measurements by simulating a constant production from the sample of 10cc/hr, which is the order of magnitude of the flow rates encountered in centrifuge experiments. For this purpose, a pump (1, Figure 3

left) injects oil into the tube at constant flow rate and the precision pump 2 removes the same amount, based on the level detector signal (the control system is a standard PID circuit). When plotting the difference between the known injected volume and the measured volume (Figure 3), we observe oscillations of about 0.02 cc which characterize the maximum errors of volume measurements. While centrifuging, measurement conditions are more favorable because the large acceleration (from 20 to 1000g) will flatten the meniscus around the detector and yield more sensitivity.

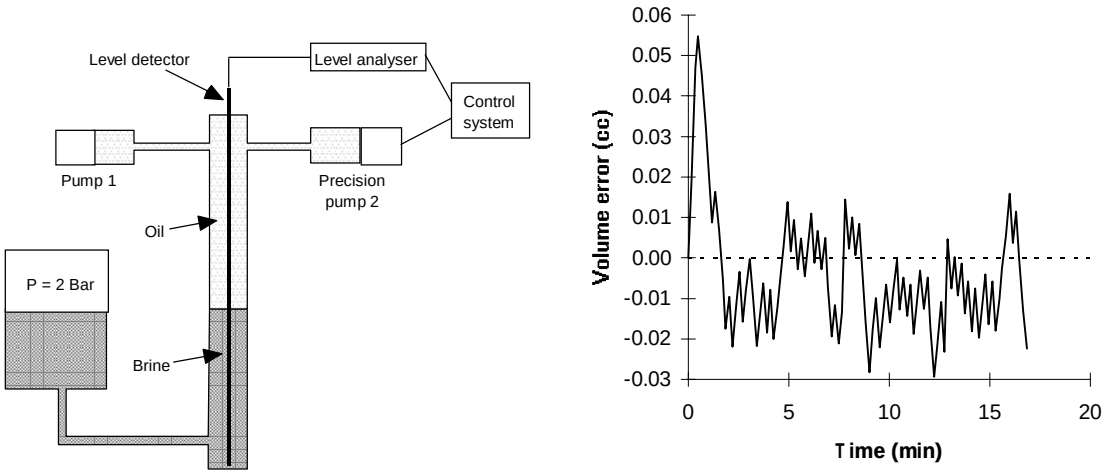

Figure 3 : Test of the volume measurements. The metering pump 2 compensates for the volume injected by pump 1 (left) through the detection of the level and a PID control system. The difference between the known injected volume and the measured volume is very small (right). The flow rate was 10 cc/h.

For optimum volume measurement conditions, the surface area around the sample must be small. In different terms, the detector has a height resolution of 0.2 mm and this height should represent a reasonably small volume. When a sample is put in the core holder, we measured (while centrifuging) a surface of 0.09 cc / mm. Hence, the volume sensitivity is consistent with the accuracy of the PID system described above. This implies a constraint on the diameter of the sample and sleeve.

Position measurements

The detector must provide a precise measurement of the radius of rotation and therefore must be calibrated. A simple experiment was performed using a metallic cylinder with internal serrations at 1 cm intervals as shown in Figure 4. At a speed of 1000 rpm, oil was injected in the core holder at a constant rate and the level detector signal recorded. From the variation of slope (Figure 4), we determined precisely the detector signal corresponding to various radii of rotation, especially the positions of the lower and upper faces of the sample. Repeating this procedure for different speeds of rotation, we verified that the drift of the detector is very small.

