IMPROVEMENT OF DIAPHRAGM METHOD FOR DRAINAGE CAPILLARY PRESSURE MEASUREMENT WITH MICRO PORE MEMBRANE

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Abstract A procedure is presented for measuring the drainage liquid-liquid capillary pressure curves by the diaphragm method. The customary ceramic porous plate is replaced by a thin micropore membrane that significantly reduces measurement time.

A coreholder with a rubber sleeve allows for confining pressure on the core and is used with a refined oil-brine system at ambient conditions. Eight drainage capillary pressure curves have been measured by the membrane method on Berea sandstone cores with different permeability's. For two of the core samples, the curves were also recorded by the traditional ceramic plate method. There is good agreement between the results of the micropore membrane and the ceramic plate methods.

The main advantage of the new method is the reduction in equilibration time. Plots of produced volumes following a step increase in differential pressure exhibit a considerable decrease in equilibration time when the micropore filter is used. The time ratio between the two methods is typically a factor of 10. A drainage capillary pressure curve measured by the micropore membrane method with e.g. seven data points may take approximately

ten days, compared with about 3 months for the ceramic disk method.

INTRODUCTION

One of the most popular methods for measuring the capillary pressure curve is the diaphragm method with a ceramic disk acting as a diaphragm. In a capillary drainage experiment, the ceramic disk conducts flow of the wetting phase while the nonwetting phase is repelled as long as the capillary pressure is below the entry pressure of the disk. For incrementally higher pressure, a droplet of the nonwetting phase will penetrate into the disk, at the largest pore entry. If the poresize distribution of the disk is fairly uniform, as soon as the nonwetting phase is inside the disk, it will shortly be at the other side, and the experiment has to be terminated. The impediment of the nonwetting phase is therefore not related to the thickness of the disk but depends on the porethroat morphology at its inlet face. The thickness of the disk just contributes to the flow resistance of the wetting phase and in this case has no effect on the phase separation.

To measure the capillary pressure curve, the pressure of the nonwetting phase surrounding the core is increased stepwise. At each step, capillary pressure equilibrium has to be reached before a reading is made of the produced volume of wetting fluid. This fluid has to flow through the core and the porous disk while the differential pressure in the wetting fluid is gradually reduced to zero. If the porous disk is thick, the equilibration time may be controlled by the disk, not by the core itself.

To reduce the rather impractical measurement time of several weeks for a capillary pressure curve, it would seem

reasonable to reduce the thickness of the ceramic disk. Previous studies [1] have shown a considerable reduction in time required for the experiment by use of a membrane for a water/gas system. The authors remark that "In principle, there is no reason that the method cannot be applied to the measurement of water-oil capillary pressure and relative permeability's. Before this can be done a membrane must be found with the appropriate properties. The requirements for such a membrane are that it be sufficiently waterwet to enable an adequate entry pressure and a low flow resistance."

Other authors have applied the membrane technique to resistivity measurements [2,3] by the continuous injection technique, for liquid-liquid systems. Membranes with rather high entry pressures of 6, 9, and 50 bars were used. No equilibrium measurements of a capillary pressure curve were reported.

In our experiments, we have mostly used a thin membrane with an entry pressure of 4 bar for oil/water drainage curves, and waited for equilibrium at each new pressure point. It is demonstrated that the consumed laboratory time may be reduced by a factor 10.

In future work, we plan to use membranes for wettability determination according to the USBM procedure [4], at reservoir conditions, instead of the more commonly used centrifuge technique.

EXPERIMENTS

Equipment

Membrane

Two types of waterwet membranes were tested: From the manufacturer Millipore Co. a cellulose filter with irregular morphology, and from Nuclepore Co. a polycarbonate membrane with regular, cylindrical holes of equal size.

The membranes were tested in an empty coreholder for the entry pressure to oil, calculated equivalent permeability, and time to produce the water in the coreholder (20 cm^3) at a 0.3 bar pressure drop. The Nuclepore membranes had a thickness of 6-11 µm, and the Millipore membranes 150 µm. The results are shown in Table 1, where d is the diameter of the largest pore, u_w values are from product catalogue, N denotes Nuclepore membrane, and M denotes Millipore membrane.

