FULL IMBIBITION CAPILLARY PRESSURE MEASUREMENTS ON PRESERVED SAMPLES USING THE MICROPORE MEMBRANE TECHNIQUE.

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Abstract

The determination of the full imbibition capillary pressure curve is essential to evaluate the potential of water flooding of reservoirs that are not strongly water wet. We show how the positive and negative parts of the imbibition capillary pressure curve can be measured using preserved samples without an ageing procedure. The micropore membrane technique (CAPWET) is used on thin samples extracted from a full size reservoir core. A simple procedure is performed to resaturate the sample after drilling in the laboratory.

Drainage and imbibition capillary pressure experiments were performed on three samples from the same core using both refined and crude oil. The experiments were conducted by imposing successive pressure steps across the sample and the two water-wet and oil-wet membranes. Cumulative volume of water expelled or entering the sample is measured using a capacitive based detector. Due to the oil-wetting nature of the porous medium, capillary equilibrium is achieved only over very long periods of time. Equilibrium criterion based on a maximum flowrate are inadequate because the volume resolution (<0.01 cc) required is very small.

Observations from this work suggest that true equilibrium at each pressure step is required to reach a representative residual oil saturation (Sor). It is shown that production data can be fitted with exponential functions leading to a characteristic time and the asymptotic production for each pressure step. This leads to a simple criterion to find the experimental time required to insure capillary equilibrium. A lack of equilibrium can lead to an overestimation of the residual oil saturation by as much as 6 saturation units.

1. Introduction

Knowledge of the water-oil capillary pressure saturation relationship is necessary for many reservoir engineering tasks. In particular, the imbibition curve is used to calculate oil-in-place and to model oil displacement either by free water imbibition and /or water injection. Capillary properties of core samples are difficult to measure and the experiments are usually time consuming. In recent years, progress has been made in restored state experiment by using micropore membranes (Jennings et al., 1985, Hammervold et al. 1992, Longeron et al. 1995). The experiments can be about ten times faster and full cycle of capillary pressure can now be obtained in a reasonable time. In parallel, there has been also considerable progress in coring techniques (Pallatt et al., 1991). However, the problem of having representative wettability properties during the laboratory measurements is still somewhat empirical and there is no clear evidence of the best procedure between restoration and preservation.

If low invasion coring techniques are used (Pallatt et al., 1991), preserved samples do have the advantage of having representative wettabiity properties. They are used in general to estimate initial water saturation and for flooding experiments. To our knowledge, there has been no attempt to measure capillary properties directly on preserved samples without cleaning or ageing procedure. When first drainage curves are not needed, full imbibition curves can be determined using a simple preparation procedure to remove the gas in the sample and replace the fluids.

2. Fluids and sample preparation

Fluids

Both refined and crude oil were used. We intended initially to use dead crude oil from the field in all experiments. However, available micropore membranes (water wet only) used in capillary pressure experiments cannot be used at temperatures higher than 50°C. Therefore, when wax is present in the crude (which was the case here), plugging of the membranes can occur. To avoid this problem, the crude oil has been filtered through a 0.6 µm Millipore membrane at 40 °C before being used for Pc measurements. During this operation, the filter is gradually plugged (after 10 cc approximately) by a wax deposit on the membrane, although the wax-apparent temperature is 34 °C for this oil.

The measured crude oil/brine interfacial tension (IFT) at 40°C was 15.9 mN/m. When experiments are performed with refined oil, we used Soltrol 130 (IFT=34.9 mN/m). Formation brine has been synthetized to reproduce the main characteristics of the formation brine (essentially NaCl: 22.0 g/l and $CaCl_2$, $6H_2O$: 1.5 g/l).

Sample preparation

Capillary pressures were measured on plugs drilled out of a preserved core from a sandstone reservoir located in the North Sea (Rannoch formation). The drilling direction is perpendicular to the axis of the core. The purpose of the preparation is to remove the gas and replace oil and brine in the preserved sample by successive water and oil flooding. An advantage of this procedure is that neither cleaning nor ageing process is required but it assumes that the field preserving technique is performed properly. A drawback is that additional drainage-imbibition cycles are performed on the sample and there is a possible cycle hysteresis effect.

The following operations have been performed:

- after drilling at diameter 39.5 mm, sample length set to 24.0 mm (thus, two companions plugs can extracted at the same height for standard full size cores)
- sample mounted in cell (without membranes, see next section)
- injection of crude oil (> 20 PV) at T=70°C with a back pressure of 10 Bar during several hours, measurement of Kro at three flow rates (saturation unknown)
- injection of refined oil (> 20 PV) at T=70°C, measurement of Kro at three flow rates. When the experiment is performed with crude oil, this step is skipped.
- temperature decreased to T=40°C and injection of brine (> 20 PV), measurement of Krw at three flow rates (saturation unknown)

Due to the small size of the sample and the inescapable dead volume of the cell, it is practically impossible to measure the change in saturation during the preparation. Pore volumes are measured at the end of the capillary pressure experiments after standard cleaning procedure.

