EXPERIMENTAL MEASUREMENTS OF CAPILLARY PRESSURE WITH THE CENTRIFUGE TECHNIQUE – EMPHASIS ON EQUILIBRIUM TIME AND ACCURACY IN PRODUCTION

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ABSTRACT
We present an experimental study of capillary pressure measurements by the multi-rate centrifuge technique in water-wet and oil-wet carbonate core plugs. The time to achieve hydrostatic saturation equilibrium was experimentally investigated at each speed step. The time to reach equilibrium was dependent on the wettability preference and absolute permeability of the core samples, and spinning time varied from a few days to over a month at some rotational speeds.

Capillary pressure curve measurements by an automated data acquisition centrifuge requiring little or no human intervention is reported. The high degree of automation eliminated the chance for biased production measurements and enhanced the reliability and consistency of the obtained experimental data. The high accuracy in the camera device on the centrifuge provided accurate production measurements, where very small volumes changes (~0.01 cc) were registered. Although the automatic centrifuge provided excellent accuracy in production measurements, it was still limited to average saturation measurements during centrifugation. However, the combination of high accuracy and high frequency data acquisition provided high-quality transient data from the rapid saturation changes associated with increase in RPM. A commercially available core analysis simulator was used to history match the experimental data in an implicit method to determine the capillary pressure curves.

INTRODUCTION
Capillary pressure curves, especially as they depend on wettability, are important in many aspects of reservoir physics. Capillary pressure is a vital input in the calculation of oil above free water level and the volume of oil in the transition zone. In addition, it is a critical parameter in reservoir and core analysis simulations, although often ignored for computational efficiency.

The centrifuge technique consists of rotating core plugs at increasing angular velocities to measure equilibrium saturation as a function of capillary pressure. Measurements are made with two or more fluids in the pore space. Core holders can be configured for drainage or imbibition tests with a more dense phase (water in most cases) changing places with a less dense phase (oil or gas). The core holder orientation determines which phase enters and which phase exits the pore space. Fluid production, or average fluid saturation, is measured as saturation equilibrium is reached at every rotation speed. Equilibrium saturation is determined when fluid production ceases at each centrifuge speed. The ability to measure small increments of fluid production improves the laboratory measurement of saturation and the end point for equilibrium. The main assumptions in this method are 1) capillary equilibrium has been reached at each rotation speed, and 2) capillary pressure is zero at the outlet (producing) end of the core. Once the average saturation from the centrifuge data is obtained, mathematical models are used to compute saturation as a continuous function of capillary pressure.

Extensive efforts have been dedicated to the improvement of centrifuge derived capillary pressure; however, this work has focused on improving the mathematical framework used to compute capillary pressure curves from the experimental data. Ruth and Chen (1995) summarized numerous models that have been proposed over 50 years for modelling capillary pressure from centrifuge data. Fishbane *et al*. (1996) argued that the model chosen to interpret data would influence the shape of the calculated capillary pressure curve. Forbes (1997) investigated nineteen interpretation methods used by different laboratories in a SCA survey. He concluded that the main source of inaccuracy in the drainage capillary pressure curve determination by the centrifuge method was related to the interpretation process. The methods presented by these authors rely on true equilibrium data from the laboratory tests. This may not always be the case since achieving equilibrium in the time available for laboratory work may be difficult. Fleury *et al.* (2000) presented a method to extrapolate the transient saturation curve during multi-speed centrifugation to obtain accurate equilibrium saturation.

An automatic centrifuge setup was utilized to improve quantification of produced fluid volumes. Produced volumes of the displaced phase were measured with a high degree of accuracy while the centrifuge was spinning. The automated equipment improved incremental volume measurements by an order of magnitude when compared to human visual readings.

EXPERIMENTAL

One outcrop chalk core and four limestone core samples were tested for capillary pressure measurements over a range of wettability states. One low permeability homogeneous Rørdal chalk rock, obtained from the Portland quarry at Ålborg, Denmark, often used as a North Sea chalk reservoir analogue, was tested at a moderately water-wet (MWW) state. Four low permeability limestone samples from the Edwards formation in New Mexico, USA were also tested, two at strongly water-wet (SWW) conditions and two at moderately oil-wet (MOW) conditions. The MWW and MOW conditions were produced by flushing crude oil through the samples at elevated temperature suggested by Graue *et al.* (1999 and 2002) and Aspenes *et al.* (2003). Although similar in permeability, the chalk and limestone show large differences in pore structure and the ability to change wettability during aging. The Amott-Harvey (A-H) wettability index and basic rock properties are listed in Table 1.

