A METHOD FOR MEASURING IN-SITU CAPILLARY PRESSURES AT DIFFERENT WETTABILITIES USING LIVE CRUDE OIL AT RESERVOIR CONDITIONS, PART 1: FEASIBILITY STUDY

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ABSTRACT

This paper reports the development and preliminary testing of a method capable of measuring two- and three phase capillary pressures at reservoir conditions with respect to pressure and temperature, using live crude oil.

The method uses nuclear tracers to measure in-situ saturations while the core plugs are spinning in a centrifuge. The apparatus consists of 1) a centrifuge with swinging bucket type core holders, capable of achieving and holding reservoir conditions with respect to pressure and temperature while spinning and 2) an imaging system using nuclear tracers to obtain the in-situ saturations in the core plugs while spinning in the centrifuge.

The advantages of this system compared to existing methods of in-situ measurements of capillary pressures are that 1) crude oil may be used, even at reservoir conditions, 2) capillary end effects are directly measurable, 3) no assumptions are needed to calculate the capillary pressure since the local fluid saturations are directly measured at various capillary pressures and 4) there is no need to solidify one of the phases and remove the cores, as in the direct saturation measurement or DMS method.

The paper describes the experimental method and demonstrates the measuring capability and the reproducibility of the technique. Some preliminary data is compared to capillary pressure curves measured by a standard centrifuge method and by the DMS method with magnetic resonance imaging. The paper reports on the design and construction of the Nuclear Tracer Imaging rig over the centrifuge. A summary and a comparison of other existing techniques are included. Calibrations have been performed and imaging capabilities for the NTI method have been evaluated.

The method may be expanded to measure three-phase capillary pressures for water and live crude oil at reservoir pressure and temperature, by using nuclear tracers to label both liquids. For three-phase measurements two tracers are needed. With some apparatus modifications it will also be possible to measure the positive and the negative parts of the capillary pressure curves, at various wettabilities, for imbibition/waterfloods and drainage processes using crude oil.

INTRODUCTION

The centrifuge has been used to determine capillary pressure curves since the original work reported by Hassler and Brunner nearly half a century ago (ref. 1). However, most of this work has measured the fluid expelled as a function of speed and fit these volumes to a variety of models (ref. 2). Several attempts have been made to measure saturation along the centrifugal axis while the centrifuge was running, but for most of this work, the data has been compromised by the limited number of points measured along the axis and/or the lack of adequate saturation resolution. A direct method of measuring saturation along the centrifugal axis with, at least, one hundred points and a saturation resolution of about 2% pore volume has been reported (ref. 3), but it required the freezing of one phase and removal of the core for saturation measurements.

This work describes a feasibility study to determine whether radioactive tracer technology can provide good saturation resolution, in porous outcrop rocks, while spinning in the centrifuge. The feasibility study included a literature survey (ref. 1, 4-16), hands on training for an existing method of direct measurement of saturation, and calculations of saturation accuracy and spatial resolution for the Nuclear Tracer Imaging (NTI) technique with respect to the collimator geometry. The feasibility study also included the calibration procedures and collection of static and dynamic in-situ saturation data in the centrifuge by the NTI technique. The ultimate goal is to determine capillary pressure curves at reservoir temperature and pressure with live reservoir fluids.

EXPERIMENTAL

The centrifuge was a Beckman J-6B with a rotor that carries four individual core holders. The core holders are capable of holding a confinement pressure up to 300 bars. A schematic drawing of the centrifuge is shown in Figure 1. The centrifuge/rotor has a maximum speed of 3600 rpm, which translates to a maximum capillary pressure of about 3bar for a brine/decane system in a 6cm long plug.

During the feasibility study, the imaging system, shown schematically in Figure 2, includes a sodium iodide scintillation detector that can be moved horizontally along the centrifugal axis of the core plugs while they are being spun in their horizontal orientation. The detector is mounted on top of the centrifuge with a collimator focusing on an adjustable 2 – 4mm cross-section of the cores. In the feasibility study, one of the cores contained a radioactive labeled fluid, in one of the core holders below the detector. Thus no gating of the detector was needed. A steel cylinder with 20 cm thick walls was placed around the detector to minimize signal from background radiation. The sodium iodide detector was a Canberra NaI(Tl) detector with a 2”x 2” crystal. The signal was counted and stored using a Canberra multi-channel analyzer.

A picture of the centrifuge with the detection system in place for measurement is shown in Figure 3. The centrifuge is enclosed in the aluminum bars, which support the detector system. The controls and readout for the centrifuge are seen above the bars, near the wall. The aluminum bars allow the detector, the collimator (seen as a circular drum) with connected wires, to be positioned horizontally. While the centrifuge is operating, the detector needs to be moved horizontally along the centrifugal axis to measure the radioactivity at selected locations along the plug. The upper bars are also for vertical adjustments, to place the collimator as close as possible to the lid of the centrifuge.

