A NEW WATERFLOOD INITIALIZATION PROTOCOL FOR PORE-SCALE MULTIPHASE FLOW EXPERIMENTS

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ABSTRACT

In the context of digital rock analysis, pore-scale imaging of multiphase flow experiments using X-ray microtomography can be used to obtain fundamental insights into pore-scale displacement physics. This provides a basis to better calibrate numerical pore-scale simulators, or it can be used to understand local fluid distributions, while simultaneously measuring average properties, equivalent to a traditional SCAL experiment.

Imaging studies in the literature have historically been conducted on water-wet micro-plugs, using kerosene, or another refined oil, as the non-wetting phase. Prior to conducting waterflood experiments, the initial water saturation has been established by dynamic flooding. The disadvantage with this is that a non-uniform saturation profile is established due to the capillary end effect. This will result in a higher average initial water saturation compared with, for instance, standard SCAL techniques such as the porous plate method or centrifugation.

In this paper, a methodology for initializing multiple micro-plugs to connate water saturation has been developed by adopting best SCAL practices, namely the porous plate method or centrifugation using crude oil, followed by ageing. We drill multiple micro-plugs from a full size SCAL core sample, without losing capillary continuity with the base of the original sample. In the example presented, for Bentheimer sandstone, the initial saturation was established using centrifugation. The experiment is designed to prevent a non-uniform saturation profile in the micro-plugs. We use *in situ* imaging to determine the water saturation after primary drainage and show that it is indeed uniform across the micro-plug with a value consistent with large-scale SCAL measurements and the measured mercury injection capillary pressure. We also show that a significant wettability alteration had occurred by measuring *in situ* contact angles.

INTRODUCTION

Flow experiments using X-ray micro-tomography (also called micro-CT) are commonly used to gain insights into rock properties and pore scale displacement physics [1-12], to calibrate numerical simulators [13-19], or to directly determine wetting properties by measuring the *in situ* contact angles [20-22]. These flow studies have generally used water-wet rocks, where the initial water saturation was established by flooding, and the waterflood experiments were conducted with inert model fluids. Recently, some experiments have been conducted with crude oil to obtain a representative reservoir wettability [23-24]. This requires preparation of micro-plugs, a few mm in diameter and 2-2.5 cm length.

The procedure by which samples are prepared, including oil injection to connate water saturation (S_{wc}), is expected to have a major impact on ageing. The initial water film thickness in the pore space affects the ageing potential of a given crude oil–rock–brine combination [25]. With thick water films or layers, the rock is expected to remain more water-wet [26], preventing any direct interaction between crude oil and rock. For thinner water films, polar crude oil components can either diffuse through the water and adsorb on the rock, or the film becomes unstable due to attractive electrostatic forces between rock/brine and brine/crude oil interfaces, which brings crude oil directly into contact with the surface [26]. During a primary drainage process, the water distribution is controlled by the capillary pressure [27]. For low (connate) water saturation, the film thickness is controlled by the disjoining pressure as illustrated in Figure 1. Depending on the salinity of the water phase and other parameters, the water film thickness ranges between 100 nm and 1 nm [28], which is below the imaging resolution of micro-CT scanners (which is about 1 μm). Such thin films have practically negligible hydraulic conductivity compared to the corner menisci (layers) which are much thicker and have long-range connectivity. The corner menisci provide the main hydraulic conductivity for the water phase at low water saturation [29].

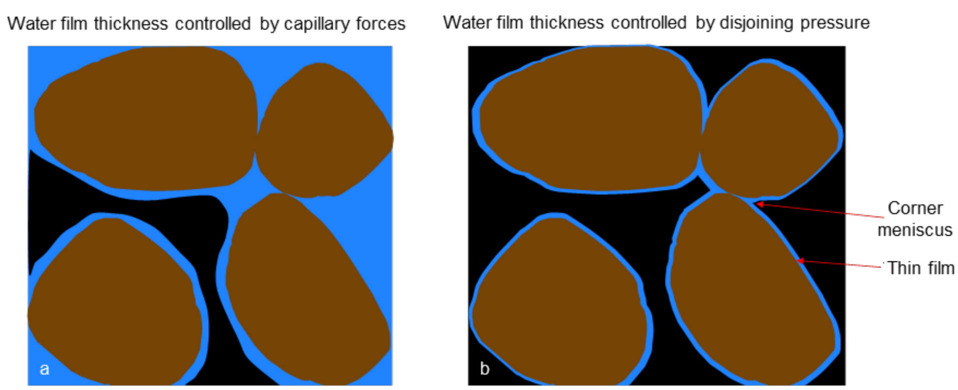

Figure 1. (a) Water film thickness controlled by capillary pressure. (b) Water film thickness controlled by disjoining pressure.

