

Journal of Petroleum Science and Engineering 20 (1998) 253-258

# Capillary pressure scanning curves by the micropore membrane technique

Wibeke L. Hammervold<sup>a,1</sup>, Øystein Knutsen<sup>b,2</sup>, Jan Erik Iversen<sup>a</sup>, Svein M. Skjæveland<sup>b,\*</sup>

> <sup>a</sup> RF-Rogaland Research, Rogaland, Norway <sup>b</sup> Stavanger College, Stavanger, Norway

> > Accepted 21 January 1998

## Abstract

Experimental procedures have been developed for capillary pressure hysteresis measurements. Capillary drainage and imbibition bounding curves (complete bounding cycle) and a number of scanning curves have been measured for a water-wet Berea core and an oil-wet reservoir core by the micropore membrane technique. The curves are measured both with stepwise and continuous change of the differential pressure. A continuous and slow change of capillary pressure gives considerable savings in experimental time. Reversibility of scanning curves is observed for the Berea core when the range of saturation reversal is less than 5% and may be explained by a pinning effect. The oil-wet reservoir core, however, exhibits a hysteresis loop even for this small saturation range. For saturation reversals in excess of 5%, the scanning curves for both samples form closed loops that are similar in shape to the bounding loop, i.e., for each sample, all hysteresis loops have the same shape but different sizes. This fact may be used to improve the algorithm for hysteresis modelling. © 1998 Elsevier Science B.V. All rights reserved.

Keywords: capillary pressure; micropore membrane technique; bounding curves

# 1. Introduction

Direction reversal of saturation change may take place during laboratory experiments and in field operations, e.g., WAG operations. The capillary pressure will then scan from one bounding curve to the other (Dullien, 1992). In Fig. 1, the bounding curves of primary drainage, starting at 100% water saturation; the (bounding) imbibition curve; the secondary drainage curve, together with several examples of scanning curves between the secondary drainage and imbibition bounding curves are shown. Most experimental evidence indicate that the secondary drainage curve and the (bounding) imbibition curve constitute a closed and reproducible hysteresis loop (Morrow, 1970).

<sup>\*</sup> Corresponding author.

<sup>&</sup>lt;sup>1</sup> Now with Statoil.

<sup>&</sup>lt;sup>2</sup> Now with Esso Norge.

<sup>0920-4105/98/\$19.00 © 1998</sup> Elsevier Science B.V. All rights reserved. *PII* S0920-4105(98)00028-X



Fig. 1. Bounding and scanning curves.

Morrow and Harris (1965) found that a scanning curve and its reversal constitute a closed and reproducible scanning loop. This result was verified by Szabo (1974). All the capillary hysteresis studies reported have been performed on water-wet material, and the scanning curves have been measured for positive capillary pressure values.

This work presents capillary pressure curves obtained by the micropore membrane technique (Hammervold and Skjæveland, 1992; Longeron et al., 1994). By replacing the traditional porous plate (diaphragm) with thin membranes, considerable time can be saved. However, since measurement of a complete set of scanning curves requires a large number of datapoints, each at a state of equilibrium, the total time needed is still considerable. For instance, if one scanning curve can be constructed from 5-10 data points, each with an equilibration period of 1-5 days, a set of five scanning curves will require from 25 to 250 days, which is rather unrealistic for routine analysis. To reduce the experimental time, a continuous change of capillary pressure has been tested in some of the experiments. This method seems well-suited for supplying scanning curves with good accuracy.

## 2. Experimental

# 2.1. Apparatus

The experimental setup (Hammervold and Skjæveland, 1992; Longeron et al., 1994), consists of the following components:

- Two Quizix pumping cylinders model 5600; maximum cylinder volume 20 ml; maximum pumping rate 7.5 ml/min.
- One Honeywell pressure transmitter with adjustable measurement range; maximum line pressure 7000 psi.
- One computer for pump control and data logging.
- Coreholder with special constructions for the micromembranes.

The coreholder consists of three parts; two endpieces that can be dismounted separately and a cylinder for the coated core. The coating is a low-melting-point ( $124^{\circ}C$ ) alloy. The core and the coreholder are being soldered together into one piece. The endpieces have two connecting tubes, so circulation of fluid is possible (without fluid entering the core). This is to ensure complete saturation of the endpiece and membrane arrangement after the core has been mounted. The diameter and the thickness of the core are approximately 5 cm and 2 cm, respectively.