The poresizes are as listed in the product catalogue. The Nuclepore polycarbonate membranes have well-defined poresizes which permit precise particle fractionation and separation. The cellulosic membranes have series of channels with no clearly defined or regular porestructure. They are used where the tight specification of poresizes is unnecessary.

Table 1: Test results of Nuclepore and Millipore membranes.

d µm	p_e bar	u_w ml/min /cm^2	k md	Time min	Type N or M
0.2	1.5	20.0	0.292	2	N
0.1	2.	4.0	0.058	2	N
0.08	4.	2.0	0.029	30	N
0.015	6.5	<0.1	0.002		N
0.45	0.9	38.5	1.874	4	M
0.1	5.2	1.5	0.073	60	M

Breakdown of the membrane occurs because of mechanical rupture or because of too large pressure difference across the membrane. Breakthrough pressure is the pressure at which the non wetting phase passes the membrane. The results show that the Nuclepore membrane with the same pore diameter has a higher water flow rate than the Millipore membrane while the breakthrough pressure is lower. Millipore membranes are more than three times as thick and act more like a porous medium with irregular porechannels, while

Nuclepore polycarbonate membranes have patterns of circula holes.

The Nuclepore membranes tend to break in the middle just where the annulus is located in the endpiece. This may be the cause of the surprisingly low breakthrough pressure compared with the Millipore membrane. A flat support screen was put between the membrane and the endpiece to achieve a favourable strain distribution. Another problem was to place the membrane without wrinkling it. A support screen helped alleviate these problems.

The advantages using the Nuclepore membrane are the high flowrate and the good mechanical strength as compared with the Millipore membranes. It can also bend and stretch more easily than the Millipore membrane.

A Nuclepore membrane with poresize of 0.2 μm was tested with a support screen and the breakthrough pressure was between 1 and 2 bar. This is higher than expected when compared with the 0.1 μm membrane that was tested without support screen. The membrane with poresize 0.08 μm broke down at 4 bar, with support screen. It is reasonable that the breakthrough pressures of the 0.1 and 0.015 μm membranes are too low since they were measured without the support screen.

The Nuclepore membrane with poresize 0.015 μm exhibited a low flowrate and the time to produce the water from the coreholder was not measured. The rate will increase with the differential pressure, however, and the membrane can be suitable for establishing initial water saturation, S_{wi}, of a core sample.

Data from other publications [5,6] indicate that capillary pressures at least as high as 1.5 bar need to be measured defining the lower limit of the breakthrough pressure to oil. Much higher breakthrough pressures should be avoided

however, since a membrane with high entry pressure would have lower effective permeability, and the equilibration time would increase.

Nuclepore polycarbonate membrane with a poresize of 0.08 μm and entry pressure of 4 bar was chosen for the experiments in this paper.

Coreholder

The main problem with the membrane is to get it properly placed in the coreholder without leaks. The membrane is very thin, 6-11 μm, and one cannot seal the edges with a rubber sleeve because it is not stiff like a ceramic disk. Different endpieces were therefore constructed and tested.

One important point regarding the membrane assembly is to avoid twisting the endpiece relative to the core. The thin membrane may then be damaged, resulting in a lower breakthrough pressure than its rated entry pressure, and the experiments will be difficult to reproduce, especially at high pressures.

A coreholder was therefore constructed as sketched in Fig. 1, with the axial pressure independent of the confining pressure. There are no threads in the coreholder, so no twisting of the endpiece occurs during installation. The confining pressure was set to 30 bar. In this set-up, the differential pressure was increased to 2.4 bar without any breakthrough of oil.