An initial water saturation can be established by the porous plate method, centrifugation, or by dynamic displacement [30]. Most oil companies recommend the porous plate method or centrifugation as the standard for SCAL. Dynamic flooding is not usually recommended, since the capillary end effect is likely to lead to a non-uniform saturation distribution in the sample.

The porous plate method is a direct technique, where a water-saturated core and a ceramic plate are installed in an isostatic core holder under stress [30-31]. The purpose of the ceramic plate is to prevent the non-wetting phase being produced during primary drainage. Primary drainage is conducted by imposing a capillary displacement pressure limited by the entry pressure of the ceramic plate. The only disadvantage of the method is that it is time consuming. This approach should not be mistaken with the large porous plate technique where multiple samples are placed on a large porous plate in a pressure chamber without stress [30-31]. The centrifugation method [32] is an alternative technique in which oil is forced to flow into a core at a specified rotational speed. This method is fast and provides an effective method to reach connate water saturation for permeable core plugs. However, for low permeability rocks, there may be a noticeable saturation profile in the core plug at the end of the experiment [30]. For the dynamic displacement method, oil is injected at constant pressure or rate to displace water in the sample. The main disadvantage is that drainage core floods are affected by the capillary end effect, which cannot easily be prevented, resulting in a non-uniform and higher average initial saturation than other methods [30].

In micro-CT flow experiments, dynamic displacement by high flow rate flooding is currently the prevailing approach for pore-scale imaging. The initial wetting phase saturation is established by injecting the non-wetting phase (crude oil) into the core at an incrementally increasing flow rate [33], followed by *in situ* ageing to restore rock wettability inside the core holder at reservoir conditions for about four weeks [24]. The main limitations for this method are two-fold. Firstly, the established initial water saturation is not necessarily the connate water saturation. Furthermore, the distribution of initial water saturation, from inlet to outlet end, is non-uniform in most cases. This is mainly caused by the capillary end effect where the wetting phase is retained near the outlet of the sample. Consequently, water films are too thick to allow for efficient ageing [27] and the wettability distribution may not be representative, particularly tending to under-state the degree of wettability alteration. The results from micro-CT flow experiments therefore cannot be directly compared with SCAL measurements. Secondly, this method is time consuming since it occupies flooding loop equipment throughout flooding and ageing.

In this paper, a methodology for initializing micro-plugs for flooding experiments has been developed by adopting best SCAL practices. Multiple mm-scale micro-plugs were drilled from a 1.5-inch (38 mm) core without breaking capillary continuity between the distinct micro-plugs and the base of the core. The benefit of this approach is a systematic and repeatable way of establishing S_{wc} for all the micro-plugs. This protocol is aligned with SCAL practice, which can be used as validation for the pore-scale experiments. In addition, it becomes possible to prepare a large number of samples with the same properties, given that the rock is homogenous, for micro-CT flow experiments. By deploying this protocol, up to 45 micro-CT samples can be prepared simultaneously in the same centrifuge batch, followed by ageing. A strict protocol needs to be followed with respect to mounting/dismounting the micro-plugs in sleeves and core holders prior to waterflooding experiments in the micro-CT apparatus. The only disadvantage of the new initialization protocol is that dry scans of the micro plugs cannot be obtained at the beginning of the experiment. If a dry scan is needed to assist segmentation, or needed as an input for modelling, it needs to be obtained at the end of all flow experiments after re-cleaning.

The water saturation was measured by imaging. The saturation was uniform along the core with an average value of 8%, consistent with independent SCAL experiments. We also show that a significant wettability alteration had occurred by measuring *in situ* contact angles. In contrast, dynamic flooding in similar micro-plugs resulted in an average saturation of 14%.