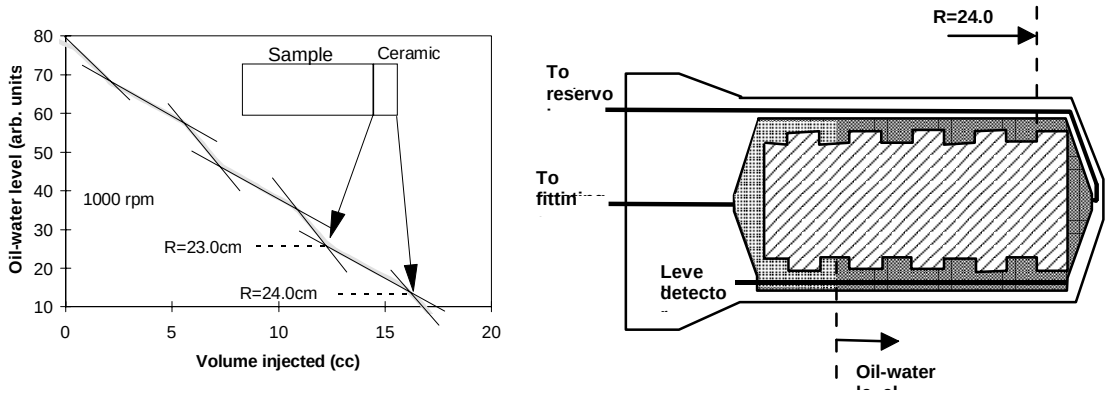

Figure 4 : Calibration of the level detector while rotating. Oil is injected at a constant flow rate and the level measured (left). The variable diameter of the test piece (right) allows a precise determination of the oil-water level in the core holder using the variation of slope.

Leak tests

The rotating fitting is a key component of the system and the accuracy of saturation measurements depends critically on the leak rate. We tested a large number of industrial products. Although it may not be possible to achieve a complete seal, very low leak rate compatible with the experiment (e.g. 1% of the pore volume during the experimental time) can be obtained by choosing rotating fittings that are over-designed by an order of magnitude. In our case, we used a product from TECMECA (France) designed for 150 Bar and 15000 rpm despite the fact that the speed of rotation in our experiments is less than 3500 rpm and the pressure is limited to about 7 Bar. Conca et al. (1992) also used successfully a rotating fitting to inject a fluid while centrifuging, but in a hydrogeology context of air-water systems to determine water relative permeability.

The experimental set-up must also be designed such as to avoid elevated pressure at the rotating fitting. This is achieved by setting the water reservoir at an appropriate radius of rotation. The pressure at the rotating fitting at the center is given by:

$$P_{rf} = \frac{1}{2}\omega^2(\Delta\rho R_{ow}^2 - \rho_w R_w^2) \qquad (1)$$

where R_{ow} is the radius of rotation of the oil-water level, R_w is the radius of rotation of the water in the water reservoir, $\Delta\rho$ is the positive density difference between oil and water, ρ_w is the water density, ω is the speed of rotation. In principle, P_{rf} can be set close to zero at any speed (for a given set of fluids). However, a pressure too low will not be appropriate for regulation of the oil-water level. In our device, R_w=3.5 cm, R_{ow}=23 cm and therefore, the pressure at 3000 rpm is about 7 Bar for a dodecane/brine system.

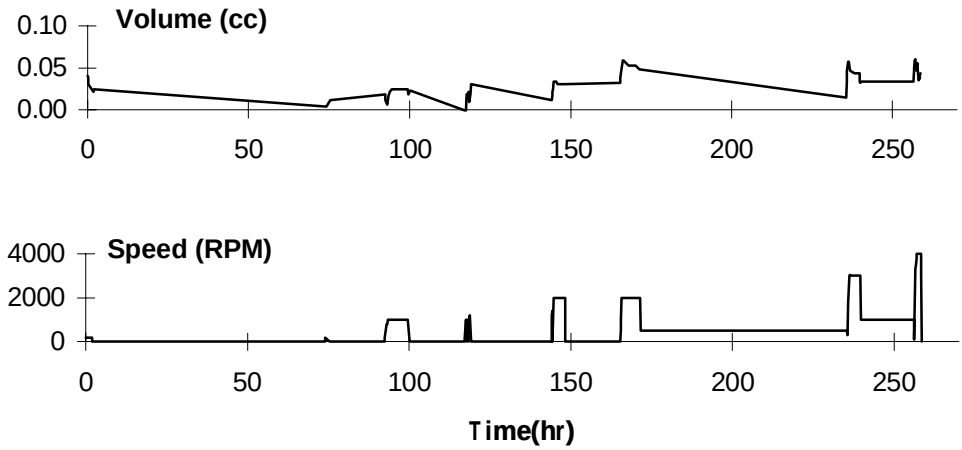

Figure 5 : Leak tests of the rotating fitting used in the experiments, with and without rotation. The imposed pressure was 5 Bar. The records of volume were taken manually and there are a limited number of data points. The fluctuations are mainly due to temperature variations in the laboratory.