# 2.2. Measurements

For the positive part of the capillary pressure curve (for a water–oil system), the oil pressure is always higher than water pressure, and a water-wet membrane is used. When measuring the negative part, it is necessary to have an oil-wet membrane, placed at the top of the core. Use of two membranes may increase the equilibration time. Therefore, only one membrane is used initially for positive capillary pressures. To measure negative capillary pressures, an oil-wet membrane is installed at the zero capillary pressure point.

One pump cylinder is connected to the top of the core (oil pressure), another to the bottom of the core (water pressure). For drainage and spontaneous imbibition measurements (positive capillary pressure values), the first cylinder is used to keep the water pressure constant at the bottom. The second cylinder (containing oil) is operated in a constant differential

pressure mode to keep the pumping pressure constant, equal to a preset value. For forced imbibition and spontaneous drainage measurements (negative capillary pressure values), the cylinder functions are the opposite; the first cylinder is operating in a constant differential pressure mode and the second cylinder is used to keep a constant pressure in the oil phase.

The pump volume readings have a resolution of 0.0001 ml. We consider a volume measurement accuracy of 0.01 ml which corresponds to a water saturation of about 0.1% for samples with porosity around 20%. The differential pressure transmitter has an accuracy of 0.1% of the measurement range. With a 0–350 mbar range transmitter, the pressure accuracy is approximately 0.35 mbar.

The water-wet membrane is a Millipore VVLP membrane with a poresize of 0.1  $\mu$ m. The oil-wet membrane is a PTFE-membrane from GoreTex. The oil-wet membrane has a poresize of 0.02  $\mu$ m.

The measurements were performed at ambient pressure and temperature, except for the reservoir core which was measured at 40°C.

### 2.3. Continuous change

Instead of the conventional stepwise sequence of capillary pressure points, a continuous change of differential pressure may be applied to reduce the experimental time. This technique has been used to measure relative permeability (Kokkedee and Boutkan, 1993). It is valid for a quasi steady-state condition with a slow change of the differential pressure. Obviously, a low ramping speed will give more accurate measurements, and a high ramping speed will reduce the experimental time.

The ramping speed in this work had to be determined on a trial and error basis. For the Berea core, the validity of the continuous method was checked by measuring the secondary drainage curve and the spontaneous imbibition curve both by the stepwise and the continuous method. A pressure differential change from 0 to 40 mbar in 4 days corresponds to an average injection rate of 0.0003 ml/min ( $\approx 0.05$ PV/day). According to Kokkedee and Boutkan (1993), this is sufficiently slow to restrict the measurement error from the quasi-equilibrium to an acceptable level.

## 3. Berea core

The Berea core has a diameter of 5 cm, length of 2 cm, and the brine permeability is around 500 mD. The core was initially saturated with 100% brine and the oil injected was Isopar-H (refined oil). The residual water saturation was 24.9% PV at a capillary pressure of 340 mbar. The reduction in oil saturation during forced imbibition was small since the core was strongly water-wet. The residual oil saturation was 36.7% PV at a capillary pressure of -350 mbar.

Three wettability indices for the core were determined. The HL-index (Longeron et al., 1994) was 0.98, in agreement with the Amott index of 0.95. The USBM-index was calculated as 1.97. All indices indicate a strongly water-wet core. The capillary pressure bounding loop measured by stepwise increase of differential pressure is shown in Fig. 2, together with the primary drainage curve.

## 3.1. Continuous measurements

The ramping speed was kept constant for each series of measurements and varied from 60 to 4 mbar/day. The time to reach equilibrium was observed to be longer for the imbibition process than the drainage process, and comparing stepwise and continuous measurements for the imbibition bounding curve indicate that a ramping speed of 60 mbar/day is too high, particularly at low capillary pressures.



Fig. 2. Bounding curves for Berea core.





Fig. 3 shows the bounding curves and a set of scanning curves. Although the scanning curve is reversed after it merges with the bounding curve, the capillary pressure scanning loop is closed. The shape of the scanning loop is similar in shape to the bounding loop.

Fig. 4 shows the bounding curves and another scanning loop, exhibiting very little, if any, hysteresis. Note that the saturation reversal range is only 5% PV, much smaller than that of Fig. 3. This observation is in agreement with Braun and Holland (1994) for relative permeability scanning curves. They observed that on a scanning curve, the cores were free of hysteresis if the saturation reversal range was limited. The cores used by Braun and Holland was (1) a Berea core with permeability of around 500 mD (i.e., a core similar to that used in this work) and (2) a reservoir core from the Kingfish field. Fig. 5



Fig. 4. Reversible scanning curves, Berea core.