3. Capillary pressure measurements.

The main objective is to measure the full imbibition capillary pressure curve (positive and negative parts) to evaluate the performance of a capillary dominated waterflooding process. A drainage is used to reach a representative irreducible water saturation by a quasi-static displacement but the precise determination of the drainage curve is not of primary importance. The sample preparation described above imposes start of the drainage process not at 100 % brine saturation but at an unknown saturation close to 1-Sor. However, during drainage and imbibition capillary pressure experiments, the variations in saturation are measured as precisely as possible. Finally, the end point saturation Sor (at the end of the forced imbibition) is determined by Karl-Fisher titration following the procedure described by Tonstad et al. (1990). The Swi value is back calculated and therefore depends on the accuracy of the production measurement.

3.1. Experimental set-up

The experimental set-up is a computer controlled automated apparatus for measuring capillary pressure curves using micropore membranes (CAPWET, Longeron et al., Fig. 1). During the experiments, the pressure in the brine is constant and the pressure in oil is varied above or below to impose a given pressure difference. Both pressures are regulated with a accuracy of ±1 mBar. The precision of the pressure difference is important at small negative capillary pressure and for intermediate wettability conditions because saturation change can be important within 10 mBar. Mainly, pressure steps and stability criterion are programmed by the user before the experiments. Typical stability criterion for saturation changes vary from 0.02 cc/4 hr to 0.02 cc/12 hr, meaning that the system will change automatically the pressure difference if water production is not larger than 0.02cc during 4 or 12 hours (for a porosity of 30%, 0.02cc corresponds to a saturation of 0.2%).

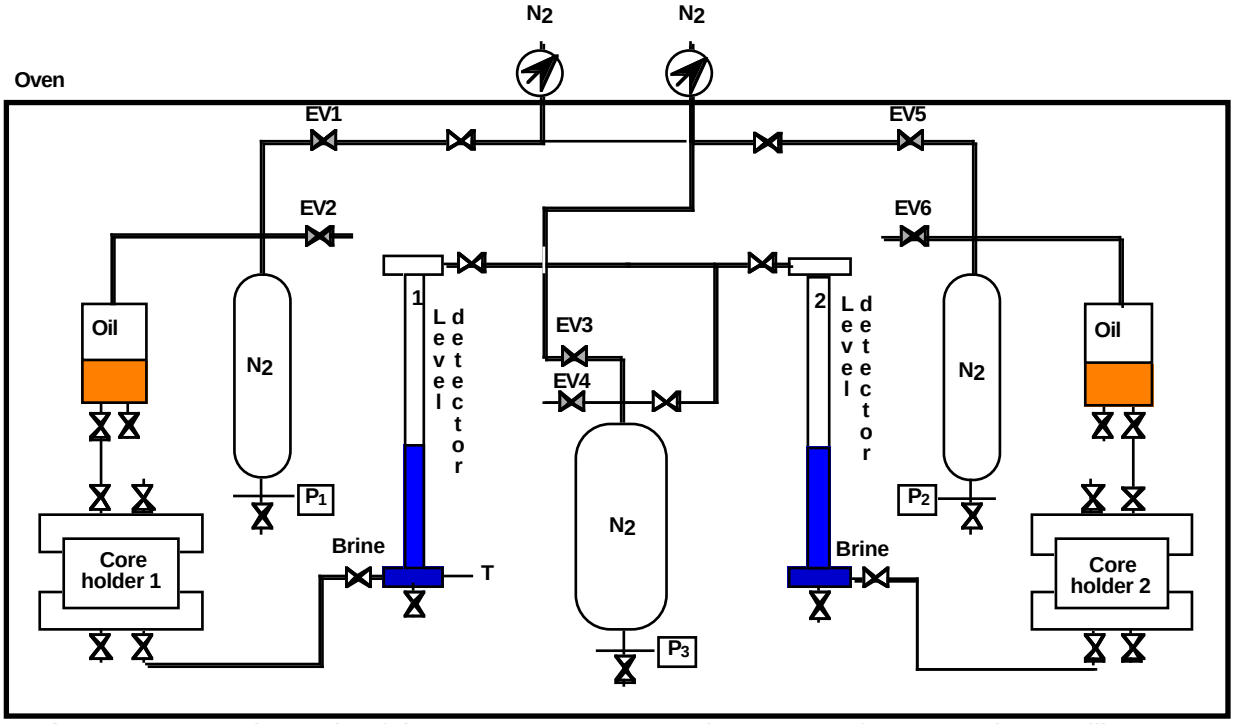


Figure 1: Schematic of the CAPWET automated apparatus for measuring capillary pressure curve.