The chalk outcrop core plugs used in this study were cored from a larger block of outcrop chalk from the Rørdal quarry in Ålborg, Denmark. The core data is summarized in Table 1. All core plugs were highly water-wet, Iw=1.0. The chalk cores were dried at 90°C for at least seven days before being used.

Before the Pc-measurements were made, the core plugs were vacuum evacuated and saturated with degassed brine containing 5wt% NaCl + 3.8wt% $CaCl_2$. $CaCl_2$ was added to the brine to minimize dissolution of the chalk. Sodium azide, 0.01 wt.%, was added to prevent bacterial growth. The density and viscosity of the brine were 1.05 g/cm^3 and 1.09 cP at 20°C, respectively. The brine was filtered through a 0.45 μm paper filter membrane. The salts used in the brine were: NaCl obtained from Phil Inc. with a purity of 99.5%, $CaCl_2$ obtained from Phil Inc. with a purity of 99.5%. Sodium azide had a purity of 99.5%. The materials were used as received. The physical properties of the fluids are summarized in Table 2. The cores were stored in brine for 5-10 days to reach ionic equilibrium with the rock constituents. Core permeability to brine was measured by using a biaxial core holder with a slight confinement pressure. Due to the fragile nature of the chalk material, net confining pressure was less than 10bar (147 psi), to keep the cores in the elastic compression region.

At 100% brine saturation the core was flushed with 5PV of non-radioactive brine to saturate potential absorption spots for the salts. Then the non-radioactive brine was miscibly displaced by brine containing the appropriate radioactive tracer. For two-phase experiments only one tracer was needed; in the current work Na^{22}, in the form of NaCl, was used to label the brine. Porosity was determined from bulk measurements and the difference in weights before and after saturation.

The fluids used for the Pc-measurements were synthetic brine as the water phase and decane as the oil phase. The radioactive brine had an activity of 2mCi/l. Properties of the fluids are summarized in Table 2.

RESULTS AND DISCUSSION

Figure 4 shows the detailed geometry of the collimator system used for the detection of radioactivity from the core plugs in a centrifuge and defines the nomenclature. Several factors have an impact on the quality of the measurements, the most significant include the collimator slit width, b, and depth, c, the distance from the core to the collimator, d, the plug diameter, D, the activity of the tracer used to label the water phase, and the porosity of the rock material. Clearly the easiest factors to control are those concerning the collimator geometry, thus Table 3 shows the calculated accumulated counts as a function of b, c and d over a reasonable range for each. The dimensions of the viewed area at the front, Fx, and back, Bx, are used to calculate the volume of the plug's porosity, S in %PV, which contributes to the signal for that specific collimator geometry. A lower value for S means a greater resolution along the centrifugal axis, while a greater volume produces higher accumulated counts. The plug diameter, D, is largely dictated by convention and the maximum size that can be used in the centrifuge. Typically plugs are cored with a 2.5 or 3.8 cm diameter. By using these conventional sizes, core plugs collected and used for standard petrophysical tests can also be used in this centrifuge procedure. For this feasibility study 3.8 cm plugs were used. The activity of the radioactive tracer was dictated by cost and safety, typically 1 to 2 mCi/l is acceptable. Porosity was dictated by the geology and morphology of the specific reservoir rock; for these samples it was 45%. The number of radioactive counts is proportional to the water saturation and is the parameter that was determined.

Theoretical Predictions

Table 3 summarizes the theoretical activity, in accumulated counts, for three values of each tested variable in the collimator geometry. The simplified calculations were made for a plug 3.82 cm in diameter, 46% porosity and at 100%PV water saturation. These calculations were made assuming static samples. When the sample is spinning the detector will be exposed to the full diameter of the sample for varying length of time, which will complicate the calculation of absolute accumulated radioactive counts. However, it is expected that the relative relationships between the most significant variables at static conditions will hold for dynamic conditions. By plotting the effect of the individual geometric variables against the accumulated counts it was possible to determine the importance of the individual variables and select those that are most important for optimizing signal and resolution. Based on the requirement that a minimum of 10 measured points along the plug were needed to define the Pc-curve and that the measurements should be completed within 24 hours, the following collimator geometry was selected for the experimental set-up: collimator width: 2mm/4mm, collimator depth 15cm and distance to plug 15 cm, the latter choice allowed the detector to be mounted outside the centrifuge.