A long term test of the rotating fitting has been performed with and without rotation (Figure 5). The leak rate is difficult to determine and measurements are sensitive to small temperature fluctuations. There is a probable rise of temperature of the oil inside the rotating fitting but this is limited to a small volume near the spinning surfaces of the joint. The leak of the fitting can also, in principle be tested during drainage or imbibition experiments but in practice, a stable saturation is sometimes difficult to obtain on samples of intermediate wettability (this also occurs in standard centrifuges).

Experimental procedure

The system is planned to operate as follows:
- a sample saturated with brine is installed in the core holder; all circuits are filled with brine up to the rotating fitting (where brine is never introduced); the centrifuge is set to its minimum speed of rotation (200 rpm)
- oil is introduced in the core holder while centrifuging using the pump; the level is set close to the outer face of the sample (pre-set position) using the calibrated signal from the level detector;
- a drainage is performed by increasing the speed of rotation stepwise
- at the maximum speed of rotation (3000 rpm), the level is moved 1 cm below the bottom face to obtain a uniform saturation profile in the sample (see data analysis section); when stabilization is obtained, the level is set back to the bottom face of the sample
- a spontaneous imbibition is performed by decreasing the speed of rotation stepwise
- at the minimum speed of rotation, the oil-water is set near the top face of the sample to begin the forced imbibition cycle. There is no need to remove the sample from the core holder.

The data recorded during the experiment are:
- the volume pumped in or out of the core holder yielding the average saturation of the sample
- the speed of rotation yielding the face capillary pressure
- the level position and the pressure at the rotating fitting (for control)
- the temperature in and out of the centrifuge (for control)

Imbibition data analysis

The spontaneous imbibition capillary pressure curve can be calculated from knowledge of the production at equilibrium at each step of rotation. For any cycle, existing softwares (e.g. Forbes, 1991) can be used to calculate the capillary pressure curve. For positive Pc values, the capillary pressure at the inlet face (R_{min}) is:

$$Pc(R_{\min}) = \frac{1}{2}\Delta\rho\omega^2(R_{\max}^2 - R_{\min}^2) \qquad \text{Pc}(R_{max}) = 0 \qquad (2)$$

Equation 2 assumes that the capillary pressure is zero at the outlet face (R_{max}), as for drainage. In our experimental set-up, this is strictly imposed by the water level. The positive imbibition curve can be calculated the same way as the first drainage, although the physical processes involved are very different: the boundary conditions are the same and equation (2) can be applied to both cases. Identically for the calculation of the forced imbibition and negative second drainage curves, we can use the numerical treatment applied to negative imbibition Pc curves.