The centrifuge experiments were performed under ambient conditions at 500 psi confining pressure in a Beckman J-6B centrifuge. The capillary pressure was obtained using a multi-speed centrifugation procedure at speeds of 350, 600, 900, 1200, 1800, 2500 and 3000 RPM. Average saturation was measured once capillary equilibrium was reached at each rotational speed. Data was collected for the MWW chalk in one centrifuge test. SWW and MOW limestone data were collected in a separate test. Twin limestone samples were run to assess reproducibility. A similar twin sample effort in chalk samples failed due to equipment problems.

Production volumes were measured with an automated data acquisition system illustrated in Figure 1. This resulted in a reduction in produced volume uncertainty. The automated centrifuge utilized a fiber optic light line and a digital line camera set normal to the plane of rotation and orientated along the axis of the cup. The centrifuge holds up to four core holders at one time, and as each core holder cup rotated into position between the light and the camera a laser position detector triggered the electronic shutter of the camera. The acquired line images show distinct changes in light intensity corresponding to the cup bottom and the fluid interface. A second laser position detector measured centrifuge speed and provided an index to each sample position. The image data were time stamped and saved to a hard disk. At early times when oil production was fast, frequent images were captured. As time progressed the rate of data acquisition was decreased.

The image data were processed using a proprietary software package incorporating an interface detection algorithm. The software converted observed light intensity to volumetric data. With water and a transparent oil system, aids were required to clearly observe the water-oil interface. A water soluble dye (methylene blue) suited this purpose. The temporal and volumetric data were imported into a spreadsheet where capillary pressure was calculated using a conventional algorithm.

RESULTS

Capillary pressure measurements using the visual technique, relying on human visual observations of equilibrium with an estimated volumetric precision of 0.1 *cc* at best, yields a saturation uncertainty of 0.006 PV (15 *cc* PV). In this work equilibrium was defined when the production of fluid fell below the resolution of the automated system, 0.01 *cc*, over a 24 hour period. This volumetric resolution corresponds to a saturation precision of 0.0006 PV (15 *cc* PV). The increased sensitivity to fluid production had a significant impact on the recognition of equilibrium and consequently the time required to complete the tests. This is particularly true in low permeability samples where slow fluid production can exist for long periods; this was the case in the present work. The significance of the improved volumetric resolution is discussed in more detail below.

Moderately Water-Wet Chalk

The permeability of the chalk sample was 3.7 *mD*. The core plug was aged in crude oil to obtain moderately water-wet conditions (MWW) with an A-H Index of 0.2. Sample data are summarized in Table 1. Cumulative fluid production profiles are shown in Figure 2 and discrete production rates are shown in Figure 3. Zero fluid production at 300 and 600 RPM indicates insufficient centrifugal pressure to exceed the threshold entry pressure. First water production began when the centrifuge speed was increased to 900 rpm, 7 days into the test. The rate of water production was near linear for 16 days before declining to the detection limit of the centrifuge. At successive centrifuge speeds a trend in the shape of produced fluid profile was observed. At low speeds the produced fluid profile is near linear with time. The production profile progressively changes to an exponential shape with each increment in centrifuge speed. The time for equilibrium did not continue to increase with higher rotational speeds, as the longest equilibrium time, 37 days, was observed at 1800 RPM. Near equilibrium saturation values (within 0.5 *cc* of equilibrium) at this rotational speed were achieved after 24 days; however, an additional 13 days were needed to reach equilibrium. Time to reach equilibrium for each speed is tabulated in Table 2. The initial production rate associated with each rotational speed increment increased with RPM throughout the experiment, see Figure 3. Net and cumulative water production at each centrifugal speed in the moderately water-wet chalk sample is shown in Table 3.

The hump in the 3000 RPM data in Figure 3 is attributable to a laboratory artifact. After 125 days (4 months) of spinning the centrifuge bearings burned up and the motor was replaced. Total length of the experiment was 130 days. Final water saturation, reached at 3000 RPM, was 0.249.

With the exception of 3000 RPM, flow rates at all centrifuge speeds are less than 0.05 PVs per day. Spin time to equilibrium ranged from 4 to 37 days with a range in saturation change from 0.03 to 0.17 PV. If using the visual technique to measure production volume, with a production rate resolution of 0.6 PVs/day (15 cc PV), the laboratory operator may not have seen the low capillary pressure production or have waited long enough to see equilibrium. To reach the same criteria of equilibrium the laboratory operator would need to wait 10 days with zero observed production at each centrifuge speed.