The standard procedure is as follow:
- sample mounted in cell (fig. 2) with a water-wet and an oil-wet membrane,
- cell mounted in oven, temperature set to the desired temperature and stabilised (24h),
- the hermeticity of the system under pressure is tested during 24 hr,
- the pressure on water is always maintained at 2 Bar and the pressure on oil is varied above or below 2 Bar.

The average pressure of 2 Bar is not a limitation of the apparatus and can be increased if needed up to 6 Bar.

3.2. Core holder

The critical component of the system is the core holder. For preserved samples, the metallic coating used in an early design (Longeron et al., 1995) cannot be used because the sample is heated during the preparation procedure (about 80°C during several hours). More over, the radial stress on the sample is unknown and resistivity measurements are not possible. A new cell was therefore designed (Fig. 2). Lateral sealing of the sample is provided by a Viton sleeve with a confining pressure of 30 Bar (maximum 80 Bar). The

sleeve provides also the seal between the membranes and the endcaps. During preparation, endcaps can be flushed with the appropriate fluid using spirals to avoid dead volume problems. This is particularly important since pore volume are small (typically 8 cc for Ø = 30%). Mechanical constraint on the membranes on both sides is adjusted using the endcaps. The new generation will include radial resistivity measurements (Fleury and Longeron, 1996).

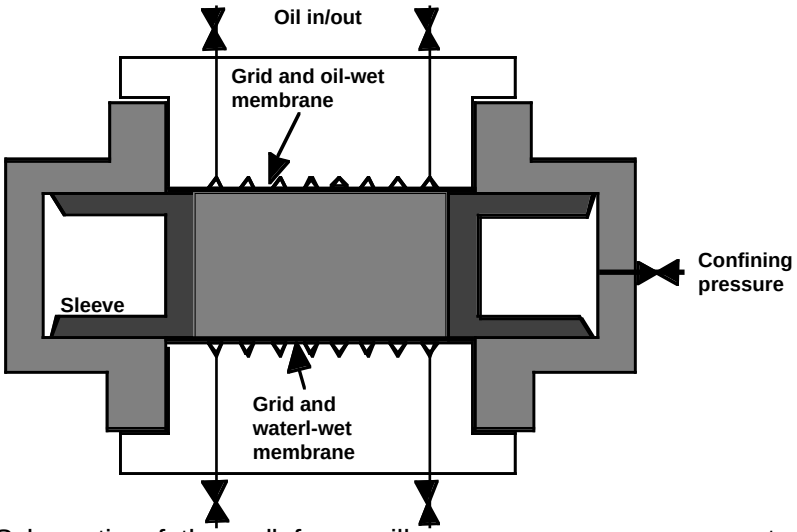


Figure 2: Schematic of the cell for capillary pressure measurements. A confining pressure of 30 Bar was applied. Endcaps can be removed independently to replace/remove membranes. The sample must have a diameter of 39.5 ± 0.3 mm and a length of 24.5 ± 0.5 mm.

3.3. Production measurements

Production is measured using a low cost capacitance based detector able to determine the amount of brine going in or out of the cell (Fig. 1). The system is well adapted to measure slowly varying production. It is basically sensitive to the height of a conductive fluid (brine) in which the sensor is immersed (Fig. 3). A non conductive fluid (air or oil) can be present above. In practice, we measure a capacity C proportional to the height h immersed in brine:

(1) $$C = 2\pi\varepsilon_0\varepsilon_r h / \ln\left(\frac{R+e}{R}\right) \approx 2\pi\varepsilon_0\varepsilon_r \frac{R}{e} h \quad \text{(assuming e « R)}$$

where $\varepsilon_r R/e$ determines the sensitivity of the device which is typically of the order of 50pF/cm. Measurements are performed at 30 kHz and do not depend on the salinity of the brine (for usual NaCl salinity > 10 g/l). They are weakly dependant on temperature, if the coating is properly chosen. Linear calibration (capacity-volume curve) is usually found over the range considered (10 cc/10cm) but this is not necessary since tabulated values are used to convert capacity into volume.