Fig. 5. Another closed scanning loop, Berea core.

shows another closed scanning loop for an intermediate saturation reversal range of about 10% PV.

#### 4. Reservoir core

Several series of experiments were performed on an oil-wet reservoir core from a North Sea sandstone reservoir. The 'seal-peel' core containing the reservoir fluids was received with brine and oil at an unknown saturation. Core samples were drilled from the material, and CT-images revealed some invasion of mudfiltrate. To avoid a possibly damaged sample, the only possibility was to put two 'half-moon' pieces together as a core; diameter approximately 4.3 cm, and length 1.5 cm. The core diameter and length were not accurately measured due to the poor consolidation of the core material. The permeability to brine was 2200 mD, measured on other samples from the same 'seal-peel' core. The permeability of the core itself is probably higher because of the fracture between the two halves and no confining pressure.

The wettability indices for the reservoir core was calculated from the bounding loop, Fig. 6. The HLindex is -0.252, the Amott-index -0.78, and the USBM-index -0.431. All indices indicate a relatively oil-wet core. The HL-index indicates a slightly oil-wet core, while the Amott-index indicates a strongly oil-wet core.

The core was not cleaned. It was wrapped in teflon before introducing the alloy in the annulus between the core and the coreholder. After the alloy had solidified, the teflon tape was removed from each endface and the core was saturated with dead



Fig. 6. Bounding curves for reservoir core.

crude oil and formation water, and the last preparatory flow sequence was a waterflood until no more oil was produced.

The saturations were determined by two different titration methods at the end of the experiment. First, a miscible displacement of chloride brine by nitrate brine was performed, followed by titration with chloride to determine the ionic strength. Then, knowing the salt concentration, the water volume was calculated. Second, a Karl Fischer titration was performed (Ritz, 1979). The fluids were extracted by solvents and the water volume was found by titrating the solvents with a Karl Fischer reagent. This method is less sensitive to the salt concentration which is uncertain due to evaporation; the experiment ran for several months and the pump cylinder was replenished several times from a container of brine.

The membranes were the same as for the Berea core measurements. The core material was poorly consolidated, and the cores were drilled out in frozen condition. Due to the presence of loose sand grains, it was difficult to get a smooth and planar core surface against the membranes. A cellulose prefilter was put against each endface of the core to prevent the sandgrains from being produced during flooding. The experiments were performed at a temperature of  $40^{\circ}$ C.

The residual water saturation was estimated at 20.3% PV (at a capillary pressure of 350 mbar), and the residual oil saturation was 18.3% PV (at -350 mbar). The residual water saturation at the end of primary drainage (approximately 20%) is higher than the value obtained by porous plate measurements, approximately 7% PV, for a parallel experiment to

determine the relative permeability relationships. This can be explained by the difference in applied maximum capillary pressure, 0.35 bar and 2.5 bar, respectively.

Fig. 6 shows the stepwise measurements. The curve denoted by 'drainage' describes the first flooding sequence starting at the residual oil saturation left by the preparatory waterflood. It is neither a primary drainage curve, since the core was not cleaned, nor a secondary drainage curve, since it does not start following a spontaneous drainage sequence (spontaneous imbibition of oil).

### 4.1. Continuous change

Three loops were measured, all curves interpreted as scanning curves. The ramping speed was 2.5 mbar/day, except for one series at 5.0 mbar/day. The curves are all plotted in Fig. 7 together with the bounding curves from the stepwise measurements.

The production data showed that a fairly long time was needed to establish equilibrium during the stepwise forced imbibition and spontaneous drainage processes, even though the core is highly permeable. This slow production is due to film drainage. The oil is attached to the rock surface in continuous oil-wet paths through the core, and slowly drains out through the film. Film drainage has been used to explain very low residual oil saturation in mixed-wet cores and mobile oil down to low oil saturations (Salathiel, 1973). During the spontaneous drainage process (spontaneous imbibition of oil), the oil gets sucked in through the continuous films established on the surface inside the pore channels. For the reservoir core, the ramping speed for the continuous measurements



Fig. 7. Scanning loops, reservoir core.

is probably too high to stabilize the production through the thin films.