Strongly Water-Wet and Moderately Oil-Wet Limestone

The influence of wettability and absolute permeability was investigated with four limestone samples in a similar multi-speed centrifuge experiment. Two of the limestone samples were aged in crude oil to obtain moderately oil-wet conditions. No water spontaneously entered the core during wettability measurements. Oil invaded the core by capillary attraction, spontaneously producing water when submerged in oil. This is typical behavior of an oil-wet state. The A-H Index was slightly different in the twin samples, -0.2 and -0.1. A significant difference in permeability was measured in oil-wet samples, 10.1 and 5.7 *mD*. The remaining two limestone samples were a close match in permeability, 11.6 and 11.8 *mD*. They were not exposed to crude oil and were thus in a natural strongly water-wet condition. Sample data are shown in Table 1. Confining pressure and experimental procedure was identical to the chalk test with one exception. Since four core plugs were spinning in the same test, increments in centrifuge speed did not occur until all four samples reached equilibrium. There is one exception to this last statement which will be discussed later. Cumulative produced water volumes and the elapsed time to reach equilibrium at each centrifuge speed are shown in Figure 4 and in tabulated form in Table 2. Discrete production rate data are presented in Figure 5.

The fluid production rate in the limestone samples was also very slow and may not have been observed without the resolution available in the automated data acquisition system. Sustained flow rates below 0.0001 PVs/day were observed in all four samples. This is below the 0.006 PVs per day of the visual volume measurement. It is possible that the 600 RPM production in the two moderately oil-wet samples would have been missed. Under different circumstances the laboratory operator would have increased the centrifuge speed to 900 RPM after 1 or 2 days. As can be seen in Figure 4, fluid production did not begin until 6 days at 600 RPM. Fluid production was observed however only because the two strongly water-wet samples, which were in the same test, did not reach equilibrium until 10 days had elapsed. By that time production in the two moderately oil-wet samples had started. This production continued for 35 days.

<u>Strongly Water-Wet Samples</u>
No water production was observed at 350 RPM and both cores started to produce water at 600 RPM. Production rates levelled out and near-equilibrium saturation values were reached within 10 days of spinning at 600 RPM. High production rates were observed in the two strongly water-wet cores and water production started almost instantaneously after each speed change. The shape of the cumulative production curves is exponential at all speeds. Residual water saturations at 3000 RPM were 0.589 and 0.487. Net and cumulative water production at each rotational speed is tabulated in Table 3.

<u>Moderately Oil-Wet Samples</u>
No water production was observed at 350 RPM. At 600 RPM, 6 days elapsed before water production started. Once initiated the water production rate remained near linear for more than 19 days in one sample and was still near linear after 32 days in the other sample. At this point an unintended increment in centrifuge speed to 900 RPM occurred. As can be seen in Figure 4, although a large volume of fluid had been produced, neither sample was at equilibrium when the speed increment occurred. Subsequent centrifuge speed increments did not show marked changes in saturation and, as seen in the chalk sample, showed a reduced linear production behavior transitioning to an exponential production behavior. Net and cumulative water production for the moderately oil-wet cores are presented in Table 4.

SIMULATIONS

All capillary pressure curves were obtained by a multi-speed centrifuge average saturation measurement approach. This method required the application of a mathematical framework to calculate the local capillary pressure curve, and was solved using available core analysis simulatoin software. The local capillary pressure was achieved by using three different correction equations (Hassler-Brunner, Forbes and Forbes/splines) on the experimentally obtained data using the Cydar core analysis software.

The equations were compared to find the optimal equation for the available data sets. A mathematical fit to the experimental data points was obtained and the local capillary pressure curves were calculated using Hassler-Brunner (H-B) and Forbes/splines from a linear fit of experimental data. The low number of equilibrium saturation points available illustrated the disadvantage of not knowing the appropriate speed schedule for optimized high quality experimental data discussed by Ruth (2006). The shapes of the capillary pressure curves, especially the large change in saturation observed at 600 RPM for the MOW cores, were not accurately matched by the H-B solution, and discontinuous saturation development was achieved in the local capillary pressure curve. The back-calculated H-B average saturation, however, fitted the experimental data point satisfactorily. The polynomial and power law did not provide a satisfactory fit to the experimental data. The Forbes/splines option produced smooth local and average saturation curves. Figure 6 shows the experimental data with the fitted Forbes average saturation and the Forbes/splines calculated local saturation at each limestone wettability conditions. No manual smoothing of the experimental data was performed.

CONCLUSIONS

The effect of absolute permeability could be observed in the twin limestone core plugs at both wettability conditions. The similarity in permeability in the SWW cores demonstrated an excellent match in production curve shape. The different permeabilities in the MOW cores produced large differences in production curve shape, especially the gradient in linearity in the relative permeability dominated parts of production.