MATERIALS AND METHODS

Rock samples and fluid properties

The rock sample used in this study was Bentheimer sandstone (containing 98% quartz, 1% kaolinite/chlorite and 1% microcline), the average porosity was 0.24, and the brine permeability was 2.4 Darcy. For the initial primary drainage step, the brine was comprised of deionised water with various salts added, see Table 1. The sample was aged using crude oil from a producing field in the Middle East, supplied by Shell Global Solutions International BV.

The aged mini-cores were then extracted from the larger core under crude oil (see later). The crude was replaced by refined oil, followed by waterflooding. For these experiments a doped brine was used to enhance the phase contrast for imaging; we added potassium iodide (KI). The oil phase was decalin (cis and trans decahydronaphthalene mixture with a dynamic viscosity of 3 mPa·s at 20 °C, supplied by Alfa Aesar). We performed a steady-state waterflood experiment to assess the wettability alteration. The viscosity of the oil, μ_o was 3 times higher than the brine.

Table 1. Ion concentration for the brines used.

	Ion concentration (g/l)	
	Brine	Doped brine
Na^+:	4.27	4.27
K^+:	7.24	8.22
Ca^{2+}:	0.30	0.30
Mg^{2+}:	0.02	0.02
Cl^-:	13.74	7.12
I^-:	0	26.99

Preparation of the micro-plugs

Standard size core samples were cleaned and saturated with formation brine. Permeability and porosity can also be measured on these samples. The micro-CT samples were drilled to approximately 2.4 cm length from a larger core 3.8 cm diameter and 4 cm long. The diameter of the micro-CT cores used in this study was 6 mm. Figure 2 shows the samples, which remained attached to the base of the original sample.

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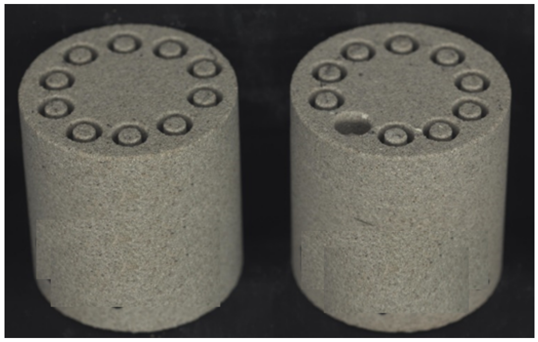

Figure 2. Drilled micro-plugs. The plugs were 2.4 cm long and remained attached to the base of the sample which retains capillary continuity during primary drainage.

Then the entire arrangement was flooded with crude oil [30]. In this study, centrifugation was used. Since the micro-plugs were attached to the core base during centrifugation, non-uniformities in saturation were largely confined to the 1.6 cm at the base of the rock that was not part of the micro-plugs. For low permeability material it is therefore important to break capillary continuity as soon as centrifugation is completed, before continuing with ageing, to prevent re-distribution caused by releasing capillary pressure. In this case, using high-permeability Bentheimer, this was not necessary.

Primary drainage

Speed design for centrifugation, or capillary displacement pressure design for the porous plate method is based on converting mercury injection capillary pressure (MICP) measurements to equivalent oil/brine systems. In this study, for the centrifuge, two capillary displacement pressures were used. The centrifuge speeds are listed in Table 2. From mercury injection tests and SCAL experiments, it was demonstrated that connate water saturation is reached at a capillary pressure of approximately 20 kPa. Therefore, a maximum speed of 3200 rpm, which represents an imposed capillary pressure of 60 kPa, was sufficient to reach S_{wc}.

The crude oil injected into the system (TAN = 0.09 mgKOH/g, TBN = 0.270 mg/g, SARA analysis: Sat=44.0, Aro=44.0, Res=9.69, Asp=2.31) had a density of 0.85 g/cm^3 and a viscosity around 8-10 mPa.s at room temperature.

Ageing

After completing centrifugation, the entire rock assembly with multiple micro-CT cores was submerged in the same crude oil. The sample was kept in crude oil at 3 MPa pressure and 80°C for four weeks to complete ageing.