Experimental Measurements

The core plugs ISS-1 and ISS-2 were mounted horizontally in the centrifuge during two separate experiments with no rotation, i.e. static conditions. By correlating the in-situ saturation measurements of the centrifuge rig with measurements on the same core plugs using the horizontal flow rig (ref. 17); an indication of the reliability and reproducibility of the measurements could be made.

After replacing the non-radioactive brine that initially saturated the core, with 2mCi/l Na-22 labeled radioactive brine by flushing 5 PV through each core plug, 100% saturation scans were obtained in both rigs. Core plug ISS-1 was then oilflooded to a water saturation of 50% using decane before it was imaged again both in the centrifuge rig and in the horizontal rig. These tests were carried out with two different collimator widths, 0.2 and 0.4 cm. The results of these measurements are shown in Figure 5 and Figure 6, respectively. The scans in Figure 6 show much better agreement between the repeated scans and closer agreement with the horizontal rig scans on the same sample than was observed in Figure 5. There is clearly a difference in reproducibility due to the difference in collimator slit width because of the poorer statistics for the narrower width. By increasing the collimator width the statistics of the measurements are improved at the expense of reduced longitudinal spatial resolution. By increasing the counting time with the 2 mm collimator slit width an improved reproducibility would be obtained, but a limit for the data acquisition of about 24 hours eliminated this possibility for these preliminary experiments.

After the 100% water saturation scan was obtained for core plug ISS-2 the plug was oilflooded, using decane, to an average water saturation of 80%. It was then imaged at Sw=80% before it was drained further to Sw=60% and then imaged again. This was to ensure that the imaging system was able to follow the reduction in water saturation and give consistent water saturation results. The results are shown in Figure 7. There is good agreement between the measured in-situ saturation data and the material balance saturation data. The material balance water saturations were 81% and 63% for the two measurements while the in-situ saturation measurements were 83.4% and 62.8%, respectively. In addition to the good saturation agreement, the scanning method showed that the water and oil were not uniformly distributed along the longitudinal axis of the plug, information that can only be obtained by a method that determines saturation at specific spatial locations.

Fig. 8 shows a preliminary primary drainage Pc-curve obtained for the outcrop chalk plug denoted ISS-3. The results are compared to data for similar strongly-water-wet Portland chalk obtained by a conventional centrifuge method (ref. 18) and by the DMS-method (ref. 19). Satisfactory results are obtained although the primary drainage curve for ISS-3 was obtained using an 8cm long collimator slit, which gave reduced spatial resolution of the saturation measurements, due to the curvature of the core trajectory. The results are also less accurate due to the fact that a NaI-scintillation detector was used during this feasibility study, rather than a more expensive detector with higher energy resolution. This would increase the accuracies of the saturation measurements, since the background radiation then would have less impact on the results.

CONCLUSIONS

- Using the Nuclear Tracer Imaging Technique, a manually operated detector system for direct measurement of in-situ saturation from cores while spinning in a centrifuge, has been designed, constructed and put into operation.
- Geometrical considerations and calculations, as well as static evaluations of the pertinent experimental parameters on a theoretical and experimental basis, have been completed.
- The encouraging results in this feasibility study strongly suggest that further work should be done to develop this technique to its full potential.

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NOMENCLATURE

b - collimator width
c - collimator depth
d - distance from the collimator to the core holders
D – plug diameter
Sw – water saturation

REFERENCES

1. Hassler, G.L. and Brunner, E.: "Measurement of Capillary Pressure in Small Core Samples", Trans. AIME, Vol. 160, pp114-23, 1945.
2. Ruth, D. and Chen, Z.: "Measurement and Interpretation of Centrifuge Capillary Pressure Curves – The SCA survey Data", The Log Analyst, Sept.-Oct. pp 21-33, 1995.
3. Baldwin, B.A. and Spinler, E.A., "A Direct Method for Simultaneously Determining Positive and Negative Capillary Pressure Curves in Reservoir Rock", J. Petrol. Sci. and Engr. Vol. 20, pp161-5, 1998.
4. M. Fleury, G. Ringot and P. Poulain, 1999 "Positive imbibition capillary pressure curves using the centrifuge technique*",* Society of Professional Well Log Analysts; Society of Core Analysts, International Symposium Proceedings; paper SCA-9913, 1-11
5. Fleury, M., Egermann, P., and Goglin, E., 2000, "A model of capillary equilibrium for the centrifuge technique", paper 2031, in 2000 International symposium proceedings: Society of Core Analysts, CD-ROM, 12 p.
6. Ruth, D., and Forbes, P., 2000, "An automatic-optimization data analysis technique based on Forbes' second method", paper 2022, in 2000 International symposium proceedings: Society of Core Analysts, CD-ROM, 11 p.
7. Forbes, P., 1997, "Centrifuge data analysis techniques--an SCA survey on the calculation of drainage capillary pressure curves from centrifuge measurements", paper SCA-9714, in 1997 international symposium proceedings: Society of Professional Well Log Analysts, Society of Core Analysts Chapter-at-Large, 19 p.
8. Sarwaruddin, M., Torsaether, O., and Skauge, A., 2000, "Comparing different methods for capillary pressure measurements", paper 2051, in 2000 International symposium proceedings: Society of Core Analysts, CD-ROM, 5 p.
9. Spinler, E.A, Baldwin B. A., Graue, A.: "Simultaneous Measurement of Multiple Capillary Pressure Curves from Wettability and Rock Property Variations Within Single Rock Plugs", Reviewed Proc.: 1999 International Symposium of Core Analysts, Golden, Co., USA, Aug., 1999.