The limitations of the system (resolution, speed) are associated with the properties of the air/brine meniscus around the rod (Fig. 3). The accuracy of the separator is clearly seen in Figure 4. A known volume of brine (1 cc) is introduced at a constant low flow rate into the system. The volume error plotted in Figure 4 is the difference between the calibrated volume from the level detector and the volume pumped into the separator. The resolution is about 0.01 cc. Similar measurements show that the precision is ±0.05 cc (about 0.5 %PV). Moreover, the stability of the measurements over long periods of time (2 months) is better than 0.5% of the full range. However, a calibration is performed before and after the experiments to check any drift of the detector (caused by the microporosity of the coating for example).

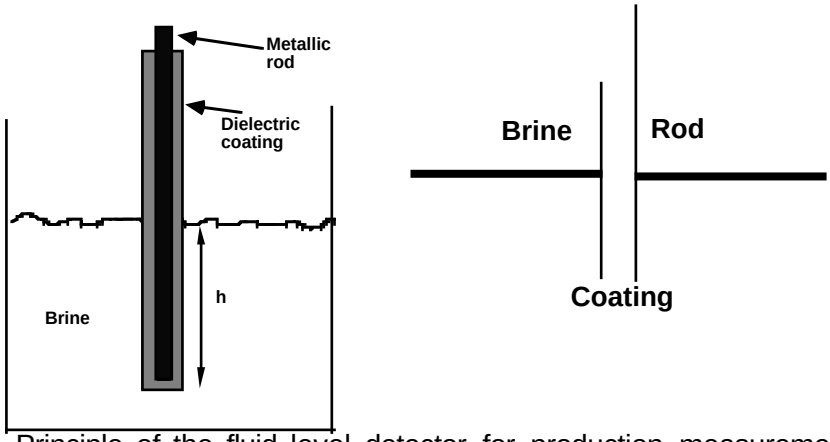


Figure 3: Principle of the fluid level detector for production measurements (left) and simplified equivalent circuit (right). Measurements are independant on brine salinity.

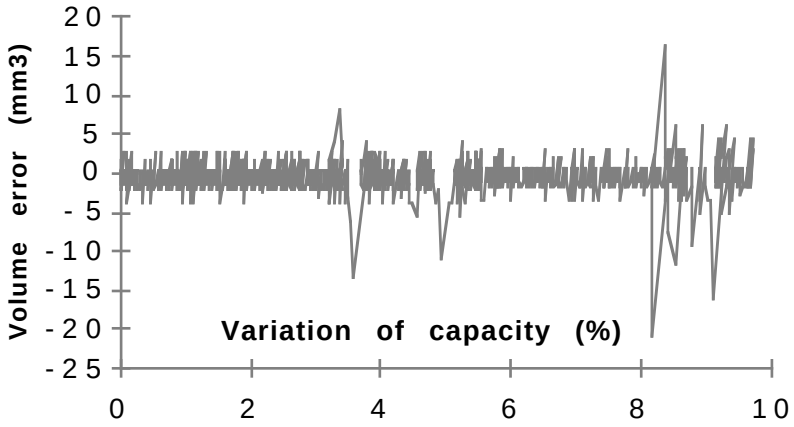


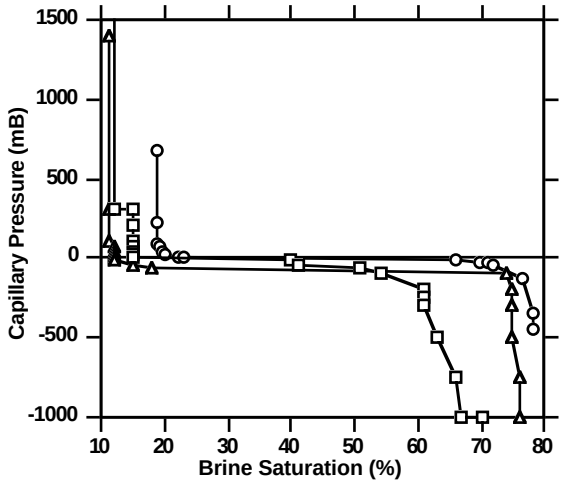
Figure 4: Test of the fluid level detector at low flow rate (0.42 cc/h). Fluctuations indicate the actual resolution of the detector. The variation of capacity is a normalized fraction of the lowest and highest measured value.

4. Results

Measurements were performed on three plugs drilled from the same preserved core. X-Ray density measurements do not indicate specific heterogeneities. CT images indicate however millimetre scale laminated structure parallel to the flow direction. The measured imbibition capillary pressure curves are shown in Figure 5. The positive parts are nearly vertical and most of the saturation variation is obtained during one step at negative capillary pressure (about -10 mBar for plug 1 and 3 and -80 mBar for plug 2, Fig. 5 and 6, plug 3 experiment is scaled to the crude oil/brine system taking into account the IFT ratio). This is a signature of intermediate wetting properties and has strong implications for the performance of the water flooding process. Different fluid systems were used between plugs 2 and 3 and we suspect that the difference between the two plateau values is due to a plugging of either the membrane or the sample when waxy crude oil is used.