The impact of wettability was illustrated by the large difference in equilibrium time, observed production rates and produced volumes. The time to reach equilibrium was larger at oil-wet conditions at lower rotational speeds from the combined effect of viscous and spontaneous displacement of water. When the effect of the spontaneous capillary attraction of oil decreased, the shape of the production curves and the calculated production rates were similar for all wetting states. Lower water saturations were achieved in the moderately oil-wet cores due to less capillary retained water.

Produced volumes of the displaced phase during centrifugation were measured with a high degree of accuracy while the centrifuge was spinning. The automated equipment improved incremental volume measurements by an order of magnitude when compared to human visual readings: to reach the same criteria of equilibrium using the manual visualization method, the laboratory operator would need to wait 10 days with zero observed production at each centrifuge speed. The increased sensitivity to fluid production had a significant impact on the recognition of equilibrium and consequently in the time required to complete the tests.

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Table 1. Rock properties and wettability indices

Core ID	B2	H10	H12	H13	H14
Rock Type	Chalk	Limestone	Limestone	Limestone	Limestone
Abs perm [mD]	3.7	11.6	11.8	10.1	5.7
PV [ml]	32.9	15.8	16.1	16.7	13.3
Wettability index (A-H)	0.19	1	1	-0.2	-0.1
Wetting state	Moderately water-wet	Strongly water-wet	Strongly water-wet	Moderately oil-wet	Moderately oil-wet

Table 2. Time to reach equilibrium in each core at each rotational speed

	B2	H10	H12	H13	H14
Centrifuge Speed (RPM)	Equilibrium time [days]	Equilibrium time [days]	Equilibrium time [days]	Equilibrium time [days]	Equilibrium time [days]
350	4.0	2.1	2.1	2.1	2.1
600	3.1	9.9	9.9	37*)	no equilibrium
900	19	2.8	1.8	2.6	20
1200	25	1.0	2.0	5.0	3.0
1800	27	3.0	3.0	2.0	4.0
2500	15	1.0	2.3	4.1	5.5
3000	4.0	2.5	3.5	3.5	4.6

*) production rates slowed down but did not quite reach equilibrium

Table 3. Net and cumulative water production in two water-wet cores and one moderately water-wet chalk core at each centrifugal speed

	Water-wet cores								
	Strongly water-wet H10			Strongly water-wet H12			Moderately water-wet B2		
Centrifuge Speed (RPM)	Equilibrium time [hours]	Net prod. [PV]	Cum. Prod. [PV]	Equilibrium time [hours]	Net prod. [PV]	Cum. Prod. [PV]	Equilibrium time [hours]	Net prod. [PV]	Cum. Prod. [PV]
350	50	0.00	0.00	50	0.00	0.00	96	0.00	0.00
600	237	0.19	0.19	237	0.19	0.19	74	0.00	0.00
900	68	0.04	0.23	43	0.02	0.21	446	0.03	0.03
1200	24	0.04	0.27	48	0.02	0.23	600	0.06	0.09
1800	72	0.07	0.34	72	0.04	0.27	652	0.17	0.26
2500	24	0.04	0.38	55	0.04	0.31	353	0.12	0.38
3000	59	0.02	0.40	84	0.03	0.34	95	0.12	0.50

Table 4. Net and cumulative water production in moderately oil-wet cores at each centrifugal speed

	Moderately oil-wet cores					
	H13			H14		
Centrifuge Speed (RPM)	Equilibrium time [hours]	Net prod. [PV]	Cum. Prod. [PV]	Equilibrium time [hours]	Net prod. [PV]	Cum. Prod. [PV]
350	50	0.00	0.00	50	0.00	0.00
600	no equilibrium	0.51*)	0.51*)	no equilibrium	0.27*)	0.27*)
900	62	0.05	0.56	476	0.23	0.50
1200	119	0.02	0.58	73	0.02	0.52
1800	48	0.02	0.60	96	0.02	0.54
2500	99	0.02	0.62	132	0.03	0.57
3000	84	0.01	0.63	111	0.02	0.59

*) Produced water until rotational speed increase. Equilibrium not reached within 1000 hours