10. Torsæter, O.:"Determination of Positive Imbibition Capillary Pressure Curves by Centrifuging", The 3rd International Symposium on Evaluation of Reservoir Wettability and Its Effect on Oil Recovery, Laramie, Wy., Sept.21-23,1994.
11. M. Ding and O. Torsaeter, and Jelmart, T.A., 1999 "Developments in centrifuge measurements of relative permeability and capillary pressure hysteresis", Society of Professional Well Log Analysts; Society of Core Analysts, International Symposium Proceedings; paper SCA-9943, 1-10
12. H. Hermansen, O. Eliassen, Y. Guo and S. M. Skjaeveland, 1991 "Capillary pressure from centrifuge--a new direct method", Gordon and Breach Science Publishers, Philadelphia; Advances in core evaluation II, reservoir appraisal (Society of Core Analysts, 2nd European Core Analysis Symposium Proceedings); 453-468
13. Fleury, M., Doevle, M., and Longeron, D., 1997, "Full imbibition capillary pressure measurements on preserved samples using the micropore membrane technique", paper SCA-9716, in 1997 international symposium proceedings: Society of Professional Well Log Analysts, Society of Core Analysts Chapter-at-Large, 10 p.
14. O'Meara Jr., D.J., Hirasaki, G.J. and Rohan, J.A.: "Centrifuge Measurements of Capillary Pressure: Part 1 – Outflow Boundery Condition", SPE number 18296, SPE Reservoir Engineering, February 1992.
15. O'Meara Jr., D.J., Hirasaki, G.J. and Rohan, J.A.: "Centrifuge Measurements of Capillary Pressure: Part 2 – Cavitation", SPE number 18296, Presented at the SPEATCE in Houston, Texas, October 1988.
16. O'Meara Jr., D.J. and Crump, J.G.: "Measuring Capillary Pressure and Relative Permeability in a Single Centrifuge Experiment", Paper presented at the SPEATCE in Las Vegas, Nevada, September 1985. SPE number 14419.
17. Graue, A., Bognø, T., Baldwin, B.A., Spinler, E.A.: "Wettability Effects on Oil Recovery Mechanisms in Fractured Reservoirs", SPE Res. Eval. and Eng., Vol. 4, No. 6, (455-466), Dec. 2001.
18. Graue, A., Bognø, T., Moe, R.W., Baldwin, B.A., Spinler, E.A., Maloney, D., Tobola, D.P.: "Impacts of Wettability on Capillary Pressure and Relative Permeability", SCA9907, Reviewed Proc.: 1999 International Symposium of Core Analysts, Golden, Co., USA, Aug. 1-4, 1999.
19. Bognø, T., Graue, A., Spinler, E.A., Baldwin, B.A.: "Comparison of Capillary Pressure Measurements at Various Wettabilities Using the Direct Saturation Measurement Method and Conventional Centrifuge Techniques", accepted for presentation at the 2001 International Symposium of Core Analysts, Edinburgh, Scotland, Sept 17-19, 2001.

Table 1. Summary of Portland chalk core plug data

CORE PLUG DATA			
Core Label	ISS - 1	ISS - 2	ISS - 3
Diameter	3,82	3,82	3,82
Length	6,03	6,03	6,05
Area	11,46	11,46	11,46
Dry Weight	98,00	98,40	102,30
After drying	97,80	98,10	102,10
Wet Weight	132,00	132,20	134,80
Bulk Volume	69,11	69,11	69,34
Pore Volume	32,38	32,19	30,95
Porosity	46,85	46,58	44,64
Core Label	ISS - 1	ISS - 2	ISS - 3
Rate [ml/hr]	60,00	60,00	60,00
Pressure [bar]	2,64	2,76	3,54
Permeability [mD]	3,67	3,51	2,74
Rate [ml/hr]	120,00	120,00	120,00
Pressure [bar]	4,43	5,38	6,94
Permeability [mD]	4,37	3,60	2,79
Rate [ml/hr]	40,00	40,00	40,00
Pressure [bar]	1,76	1,87	2,26
Permeability [mD]	3,67	3,45	2,86
Average Perm. [mD]	3,90	3,52	2,79
Density without water	1,42	1,42	1,48
Density with water	1,91	1,91	1,94