Table 2. Centrifuge speeds and saturation for primary drainage.

Centrifugation rpm	S_w from centrifugation
0	1
1000	0.432
3200	0.078

VALIDATION OF THE INITIALIZATION PROTOCOL

This initialization protocol was validated using the following criteria:

1. Confirming by imagining that S_{wc} is uniform along the entire micro-plug length.
2. Verifying that the average S_{wc} from the micro core is consistent with S_{wc} from SCAL experiments, or with mercury injection measurements.

In addition, to demonstrate that wettability alteration occurred, a steady state experiment waterflood experiment was performed on a micro-plug sample, and compared to an experiment where initial conditions were established by dynamic flooding without ageing [5].

Experimental procedure for waterflooding experiments

The experiment was conducted using the following procedure:

1. A micro-CT core (6 mm in diameter and 24 mm in length) was taken from the larger core. The micro-plugs were easily broken off from the larger core. The small sample was then loaded into a micro-CT core holder surrounded by crude oil.
2. A confining pressure of 2 MPa was applied and maintained within the cell to compress the Viton sleeve around the core sample to avoid fluid bypass.
3. With a back pressure of 3 MPa, decalin was injected to replace crude oil in the system.
4. Decalin was injected at a flow rate of 0.03 ml/min ($f_w = 0$). After reaching steady state, which was indicated by differential pressure transducer measurements, scans were taken to obtain the initial water saturation.
5. Brine and oil were both injected at the same time with a water fractional flow $f_w = 0.5$, with a total flow rate of 0.03 ml/min. Injection continued until steady state was achieved indicated by a stable pressure differential. Scans were taken after steady state was reached.
6. The differential transducer used in this study was a Keller PD-33X, with an accuracy of ±0.3 kPa. A back pressure of 3 MPa was applied throughout the entire experiment.

All the scans were taken using a Zeiss Versa 510 with a flat panel detector. Before waterflooding the entire sample was imaged at a voxel size of 6.6 μm. At $f_w = 0.5$, a scan with a smaller voxel size (3.58 μm) was taken to characterise the wettability. The wettability characterisation was compared with an unaged sample at steady state, again with $f_w = 0.5$.

Initial saturation

The results of the image segmentation and the saturation profile for each slice (6.6 µm per slice) of the oil phase for the sample prepared following the procedure above are shown in Figure 3. A uniform saturation can be observed and the average water saturation for the sample is 0.082 (computed from the entire volume). This compares well with the average for all the micro-cores plus the larger core base of 0.078 obtained after centrifugation. Figure 4 shows the comparison between the average S_{wc} from the micro-core with MICP.

Using the flooding method to establish initial saturation in a Bentheimer micro-core gave an average value of 0.14 [5] which, as expected, is higher than that established with our new protocol. The value we obtain is, however, similar to that found on larger core samples following standard SCAL protocols: in three replicate experiments on entire 38-cm cores S_{wc} values of 0.094, 0.080 and 0.068 were obtained, consistent with our values.