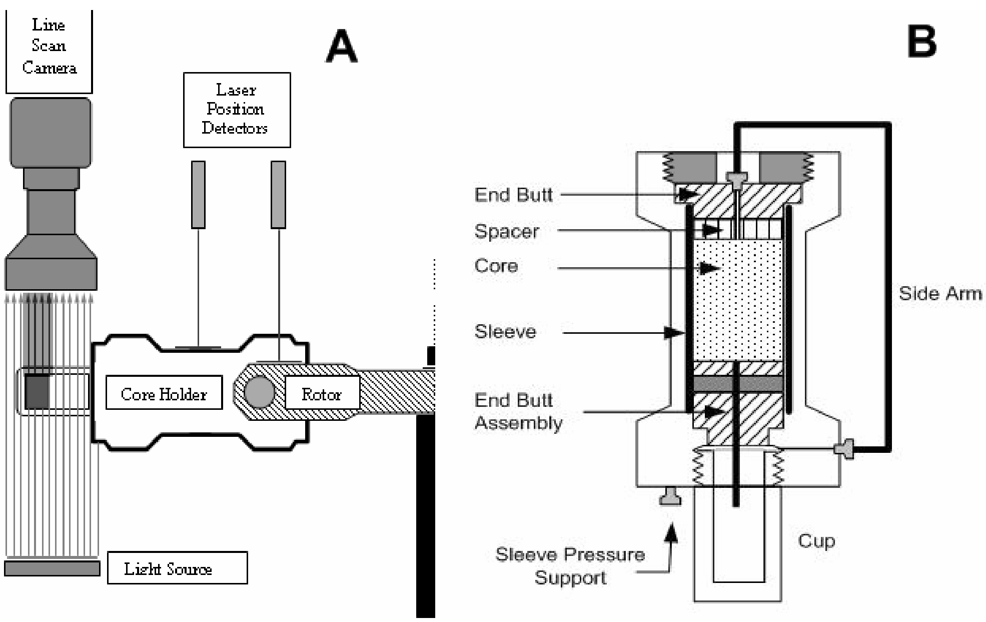


Figure 1. Experimental equipment. A) Automated centrifuge schematic and B) Exploded view of core holder

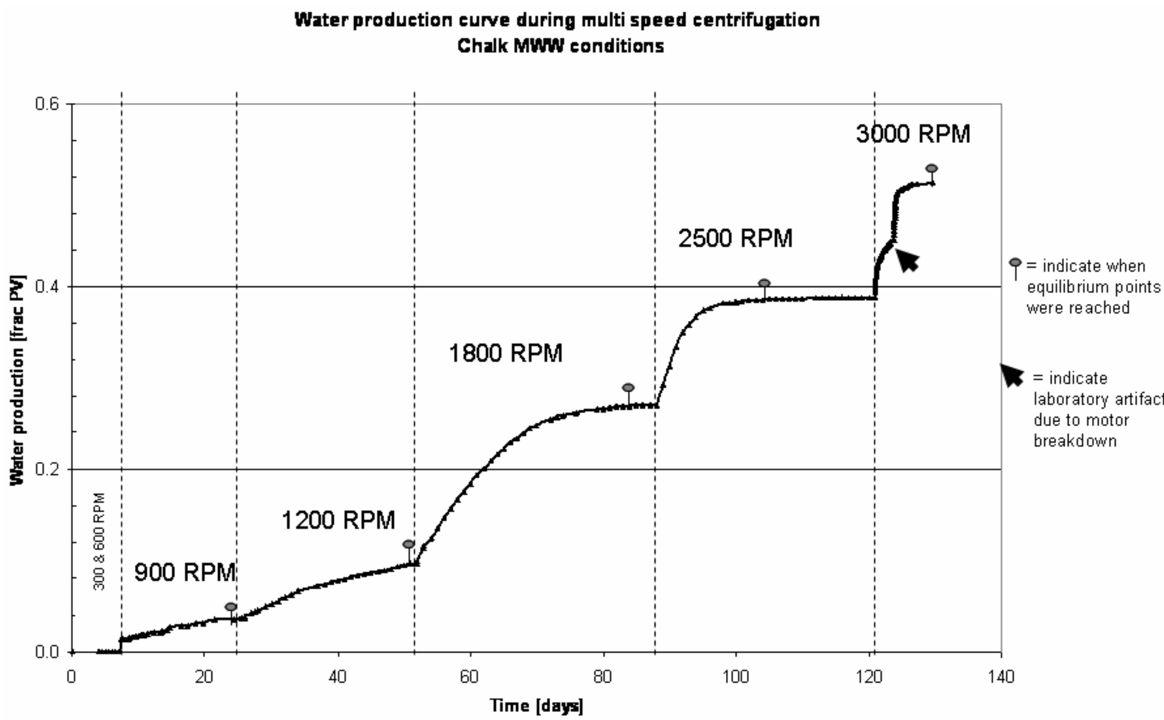


Figure 2. Water production in moderately water-wet chalk during multi-speed centrifugation. Each speed increase is indicated with a vertical dashed line.