Table 2. Fluid Properties

Fluid	Density [g/cm^3]	Viscosity [cP] at 20^{o}C	Composition
Brine	1,05	1,09	5 wt% NaCl + 3.8 wt% $CaCl_2$
n-Decane	0,73	0,92	

Table 3. Simplified radiation statistics calculations at various collimator geometries

b_x [cm]	c [cm]	d [cm]	F_x	B_x	S in % PV	Activity [mCi/l]	Accumulated counts
0,2	15,0	15,0	0,6	0,7	10,85	1	126593
0,2	25,0	15,0	0,4	0,5	7,84	1	91517
0,2	50,0	15,0	0,3	0,4	5,59	1	65210
0,5	15,0	15,0	1,5	1,8	27,12	1	791205
0,5	25,0	15,0	1,1	1,3	19,60	1	571983
0,5	50,0	15,0	0,8	0,9	13,97	1	407566
0,8	15,0	15,0	2,4	2,8	43,39	1	2025486
0,8	25,0	15,0	1,8	2,0	31,37	1	1464276
0,8	50,0	15,0	1,3	1,4	22,35	1	1043368
0,2	15,0	10,0	0,5	0,6	8,62	1	202962
0,2	25,0	10,0	0,4	0,4	6,51	1	153155
0,2	50,0	10,0	0,3	0,3	4,92	1	115800
0,5	15,0	10,0	1,2	1,4	21,56	1	1268515
0,5	25,0	10,0	0,9	1,1	16,27	1	957221
0,5	50,0	10,0	0,7	0,8	12,30	1	723750
0,8	15,0	10,0	1,9	2,3	34,50	1	3247399
0,8	25,0	10,0	1,4	1,7	26,03	1	2450485
0,8	50,0	10,0	1,1	1,2	19,68	1	1852799
0,2	15,0	0,0	0,2	0,3	4,18	1	3841754
0,2	25,0	0,0	0,2	0,3	3,84	1	3530493
0,2	50,0	0,0	0,2	0,2	3,59	1	3297046
0,5	15,0	0,0	0,5	0,8	10,45	1	24010965
0,5	25,0	0,0	0,5	0,7	9,60	1	22065579
0,5	50,0	0,0	0,5	0,6	8,97	1	20606540
0,8	15,0	0,0	0,8	1,2	16,72	1	61468070
0,8	25,0	0,0	0,8	1,0	15,37	1	56487882
0,8	50,0	0,0	0,8	0,9	14,35	1	52752741

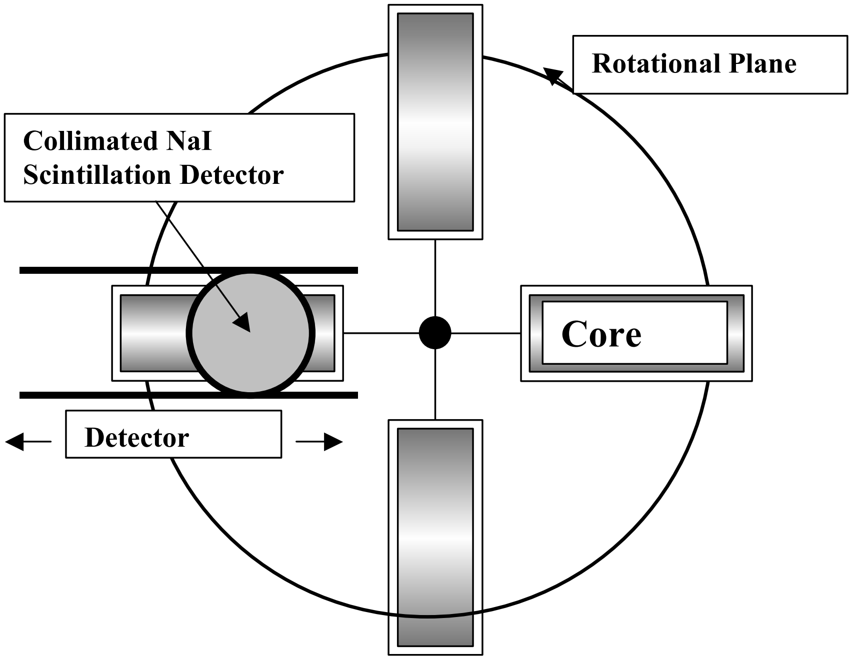


Figure 1. Schematics of centrifuge - looking down into the centrifuge the detector will move in the radial direction and measure the in situ saturation along the long axis of all the four core plugs.