There is a unexplained strong difference for the value of irreducible water saturation Swi between plugs 2 and 3 (11 and 19 %, Fig. 5) and it is not known if this is due to the use of refined oil for plug 3. Nevertheless, the same residual oil saturation was obtained for plugs 2 and 3 (1-Sor = 77 $\pm$ 1 %, Fig. 5) after forced imbibition. For plug 1, the curve is shifted to small saturation and the residual oil saturation is much lower due to an inadequate equilibrium criterion (EC=0.04cc/4hr). However, at the end of the experiment (-1000 mBar),

we expected to have a better Sor estimate by changing the EC value to 0.02cc/12hr (horizontal line in Fig. 5). Despite this change, there is still a difference of about 6 % between plug 1 and 2 (plugs 1 and 2 are companion plugs drilled at the same location). Due to the small oil relative permeability at high water saturation, production is very slow and a representative residual oil saturation is not obtained. We conclude that an inadequate equilibrium criterion can lead to an underestimation of the final saturation (1-Sor).



Plug	Ø (%)	K (mD)	Swi (%)	1-Sor (%)
1	29.8	331	12	70
2	31.5	287	11	76
3	31.4	329	19	78

Figure 5: Full imbibition curves for 3 plugs extracted from the same preserved core. The plug 1 curve (square) was obtained under non-equilibrium conditions. For this plug, various tests were performed to test the reliability of the measurements. Curve for plug 2: triangle. Curve for plug 3 (circle) is rescaled to crude oil/brine IFT value.

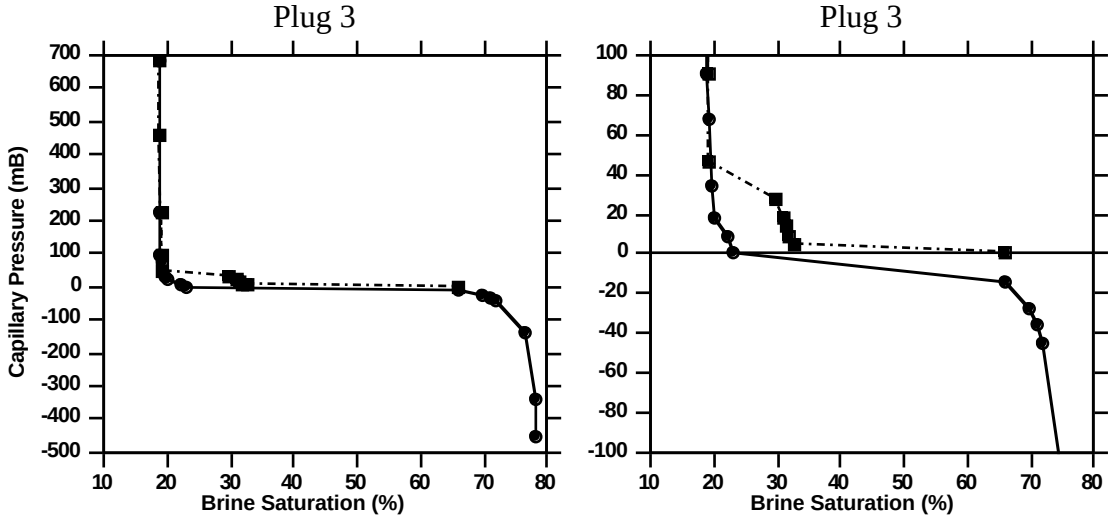


Figure 6: Drainage (square) and imbibition (circle) capillary pressure curve for plug 3 using refined oil (left and right with different axis scaling). The initial water saturation (drainage curve) is obtained after a low flow rate water flooding (sample preparation).