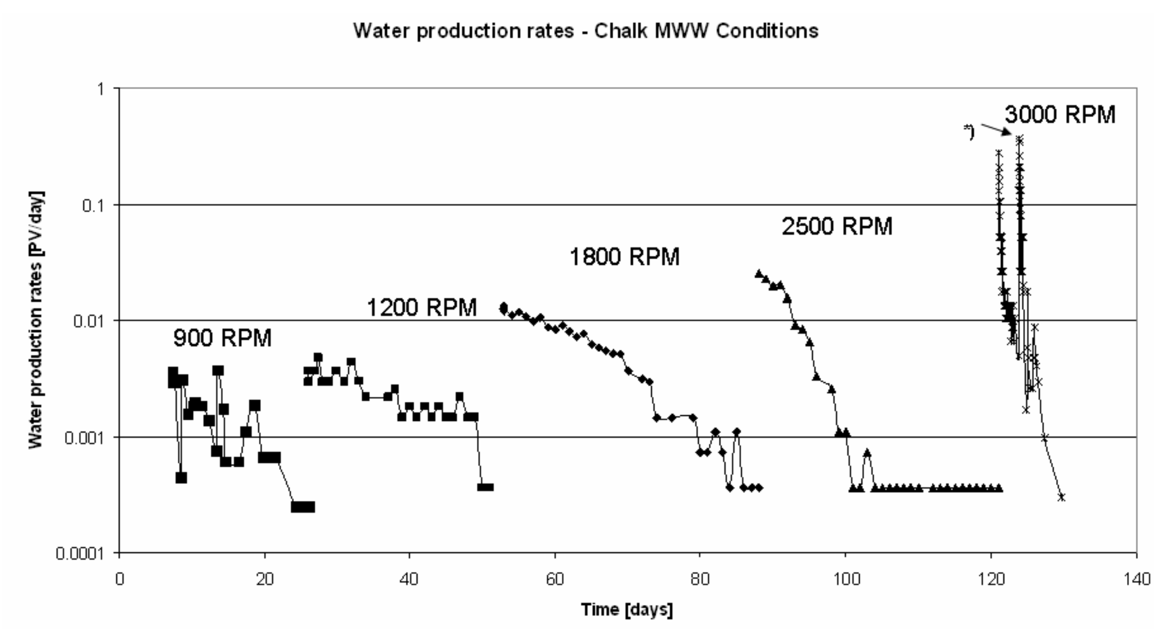


Figure 3. Large production rates associated with an increase in rotational speed. The indicated second peak at 3000 RPM is a laboratory artifact due to motor breakdown.

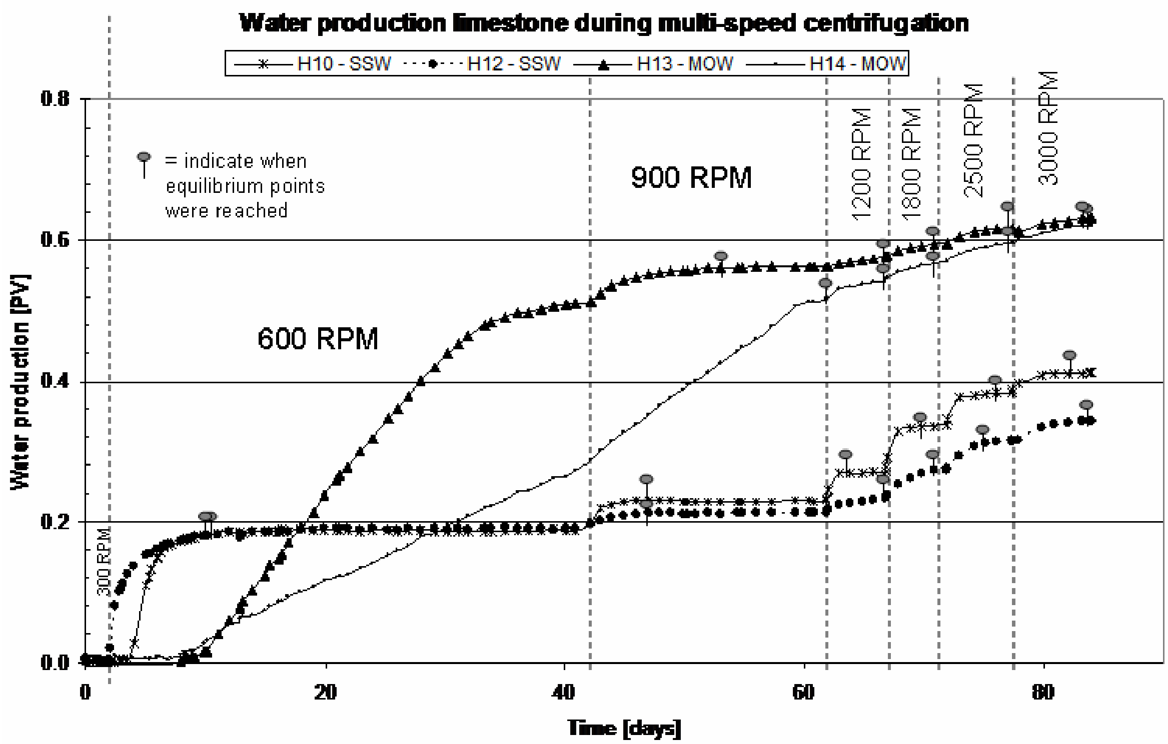
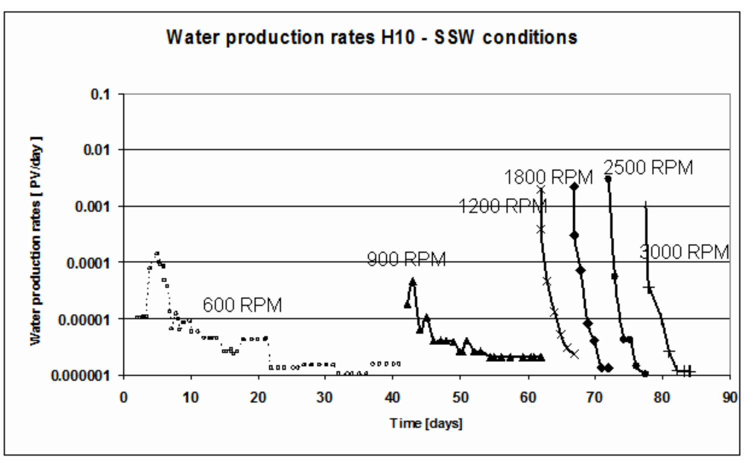