In Fig. 7, all three scanning loops are closed, and no reversibility is seen, even for the smallest loop with a saturation reversal range of 5% PV. Note again that the shape of the bounding loop is repeated on a smaller scale for the scanning curves.

We have made no systematic stepwise measurements to verify the accuracy of the continuous scanning curves, except for one point at 5 mbar for the 1-10-1 mbar loop. The equilibrium saturation was reached at 5 mbar before the ramping option was invoked for increase up to 10 mbar. The point matches well with the other two loops at 5 mbar.

The production from the reservoir core is in general slower than for the Berea core (for both forced and spontaneous processes). For the continuous measurements, the ramping speed of 2.5 mbar/day is probably too high for the spontaneous drainage curves, especially at low differential pressure. This will result in the spontaneous drainage curves (i.e., spontaneous imbibition of oil) having too high water saturation at low differential pressure values.

## 5. Modelling

We have tested the Killough (1976) hysteresis model on loops in this paper with only a limited degree of success. In Killough's model, the scanning curves are made by a direct interpolation between the bounding curves. From Figs. 3–5 and 7, however, it seems more reasonable that the scanning curves form loops that are similar in shape to the bounding curves. A hysteresis model formulated in this manner shows promising results (Skjæveland et al., 1998).

#### 6. Conclusions

Capillary drainage and imbibition bounding curves and a number of scanning curves have been collected for a Berea and a reservoir core samples. The reproducibility of the data has been verified.

A new technique with a continuous and slow change of capillary pressure has been tested and proved to be feasible. A rigorous error analysis is desirable. Reversibility of scanning curves was observed for the Berea core for saturation ranges of less than 5% PV, but not for the reservoir core.

The Killough hysteresis model needs improvement to properly trace the measured scanning curves.

# Acknowledgements

We thank Esso Norge for support and permission to publish the paper.

### References

- Braun, E.M., Holland, R.F., 1994. Relative Permeability Hysteresis: Laboratory Measurements and a Conceptual Model. Paper SPE 28615 presented at the 1994 SPE Annual and Technical Conference and Exhibition, New Orleans, LA, Sept. 25–28.
- Dullien, F.A.L., 1992. Porous Media-Fluid Transport and Pore Structure. Academic Press.
- Hammervold, W., Skjæveland, S.M., 1992. Improvement of Diaphragm Method for Drainage Capillary Pressure Measurement with Micro Pore Membrane. Paper presented at the EUROCAS meeting, Paris, September 8–10.
- Killough, J.E., 1976. Reservoir Simulation With History-Dependent Saturation Functions. SPEJ, Feb., pp. 37–48.
- Kokkedee, J.A., Boutkan V.K., 1993. Towards Measurement of Capillary Pressure and Relative Permeability at Representative Wettability. Paper presented at the European Symposium on Improved Oil Recovery, Moscow, Oct. 27–29.
- Longeron, D., Hammervold, W.L., Skjæveland, S.M., 1994. Water–Oil Capillary Pressure and Wettability Measurements Using Micropore Membrane Technique. Paper presented at the International Symposium of the Society of Core Analysts, Stavanger, September 12–14.
- Morrow, N.R., 1970. Physics and thermodynamics of capillary action in porous media. Ind. Eng. Chem. 62 (6), 32–56.
- Morrow, N.R., Harris, C.C., 1965. Capillary equilibrium in porous materials. Trans. AIME 234, 15–24.
- Ritz, C.L., 1979. Rapid and Accurate Electrochemical Determination of Water Content From Core Flood Experiments. Paper SPE 7871 presented at the 1979 SPE International Symposium on Oilfield and Geothermal Chemistry, Houston, TX, Jan. 22–24.
- Salathiel, R.A., 1973. Oil recovery by surface film drainage in mixed-wettability rocks. JPT, Oct., pp. 1216–1224.
- Skjæveland, S.M., Siqveland, L.M., Hammervold, W.L., Virnovsky, G.A., Kjosavik, A., 1998. Capillary Pressure Correlation for Mixed-Wet Reservoirs. Paper SP 39497 to be presented at the 1998 SPE India Oil and Gas Conference and Exhibition, New Delhi, India, April 7–9.
- Szabo, M.T., 1974. New methods for measuring imbibition capillary pressure and electrical resistivity curves by centrifuge. SPEJ, June, pp. 243–252.