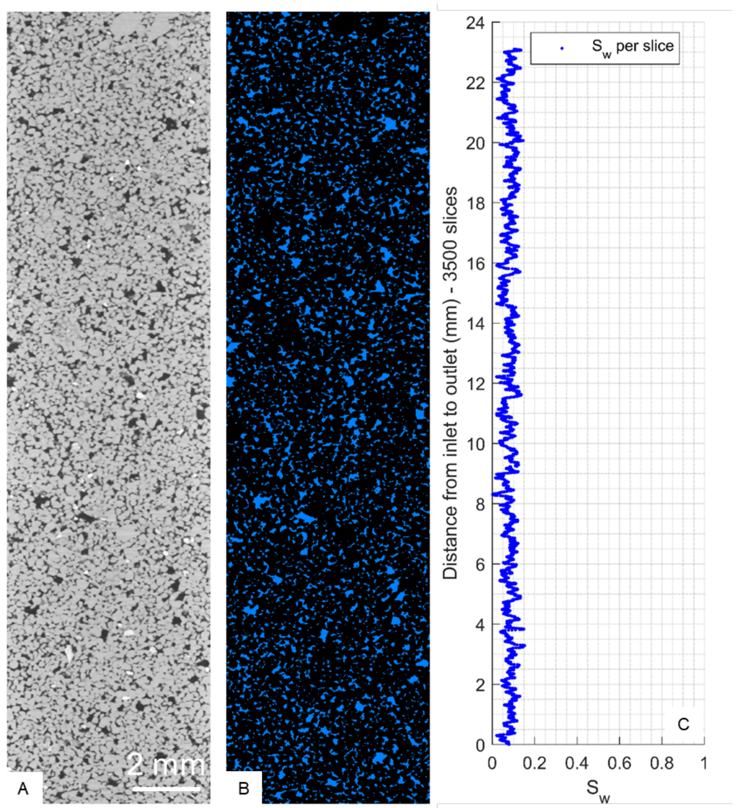

Figure 3. Tomographic image of a Bentheimer micro plug initialized to S_{wc}, following the protocol described in this paper. (A) Grey-scale two-dimensional cross-section of the three-dimensional image of the sample after applying a nonlocal means edge preserving filter. (B) Segmented oil phase shown in blue. (C) The saturation per slice along the direction of flow. The thickness for each slice is 6.6 µm.

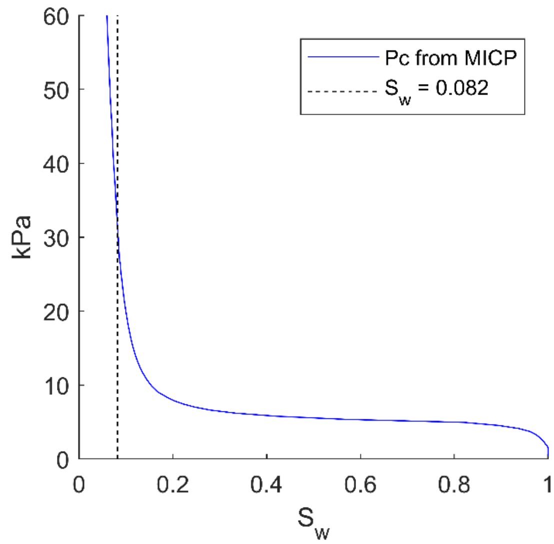

Figure 4. Comparison between the average S_{wc} (0.082) from the micro-core with the scaled mercury injection capillary pressure. We imposed a capillary pressure of 60 kPa in the centrifuge experiments.

Comparison of results with and without ageing

Figure 5 shows two-dimensional grey-scale images extracted from a 1000^3-voxel cube image after applying a nonlocal means edge preserving filter for both aged and unaged samples. The scans were taken after steady state was reached with a fractional flow of 0.5. It can be observed that, as expected, in the unaged, water-wet case, the large pores are mainly occupied by the non-wetting oil phase. The brine mainly remains in the small pores. In the aged sample, the oil phase is seen in both small and large pores. A negative capillary pressure ($P_c = P_{nw} - P_w$, where P_{nw} is the pressure in the non-wetting phase and P_w is the pressure in the wetting phase) is inferred from the curvature of the brine and oil interface, which indicates the wettability of the rock surface is oil-wet in many places. Parts of the rock surface still remains water-wet, suggesting mixed-wet conditions.

Figure 6 shows *in situ* contact angle measurements for the highlighted region in Figure 5B. Here we used the manual contact angle method [20]. We see values greater than 90°, indicative of a significant wettability alteration, meaning that the sample preparation procedure was effective. In contrast, for the unaged, water-wet sample, the contact angles are all below 90°.