Positive imbibition curves require a good capillary contact between the sample and the water-wet semi-permeable filter. From this point of view, membranes are more adequate than porous ceramics. If vertical positive imbibition curves are signatures of intermediate wetting conditions, they can also be due to artefact of measurements. Indeed, if the capillary contact between the plug and the membrane is weak (or in the worst case absent because a thin layer of oil could prevent any water from entering the sample), spontaneous imbibition will be very difficult to observe. It is also known that this process is sensitive to

pressure perturbations. For plug 1 at positive pressure (300 mBar), we tested the capillary contact between the plug and membrane by forcing a small amount of brine (3 saturation units, discontinuity on the imbibition curve in Figure 5) into the plug ; then, the pressure is set back to its initial value. Saturation did not change after this procedure and we concluded that the vertical imbibition curve is a property of the sample and is not due to an artefact. Details of the capillary pressure curves (Fig. 6, plug 3) show clearly the wetting properties of the sample. For positive values, a quasi-spontaneous drainage is observed. Then, the curve is similar to a first drainage with a pseudo entry pressure. The positive imbibition curve is not vertical at small positive pressure, indicating that the capillary contact is effective.

5. Equilibrium criterion

Optimisation of the experiments in term of duration is important. For each pressure step, a capillary equilibrium must be reached and a compromise between speed and accuracy must be found. We used in the experiments a criterion based on a maximum flow rate: a new pressure step will be started when the (instantaneous) measured flow rate is less than a given value. Duration and equilibrium criterion (EC) are indicated in Table 1. Our standard value (0.04cc/hr) was insufficient, as shown by the result on plug 1 and the EC was later empirically decreased to 0.02cc/12hr. Because the resolution of the production measurement system is about 0.02cc, the flow rate is estimated during 12 hours (i.e. a new step will be started if there is no production larger than 0.02 cc during 12 hours).

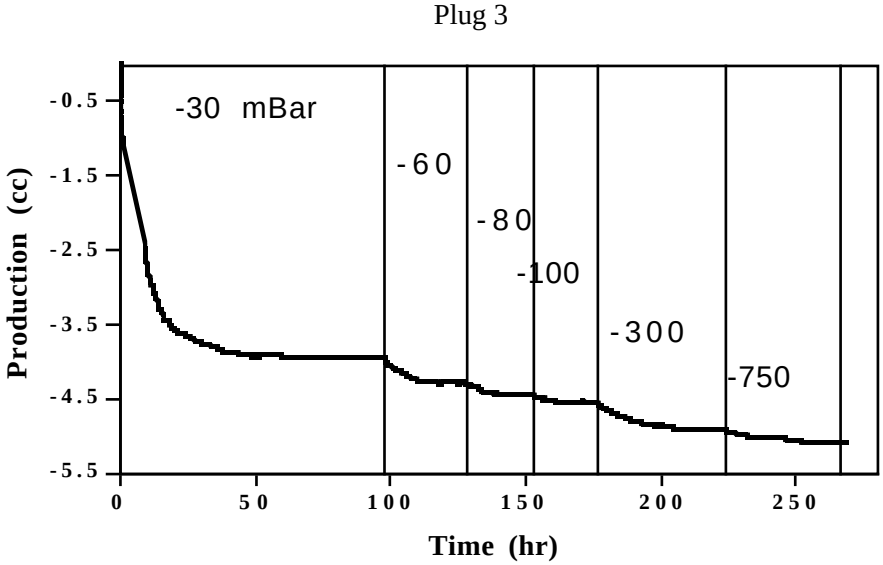


Figure 7: production data during forced imbibition for plug 3.

Table 1 : Duration and equilibrium criterion of the experiments for the different cycles. D= drainage, SI=spontaneous imbibition, FI=forced imbibition. EC=equilibrium criterion

Plug	D (hr)	EC (cc/hr)	SI (hr)	EC (cc/hr)	FI (hr)	EC (cc/hr)
1	160	0.04/4	100	0.03/4*	300	0.04/4*
2	190	0.04/4	90	0.02/12	960	0.02/12
3	350	0.02/12	300	0.02/15	275	0.02/12

(*) final step at 0.02/12

Such a procedure is not entirely satisfactory because :
- it is strongly dependant on the resolution of the production measurement system
- it is sensitive to noise in the same way as a time derivative

- there are no rules for the choice of the value of the maximum flow rate
- the quality of the experiment can only be checked qualitatively by looking at the production curve (Fig. 7) and this is extremely dependant on how the axis scalings are chosen (i.e. how the graph is plotted).