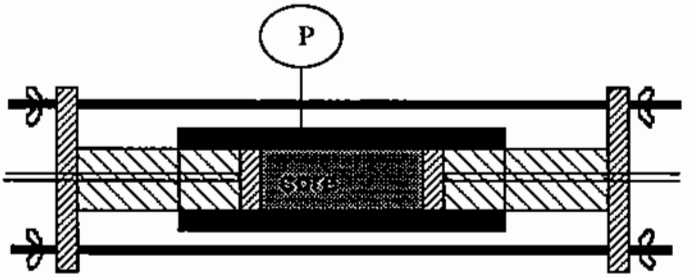

FIGURE 1: Core holder

Endpiece

There are two main problems when constructing the endpiece One is the mechanical challenge, the other is leakage. It is important to have a smooth, plane support screen behind the membrane. If this screen is too rough, it will impale holes in the membrane when axial pressure is applied. The screen must also have a high permeability so that no extra flow resistance is introduced that would increase the equilibration time. The membrane must be protected against the rough surface of the core sample. For this purpose, a Millipore membrane with higher permeability is used. The displacing phase must be hindered from passing around the membrane After testing several alternatives, we suggest the arrangement in Fig. 2.

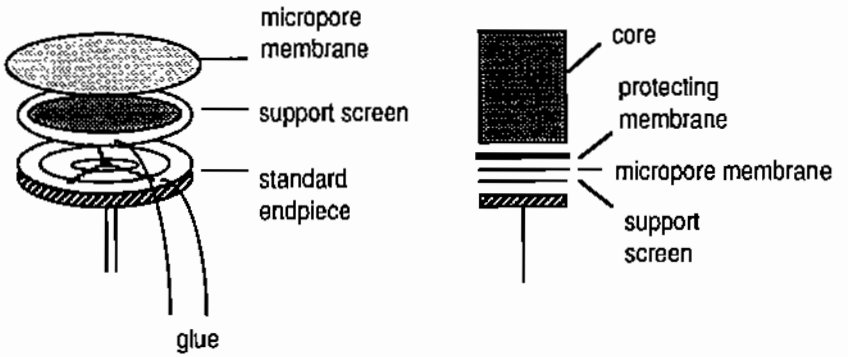

FIGURE 2: Membrane arrangement with support screen and glue.

The membrane is glued to the thin support screen which again is glued to the original endpiece of the coreholder. The protecting membrane is saturated with a solution of contact powder and brine.

Several datasets were collected with other types of endpiece arrangements as well.

Procedure

A simple laboratory set-up was made for drainage capillary

pressure measurements in order to test the method. The displaced brine was produced at atmospheric pressure and the amount measured by weight. The differential pressure was recorded as a function of time by use of a Smart transmitter and data logged and presented by a PC. The brine concentration used was 30 g/l NaCl, 10 g/l $CaCl_2{}^*H_2O$, and 0.3 g/l KCl. Ambient temperature was 20°C.

Different Berea sandstone cores with permeability's around 100, 200, and 500 md were used. Nine capillary pressure curves were measured.

Two of the core samples were sent to a commercial laboratory for traditional porous plate measurements in order to compare the two methods.

Many tests failed with oil breakthrough after one or two pressure points until we designed the endpiece arrangement in Fig. 2.

RESULTS

In Fig. 3 are shown the capillary pressure curves of two cores, labelled G-1 and G-2, whose properties are displayed in Table 2.

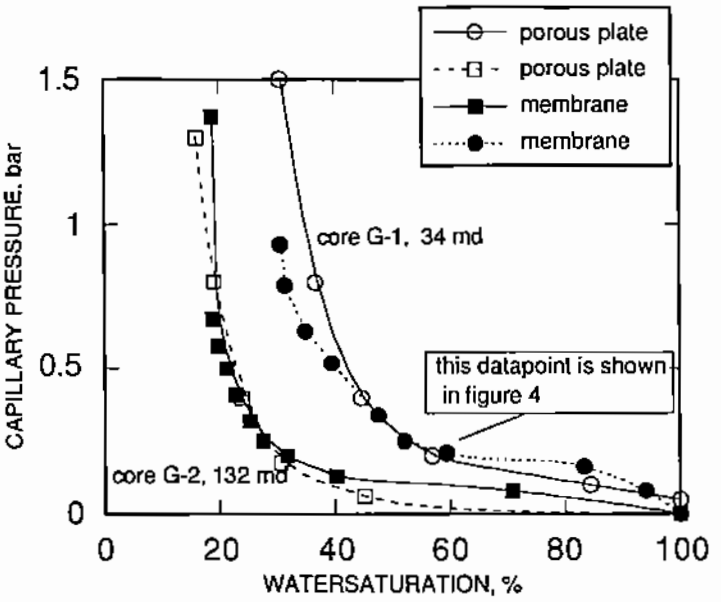

FIGURE 3: Capillary pressure curves with porous plate and membrane for cores G-1 and G-2.