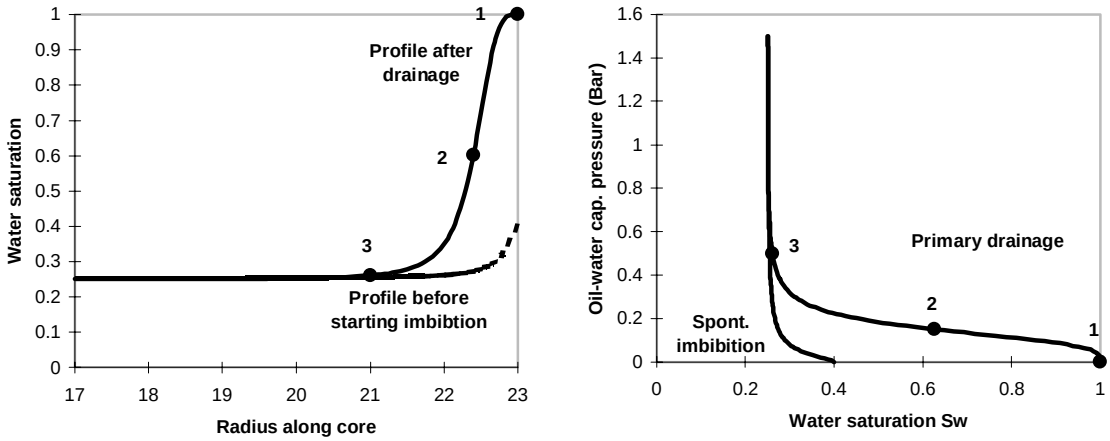

Figure 6 : Schematic showing the saturation profile obtained at the end of drainage and the saturation profile needed before the start of imbibition.

At the end of the first drainage, there is, however a difficulty related to the saturation profile in the sample. For example, location 2 (Figure 6) will follow a local hysteresis curve that cannot be determined from the available data, if the spontaneous imbibition is started directly after drainage. Equivalently, if the spontaneous Pc curve is

back calculated the same way as a drainage curve, one must end with the imbibition saturation profile (Figure 6), and not the drainage saturation profile. The imbibition profile can be obtained by using a ceramic end-piece support (Figure 2) which will decrease the outlet face saturation of the sample. At the end of drainage, the boundary condition Pc=0 is moved to a larger radius (from 23 to 24 cm) and therefore, a pressure of about 500 mBar is imposed at the outlet face of the sample. The oil entry pressure of the ceramic being about 3 Bar, there is no oil flowing into the ceramic. This ceramic is also perforated to avoid reduction in flow rate during drainage. Schematically, the saturation at the outlet face will correspond to point 3 on the drainage Pc curve (Figure 6), i.e. a quasi uniform profile. When the level is set back to 23 cm, the outlet saturation will shift to the appropriate value (Spo). An example of data recorded during this procedure is shown in Figure 7.

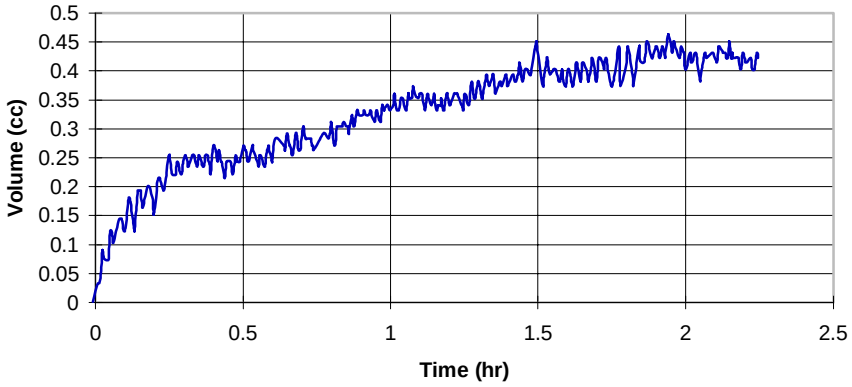

Figure 7 : An additional production is observed when the oil-water level is moved at the base of the ceramic 1 cm below the face of the sample. By capillary contact, the outlet face saturation will be close to Swirr and a new capillary equilibrium is reached after 2 hours.

Example of measurements

We show an example of drainage and spontaneous imbibition on an outcrop Vosges sandstone (K=610 mD, ϕ=23%, L=5.9 cm, D=3.92 cm, PV=13.0 cc). The equilibrium saturations are reported in Figure 8. Each step had a duration of 16 hours. At the end of drainage, the water level was moved 1 cm below the outer face of the sample to drain the water-foot (Figure 7). This lead to variation of saturation of 3% (Figure 8, left panel at Pc=1400 mBar). Then, the speed of rotation was decreased stepwise up to 200 rpm (the lowest speed programmable on our machine, Pc=+15 mBar) and a saturation of 48 % was reached. Then the level was set to the inlet face of the sample while still spinning at 200 rpm (Rmin, Pc=-15mBar) and we observed an additional increase of 4 % to give 52%. When the speed was increased again (forced imbibition cycle), there was again an increase of saturation of 2 % at Pc=-126mBar (600 rpm) and nothing below that value, as expected for a water wet sample. The final water saturation reached was 54% and the saturation measured by mass balance was 55 %, in good agreement.