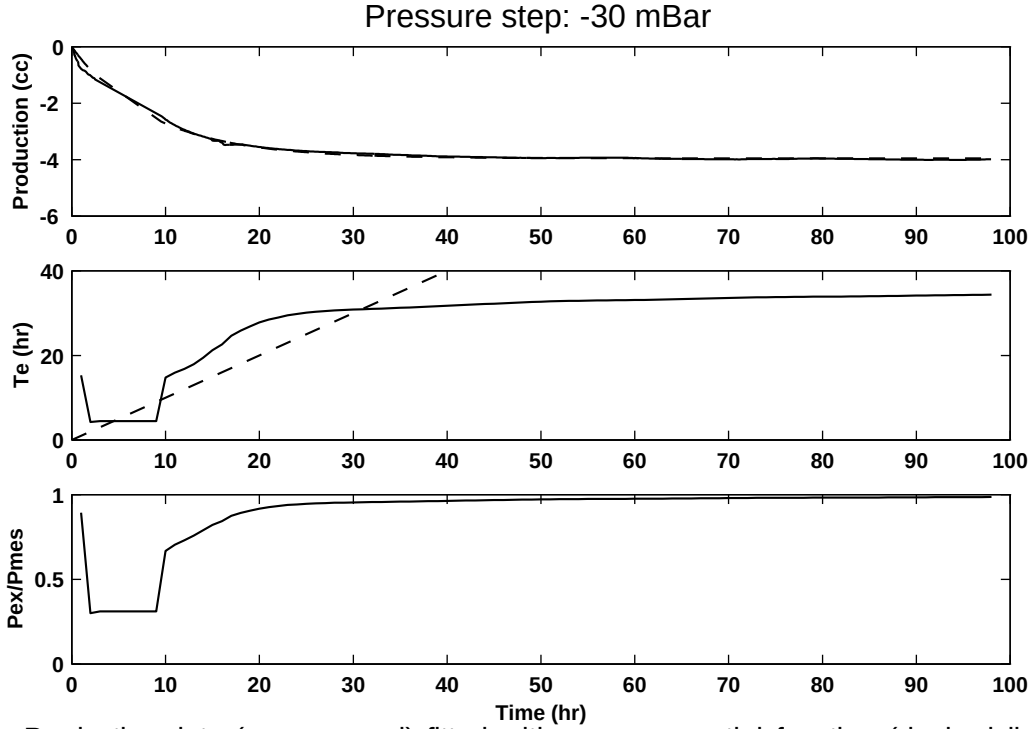


Figure 8: Production data (upper panel) fitted with an exponential function (dashed line) Evolution of Te (middle panel, the dotted line corresponds to x=y) and Pex (lower panel). To optimise the duration of the experiment, the pressure could have been changed at t=30 hr where Te=time. Pmes is the largest measured production.

We propose use of exponential laws of the form :

(2) $P = Pex (1 - \exp(-t/tc))$

where P is the production for a given pressure step, Pex is the asymptotic production at infinite time and tc a characteristic time. We found that the best compromise is at t=4tc=Te where P/Pex=0.98. The choice of the exponential function is not arbitrary. Lenormand et al (1996) have shown that an analytical solution describing the production for small pressure step is a sum of exponentials. Thus, equation 2 can be considered as a first order approximation. Data shown in Figure 8 and 9 indicate a very good fit and confirm the choice. Experimental data not shown in this paper indicate however that production obtained during the first step of primary drainage may not follow exponential laws due to the specific process occurring in this situation. Pex and tc cannot be estimated using simple least square formula but must be estimated by an optimisation routine. We used MATLAB™ routine fmins to determine these two parameters but other standard routines are also adequate. The computation time is of the order of a few seconds.

The proposed procedure is illustrated using the measured production data for plug 3 in Figure 8 and 9 for two pressure steps. It is not a mean to extrapolate the production but to have a non empirical criterion. Te is calculated every hour (for example) using the available production data. At the beginning of the pressure step, both Te and Pex are not well estimated. As more information is available, Te and Pex tend to stabilise. A new pressure step will be started when Te = t, i.e. when the calculated optimum time corresponds to the duration of the pressure step. This time corresponds to t=30hr for step -30 mBar (Figure 8) and 24 hr for step -80 mBar (Figure 9).