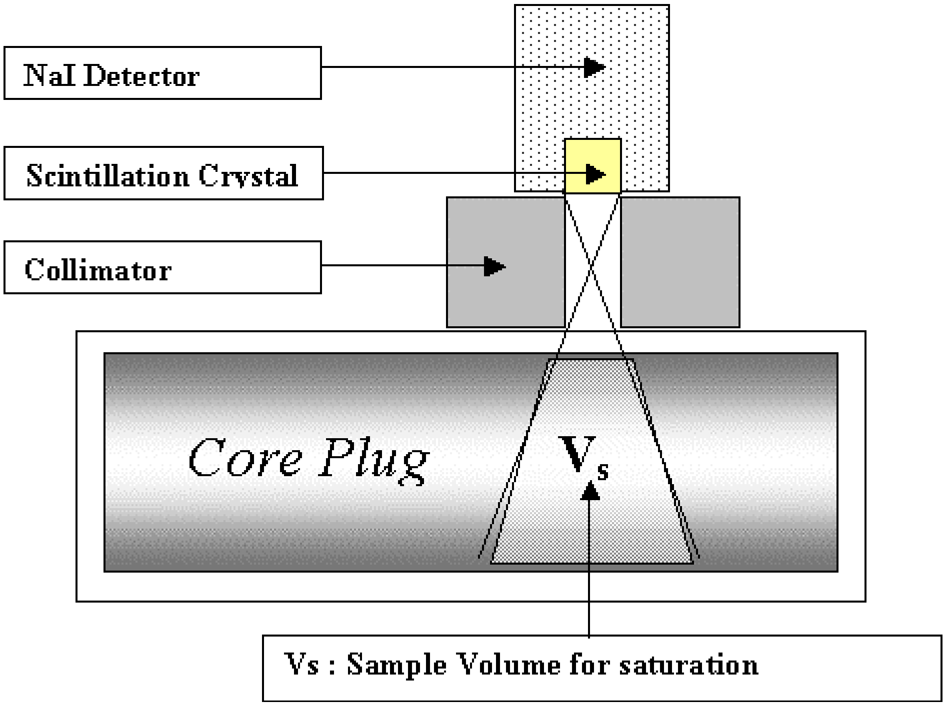
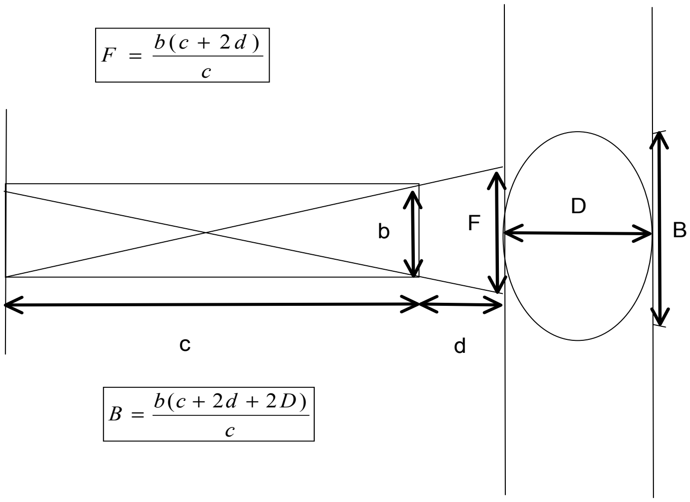


Figure 2. Schematics of the imaging system. Sample volume V_s providing the saturation information for each position along the long axis of the core is indicated.

Figure 3. Centrifuge placed within imaging system.



$$F = \frac{b(c+2d)}{c}$$

$$B = \frac{b(c+2d+2D)}{c}$$

F= Front area of sample volume
B= Back area of sample volume
D= Core diameter
d= distance from end of collimator to top of core holder
b= collimator width
c= collimator depth

Figure 4. Schematics of the geometry for the detector collimator.