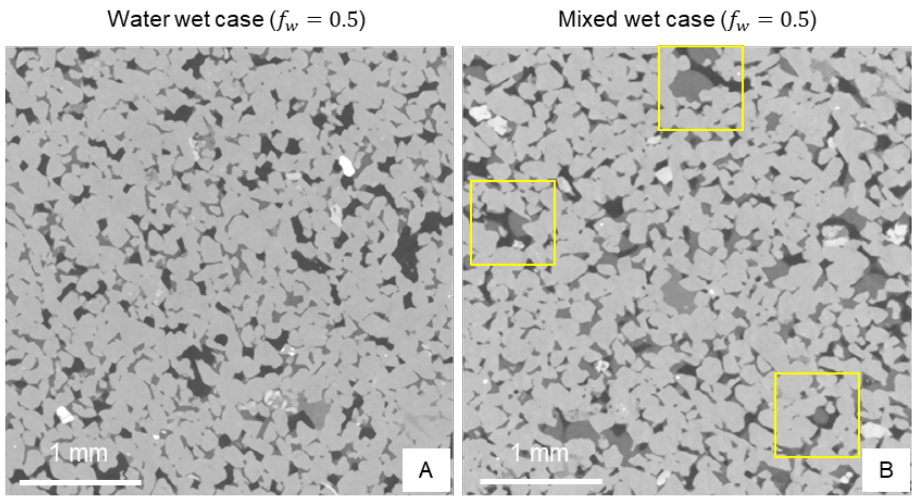

Figure 5. Grey-scale two-dimensional cross-sections of three-dimensional images after applying a nonlocal means edge preserving filter. The dimension of both images is 1000^3 voxel cubed and the voxel size is 3.58 μm. In both figures, the dark phase represents oil, the intermediate grey phase is brine and the bright phase represents rock grains. The images are taken after steady-state waterflooding as a fractional flow of 0.5. (A) Unaged, water-wet case. (B) Images of an aged sample, following the protocol in this paper. Some regions with contact angles larger than 90° with negative capillary pressure (from interfacial curvature) in the brine phase are highlighted.

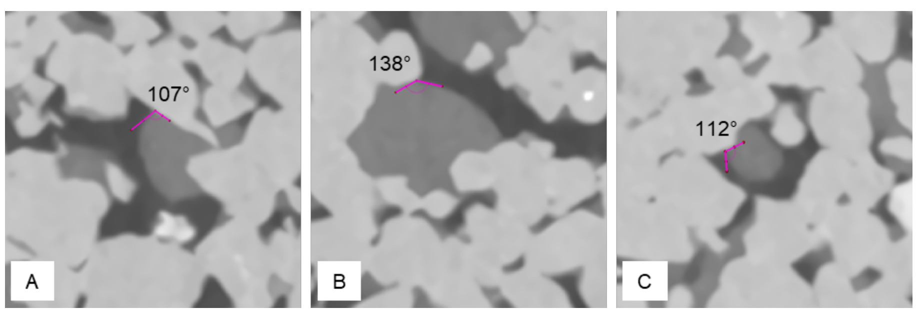

Figure 6. *In situ* contact angles for the highlighted region in Figure5B were measured showing that the wettability of the rock surface had been altered. In the figures, the dark phase represents oil, the intermediate grey phase represents brine and the bright phase represents rock grains.

CONCLUSIONS

A novel methodology for preparing micro-plugs to desired representative initial saturation has been developed. The methodology is based on following best SCAL practices for establishing S_{wc}, i.e. the porous plate method or centrifugation. The main concept is based on drilling several mm-scale micro plugs with approximately 2.4 cm length from a larger standard SCAL core plug without breaking capillary continuity.

The initialization protocol has been validated by confirming a uniform saturation distribution along the entire micro-plug length. Furthermore, the average water saturation is similar to that of the whole assembly of micro-plugs and the base, and on independent experiments on intact full-sized cores. Dynamic ageing leads to a higher initial saturation. We also confirmed that a substantial wettability alteration had occurred through measuring *in situ* contact angles and observing negative interfacial curvatures during waterflooding.

The main benefits of deploying the new initialization protocol for micro plugs can be summarized as follows:

a) It secures a uniform saturation distribution as a starting point for waterflooding, consistent with SCAL requirements.
b) It is possible to prepare a large number of micro-plugs simultaneously. Establishing S_{wc} by centrifugation takes approximately 2-3 days, while the porous plate method takes approximately 4-5 weeks. After this, core plugs need to be aged for 4 weeks.
c) Applying the methodology for homogenous rocks means that a sizeable number of micro-plugs have the same initial properties. This gives unique possibilities with respect to integration of different types of experiments in research.
d) Micro-CT flooding units are not occupied for preparation and ageing of the micro-plugs. This means that it is possible to conduct more flow experiments in research studies.

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