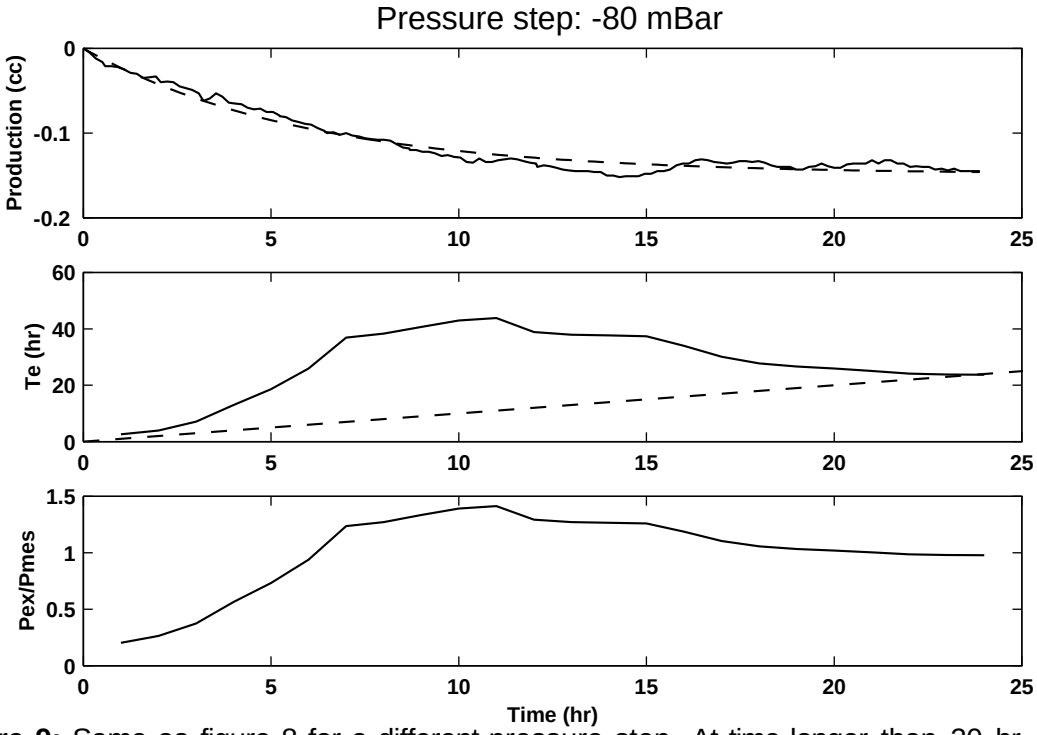


Figure 9: Same as figure 8 for a different pressure step. At time longer than 20 hr, Te is stabilised (middle panel) despite the presence of noise in the data (upper panel).

For pressure step -30 mBar, the duration could have been shorter by about 60 hours. It is interesting to note that Te and Pex are still increasing slightly after 40 hr. This is an indication of the error induced when duration is optimised. In the case considered, the error was 2 % of the total production of the step. Note that at t=100hr, the flow rate deduced from the exponential fitting is about $5*10^{-5}$cc/12hr, several orders of magnitude less than the EC value of 0.02cc/12hr (Table 1). For the pressure step -80 mBar, equilibrium is demonstrated despite the noise in the measurements.

6. Conclusions

When preserved cores are available, we propose a procedure to perform imbibition capillary pressure measurements without cleaning or ageing procedure. The sample is first oil flooded then water flooded at moderate pressure and low flow rates to remove the gas and replace the fluids. A drainage capillary process is then started at the saturation obtained at the end of the water flooding. The purpose of this drainage is to reach an irreducible water saturation using a capillary dominated process. Then, the full imbibition curve is measured up to oil residual saturation.

The above procedure has been applied to a preserved core from a sandstone reservoir (North Sea, Rannoch formation). Full imbibition capillary pressure curves were measured on 3 plugs and their shape indicate intermediate wetting properties. Despite a high permeability (300 mD) and the use of the micropore membrane technique, the duration of the experiment was about 40 days (second drainage and full imbibition curves). Classical technique using ceramics instead of membranes would be longer by a factor of ten (400 days). To optimise the duration and determine capillary equilibrium with non empirical criterion, we propose a method based on exponential law fitting.

We obtained different results with different fluid systems (formation brine/refined oil or formation brine/ crude oil) at a temperature of 40°C. Mainly, the plateau in the negative imbibition curve is larger (in absolute value) for crude oil and there is a difference of Swi

value. Using the available data, it is not possible to conclude. However, we suspect that wax present in the crude oil can be responsible for a partial plugging of the membrane and can generate artefacts. Therefore, we recommand to use refined oil when experiments cannot be performed at elevated temperature.

Future development of our equipment will focus on the 40°C temperature limitation. It is related directly to the temperature limitation of the water wet membranes (oil wet membranes have a theoretical limitation of 200°C).

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Nomenclature

C	capacity at a given frequency, practical units in pF (picoFarad)
e	thickness of the coating on the metallic rod, about 0.05 mm (e « R)
EC	equilibrium criterion, cc/hr
h	height immersed in the conducting liquid
K	permeability, mD
Pex	production at infinite time (from exponential law)
Pmes	highest production measured during a pressure step
Pc	capillary pressure
R	radius of the metallic rod (2 mm)
Swi, Sor	irreducible water saturation, residual oil saturation
Sw	average brine saturation
Te	time at which P/Pex=0.98
ε_r	dielectric constant of the coating , $\varepsilon_0 = 8.859\ 10^{-12}$ A.s/V.m (vacuum)

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