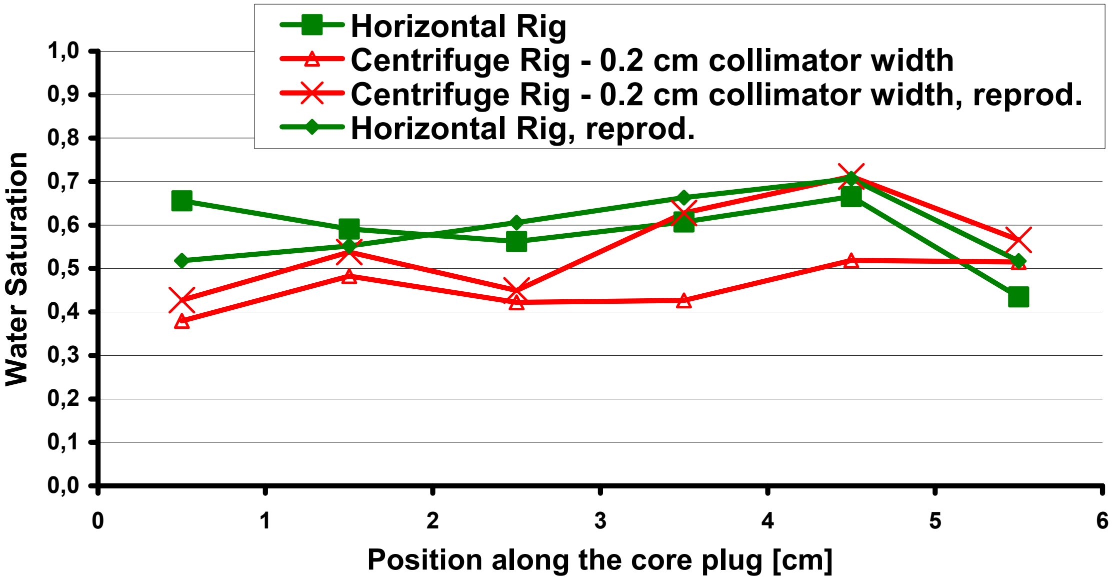


Figure 5. Reproducibility of saturation measurements with 0.2cm collimator width.

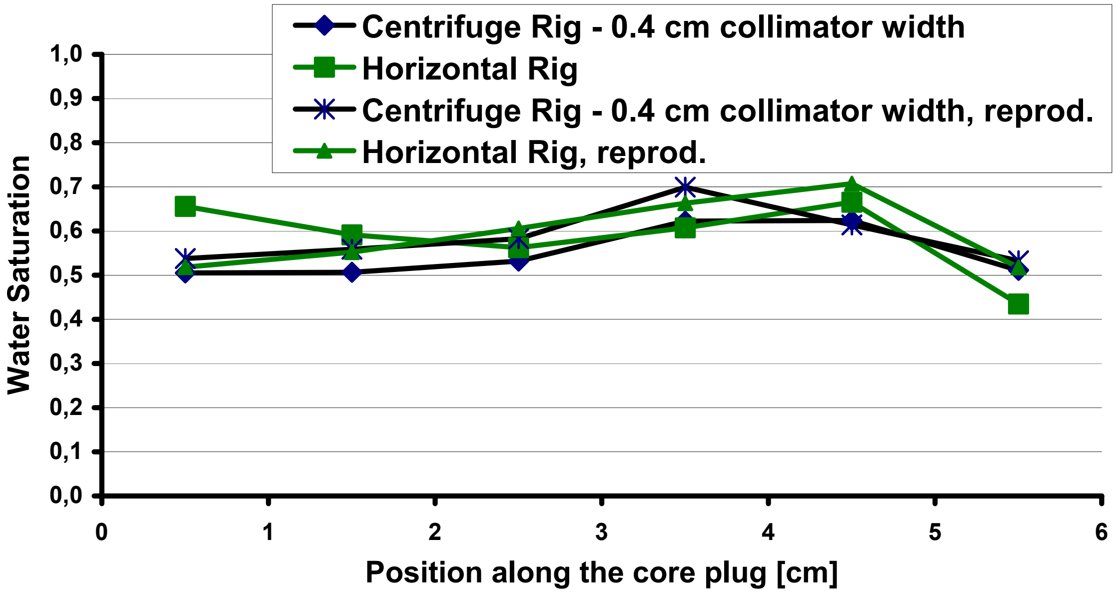


Figure 6. Reproducibility of saturation measurements with 0.4cm collimator width.