TABLE 2: Properties of core G-1 and G-2.

Label	d, cm	L, cm	V_p, cm3	ϕ, %	k, md
G-1	3.71	5.06	9.35	17.1	34
G-2	3.70	5.10	10.2	18.6	132

The data points of core G-1 are represented by circles in Fig. 3, open circles for porous plate data and filled for membrane data. Equivalent results for core G-2 are represented by squares. There is a fairly good correspondence between the two methods. The saturation data function of pressure for both the porous plate and membrane (plotted in Fig. 3) are displayed in Table 3 and 4.

One major source of inconsistency when comparing the two methods is the wait period at each new pressure step before a reading is made. For core G-1 and the 0.8 bar data point, it is possible that the match would have been better if the wait period for the porous plate method had been longer.

TABLE 3: Saturation data function of capillary pressure for cores G-1 and G-2 measured by porous plate.

core G-1		core G-2	
capillary pressure bar	water saturation,%	capillary pressure bar	water saturation,%
1.5	30.6	1.3	16.0
0.8	36.6	0.8	19.1
0.4	44.6	0.4	23.8
0.2	56.9	0.175	30.6
0.1	84.4	0.06	45.2
0.05	100	0	100
0	100		

TABLE 4: Saturation data function of capillary pressure for cores G-1 and G-2 measured by membrane.

core G-1		core G-2	
capillary pressure bar	water saturation,%	capillary pressure bar	water saturation,%
0.93	30.6	1.37	18.8
0.79	31.6	0.67	18.9
0.63	35.7	0.58	19.7
0.52	39.5	0.5	21.3
0.34	47.6	0.41	22.9
0.25	52.1	0.32	25.3
0.21	59.4	0.25	27.5
0.16	83.3	0.2	31.6
0.08	94	0.13	40.2
0	100	0.08	70.8
		0	100

DISCUSSION

To compare the wait period at each pressure step between the two methods, we will introduce the concept of *characteristic time*, t_{ch}. We have recorded the recovery (production) of brine at each pressure step as a function of time. By trial and error the data points are for all cases nicely fitted by a functional form of the Langmuir type [7],

$$R = \frac{at}{1 + bt}, \tag{1}$$

where R is recovery, a and b are constants, and t time. Although it is an empirical relationship, there exists some justification for this particular choice. For long times, when $bt >> 1$, Eq. 1 becomes $R = (a/b)/(1\text{-}1/bt)$, which is similar to expressions for the recovery curves in Ref. [8].

The characteristic time, t_{ch}, is defined by $t_{ch} = 1/b$. From Eq. 1, the ultimate recovery, R_∞, is equal to a/b and t_{ch} is the time to reach half that value. The parameter t_{ch} may be used to characterise the relaxation process following a pressure step increase, and to compare the two methods on an equal basis.

We have curve-fitted 32 recovery curves, all measured both with the porous plate and the membrane method. The average t_{ch}-value for the porous plate method is 25.8 hours as compared with 3.1 hours for the membrane method. The capillary pressure curve may therefore be determined equally accurate and a factor 8.32 faster with the membrane method.

An example of a curve-fitted recovery curve is shown in Fig. 4. The data were collected while waiting for equilibrium at the third pressure point of the membrane method for core G-1, at $S_W \equiv 60\%$ in Fig. 3. The uncertainties in *a* and *b* are indicated.