Figure 4. Water production for limestone cores at SWW and MOW conditions. Each speed increase is indicated with a vertical dashed line.

A



B
C
D

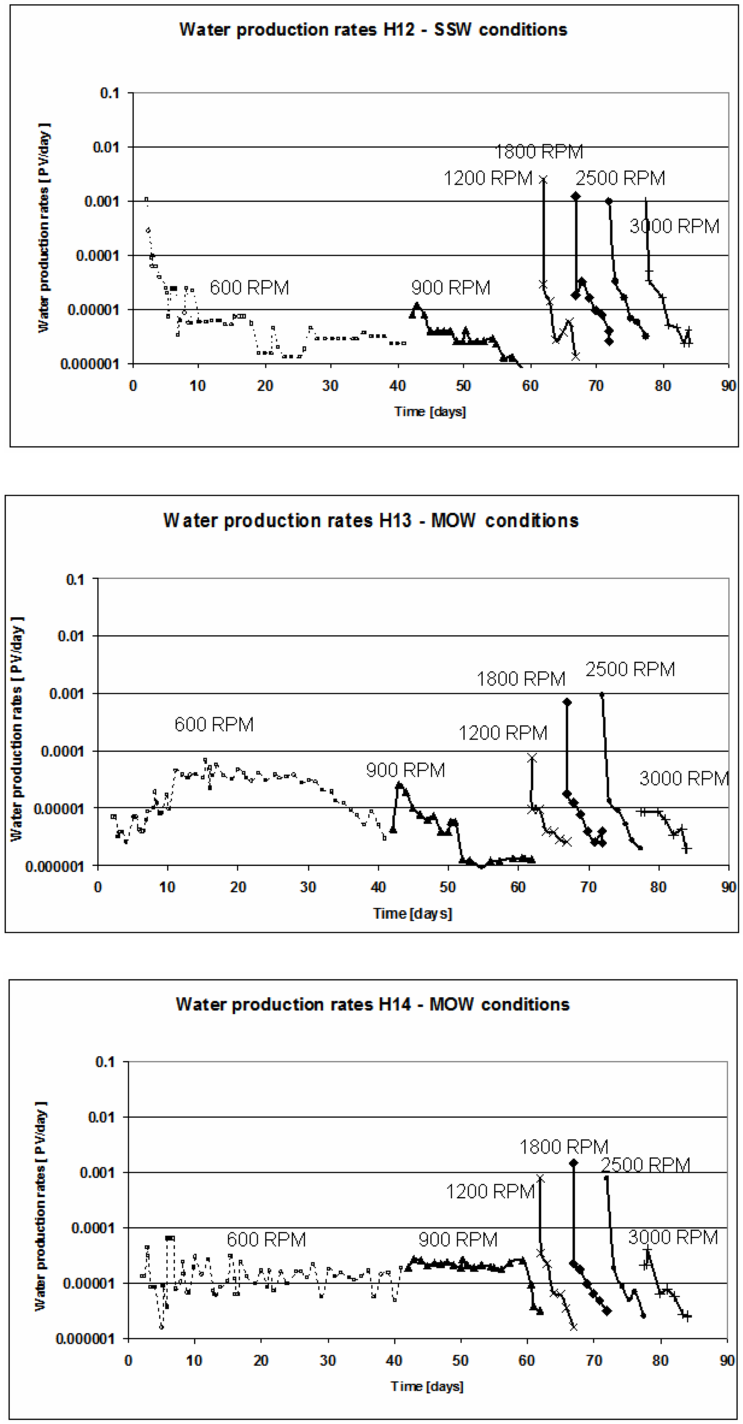


Figure 5. Water production rates observed in limestone core plugs at strongly water-wet (A and