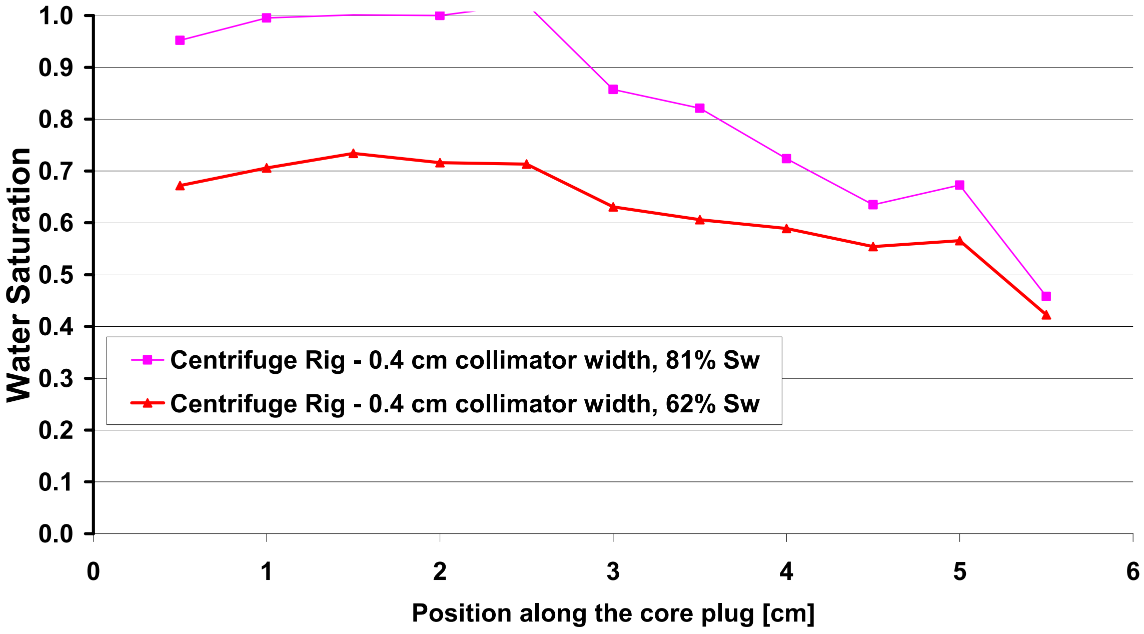


Figure 7. Consistency of in-situ water saturations at Sw = 80% and Sw = 60%.

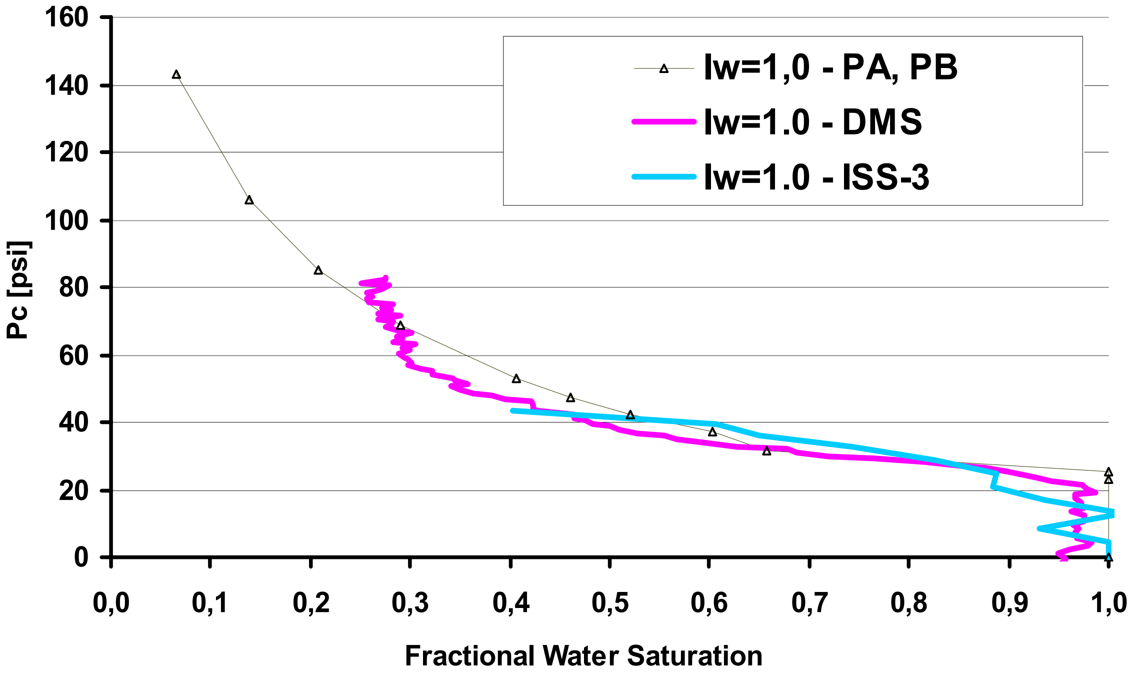


Figure 8. Primary drainage Pc-curve for strongly water-wet Portland chalk.