In the general case of samples of intermediate wettability, zero capillary pressure cannot be approached smoothly (such an approach may not be possible) and the saturation Spo is determined by interpolation between the +15 and -15 mBar capillary pressure values.

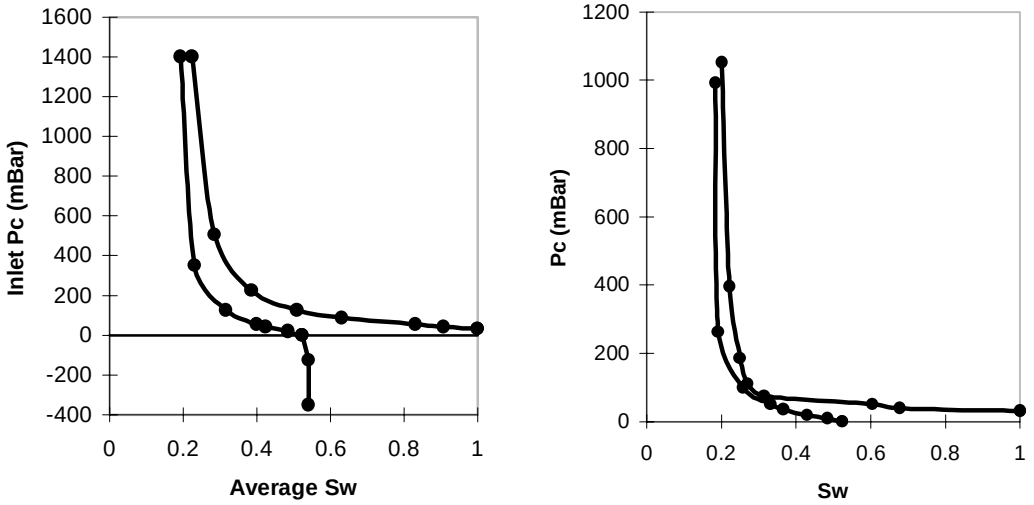

Figure 8: Drainage imbibition capillary pressure curves from equilibrium saturation data. Left: measured average saturation vs imposed inlet pressure; the shift at Pc=1400 mBar is obtained at the end of drainage. Right: drainage-imbibition capillary pressure curves when calculated from average saturation data.

From the average saturation data, the drainage and imbibition were calculated separately using the method of Forbes (1991). The curves give two independent values of irreducible water saturation. From the drainage curve Swirr=20% and from the imbibition curve Swirr=18.5%. These results are in good agreement when considering the accuracy of the Pc curve calculation (this accuracy is strongly dependent on the number of steps). The small difference between the drainage and imbibition curves (Figure 8, left panel) above 200 mBar is therefore not significant.

Conclusions

A new centrifuge experimental set-up is presented. It is designed to measure, in addition to drainage and forced imbibition capillary pressure curves, the positive or spontaneous imbibition capillary pressure curve. All the cycles are performed without stopping the centrifuge and the sample does not need to be manipulated.
The principle is to maintain the fluid produced during a cycle in contact with the sample, allowing that fluid to flow in and out of the sample according the capillary pressure curve. The system required essentially to development and testing of two challenging technologies: (i) a rotating fitting with sufficiently small leak rate with respect to the porous volume and the duration of the experiment to obtain accurate saturation measurements (smaller than 1 % of the porous volume during the experimental time) and ii) a fluid level detector to locate the oil-water interface near the faces of the sample. The tests and examples shown indicate consistent results for a water-wet sample.

This device has large potential for simultaneous determination of capillary pressure and relative permeability on reasonably long samples (8 cm) during the full imbibition process. Another attractive use is a fast wettability index determination based on the Swirr, Spo and Sor saturation value, using single step centrifuging combined with slow acceleration or deceleration.

Acknowledgments

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