B) and at moderately oil-wet (C and D) wettability conditions.

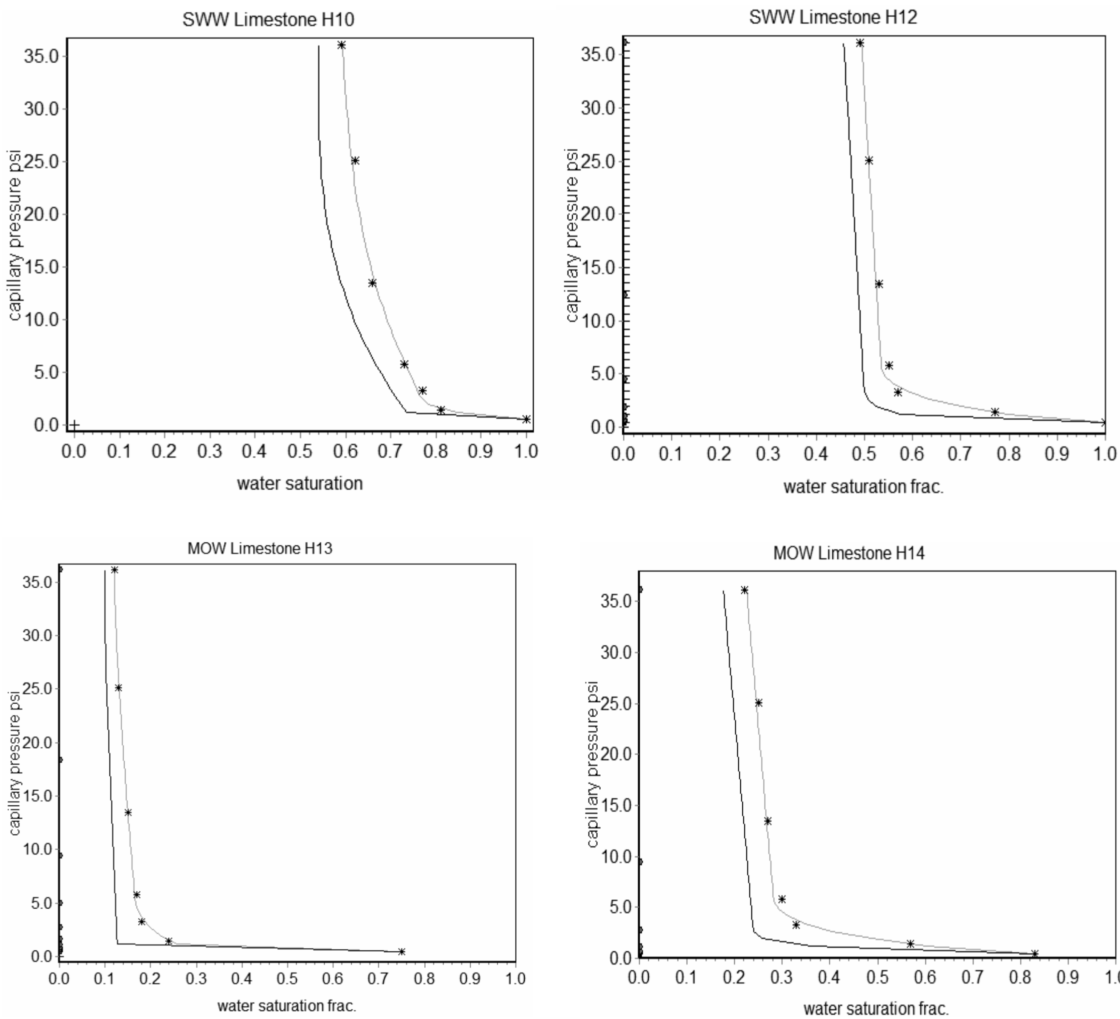


Figure 6. The experimental capillary pressure curve data (points) together with fitted average saturations (gray) and the calculated local saturation using Forbes/splines (black).