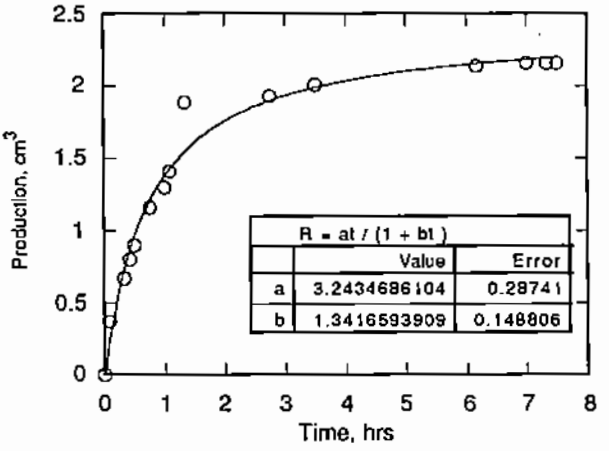

Figure 4: Produced volume as a function of time

Whether measuring by the porous plate or the membrane method, a choice must be made when to take the reading. The curve-fitting procedure described above may be helpful in making the decision. The relative uncertainty in the ultimate recovery at a given pressure step, may be estimated from

$$\frac{\Delta R_\infty}{R_\infty} = \left[\left(\frac{\Delta a}{a}\right)^2 + \left(\frac{\Delta b}{b}\right)^2\right]^{1/2}, \quad (2$$

where the uncertainties Δa and Δb are given by the curve fitting program.

The relative uncertainty in ultimate recovery may be monitored during the wait period after a pressure-step increase. In the beginning, the uncertainty is large and gradually decreases as more data are included in the curve fitting procedure. The example from Fig. 4 is replotted in Fig 5 where the development of the uncertainty in the water saturation is monitored. The uncertainty, ΔS_w, is calculated from

$$\Delta S_w = \frac{a}{b}\left[\left(\frac{\Delta a}{a}\right)^2 + \left(\frac{\Delta b}{b}\right)^2\right]^{1/2} / V_p. \quad (3)$$

We believe that certain rules of thumb may be found to terminate the wait period after a pressure-step increase. The main point is to wait for equilibrium until the uncertainty has been reduced below the required value. Our preliminary findings are promising, and it seems like a set of rules could be recommended along the following lines:

1. Relative error in R_∞ must be less than 25%

2. Relative error in R_∞ must correspond to less than 2% in saturation value

3. The collected data have to include most of the bend in the recovery curve

4. At least 6 points have to be recorded.

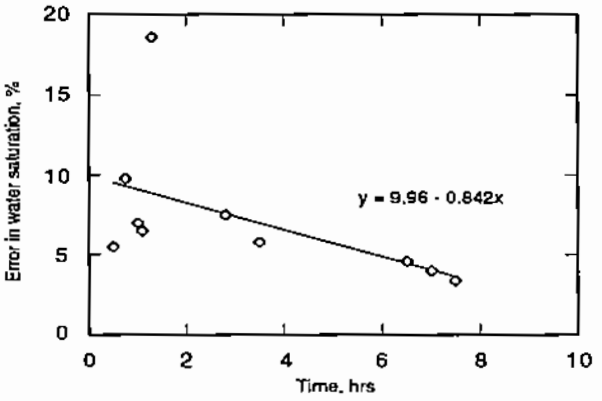

FIGURE 5: Uncertainty in water saturation as a function of time, measured data as in figure 4.

Preliminary results show that the wait period can be reduced significantly with very little reduction in the accuracy of the water saturation.

The main practical problem is the arrangement of the endpiece and the mounting of the core. The solution presented

is not yet completely satisfactory since we occasionally have premature breakthrough of displaced phase. In principle, the procedure could be extended to reservoir conditions and imbibition capillary pressure curves.

CONCLUSIONS

1. Oil/water capillary pressure drainage curves have been measured by the micropore membrane method at ambient conditions

2. A new placement procedure for the membrane shows promising results

3. The results of the membrane method compare well with those from the standard porous plate technique on the same cores, but more extensive testing is necessary

4. The wait period with the membrane method is reduced by a factor 10 as compared with the porous plate method, with the same degree of accuracy

NOMENCLATURE

A = cross-sectional area, cm^2

a = constant, cm^3 or dimensionless

b = constant, s

d = diameter, cm

k = permeability to water, md

L = length, cm

p = pressure, bar

R = recovery, cm^3 or dimensionless

S = saturation, dimensionless

t = time, s

u = Darcy velocity, $ml/min/cm^2$

V = Volume, cm^3

Subscripts

ch = characteristic

e = entry

i = initial or irreducible

p = pore

w = water

∞ = ultimate

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