DOS AND DON'TS WHEN DEVELOPING A SYSTEM TO INVESTIGATE SPONTANEOUS IMBIBITION IN UNCONSOLICDATED POROUS MEDIA

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This paper was prepared for presentation at the International Symposium of the Society of Core Analysts held in Trondheim, Norway, 27-30 August 2018

ABSTRACT

This paper describes the development of a consistent model system to measure spontaneous imbibition and determine saturation functions in unconsolidated porous media. Sand grains or glass beads were packed in up to 0.5 m long, transparent glass tubes with optical access to local saturation development during spontaneous imbibition processes. The Two Ends Open-Free spontaneous imbibition (TEOFSI) boundary condition was used, where one end face is exposed to the wetting fluid and the other end to the non-wetting fluid. Dynamic measurement of the advancing displacement front and volumetric production from each open end-face enabled estimation of capillary pressure and relative permeability for the system.

A range of wetting- and non-wetting phase viscosities and viscosity ratios was used during spontaneous imbibition in unconsolidated sand or glass packs. Wetting phase (water) viscosity was increased using water soluble glycerol or polymers. Air or mineral oil of varying composition provided a wide range of non-wetting phase viscosities. High permeable systems are extremely sensitive to laboratory properties, which may dominate the viscous resistance and determine flow behaviour. Systematic discrepancies observed in early testing indicated that end effects were present, even in long systems, in the filters at each end of the glass tube to maintain the grains or beads in place. Different filters were tested (no filter, glass, paper and micro-porous discs) to determine the impact of the filter on spontaneous imbibition. In addition to slower oil recovery than anticipated, development of a non-uniform displacement front was observed, demonstrating the large influence from minute heterogeneities within the packs, and at the end faces. A standard sand grain packing procedure, using a custom-designed packing device, was therefore developed to ensure homogeneous properties throughout the porous media, and limited the spread in porosity and permeability values. Homogeneous sand packs with reproducible properties are necessary, to systematically investigate flow parameters and changes in wettability in unconsolidated porous media.

INTRODUCTION

The use of unconsolidated sand or glass bead packs in flooding studies is well documented. This paper sets out to further investigate spontaneous imbibition in such media. A controlled system to investigate spontaneous imbibition in unconsolidated porous media is necessary, to estimate oil recovery by this mechanism in different oil/brine systems, and to quantify changes in wettability due to chemical EOR (cEOR). We have developed a model system for this purpose and describe the process in this paper.

The study of spontaneous imbibition is of widespread interest, and several research groups are actively studying various aspects of spontaneous imbibition including; pressures acting during spontaneous imbibition (Li, Ruth, et al. [1]), capillary back pressure and relative permeability behind the imbibition front (Haugen, Fernø et al. [2]), imaging of front development (Fernø, Haugen et al. [3]), and entrapment of the non-wetting phase during spontaneous imbibition (Meng, Liu et al. [4]). Recovery of oil by spontaneous imbibition is driven by surface energy, through the action of capillary pressure (Morrow and Mason [5]). The majority of current understanding of spontaneous imbibition originates from experiments, where All Faces Open (AFO) and One End Open (OEO) boundary conditions have most often been used. However, as flow during these experiments occur both co- and counter-currently, it is difficult to model the flow with established differential equations. Firoozabadi [6] and Pooladi-Darvish and Firoozabadi [7] proposed that co-current imbibition may be the dominating process during oil recovery in fractured reservoirs, where the matrix blocks are partially contacted by both brine and oil. An alternative boundary condition, termed Two Ends Open- Free Spontaneous Imbibition (TEOFSI), was suggested by Dong, Dullien et al. [8]. Their setup features two open ends, where one is contacted by the wetting phase and the other by the non-wetting phase, and favours co-current flow due to zero capillary pressure at the end face contacted by the non-wetting phase. The setup was recently studied in detail by Haugen, Fernø et al. [2] and supporting theory was established. Meng, Liu et al. [4] used TEOFSI boundary conditions to investigate spontaneous imbibition into unconsolidated sand, using cylindrical glass tubes to visualize the imbibition process. Their setup featured a sandstone core piece between the unconsolidated sand pack and imbibing fluid, which controls the capillary back pressure (CBP) and, thus, counter-current oil production. CBP, often referred to as the bubble pressure, is the capillary pressure associated with formation of non-wetting phase droplets at an end face covered by wetting fluid, and is strongly connected to the amount of counter-current oil production in TEOFSI experiments.

Initially, this work aimed to create an experimental setup where co-current spontaneous imbibition in unconsolidated porous media could be studied in detail, especially focusing on the impact of viscosity ratio between the wetting and non-wetting phase on the imbibition process. This was achieved by packing the sand or glass beads into cylindrical glass tubes, of 2.04 cm diameter and lengths varying from 0.09 m to 0.5 m. The imbibition process was monitored by measuring the effluent production at both ends of

the sand pack, and at the same time directly visualizing front development and position through the glass tubes. The additional information gained from direct visualization provides new knowledge about the spontaneous imbibition process compared to conventional experiments. Direct observation of the dynamics of spontaneous imbibition, including local flow patterns, provides additional parameters to which numerical modelling and simulation can be matched, and indicates when the spontaneous imbibition process should be expected to scale according to established models, and when it should not. The setup also enables continuous assessment of sand pack stability, where sand redistribution and formation of flow conduits, as well as development of non-uniform saturation front could be observed directly through the glass tubes. Meng, Liu et al. [4] studied spontaneous imbibition in a similar setup. We have used glass tubes of a larger diameter and with varying lengths. Further, a recent numerical study by Andersen, Brattekås et al. [9] showed that the capillary back pressure of the filter significantly influenced flow, in particular, counter-current production of oil. Here, we aimed to improve the capillary contact between the unconsolidated porous medium and the imbibing fluid at the end face by using a variety of different filters. Several challenges were encountered, either related to properties of the pack itself (section A in this paper) or associated with the boundary conditions (section B of this paper) or experimental setup (see section C). To tackle the challenges, the experimental setup was continuously developed. Four different experimental setups are considered and described in this paper.

A- PREPARATION

This section describes challenges and solutions related to the mechanical integrity of the sand and glass bead packs.

Sand grains

Geological sieves were used to control the grain size of the sand. Two different grain size distributions were sifted out: a narrow distribution of 212-250 μm and a wider distribution of 125-250 μm. The sand was washed using tap water and dried at 80 °C for 5 days. The dry sand was thereafter fired at 500 °C for five hours to remove impurities, while maintaining the quartz structure.

Glass beads

Semi-spherical unwashed glass beads of 150-212 μm with well rounded edges were ordered from Sigma Aldrich. The glass beads were washed with hydrochloric acid before use, and rinsed using distilled water (the glass beads were poured into a glass funnel with a paper filter at the bottom, and distilled water was flushed through the funnel until the effluent had the same pH as the initial DI water, pH~7). The glass beads were dried in a heating cabinet at 60 ºC for 4 days.

The packing procedure

The sand grains or glass beads were packed into vertically positioned glass tubes. At the bottom end of the glass tube, a filter and/or specially designed end piece (see section C for further description) was fitted to support the grains/beads. Two different packing

procedures were used: shaking and compacting. The methods are briefly described below. After packing and assembly (see section C), the packs were flushed by CO_2 and saturated directly by the non-wetting fluid under vacuum. Pore volumes were calculated from weight measurements, and porosities were calculated by dividing the pore volume by the bulk volume of the pack (inner cross-sectional area of the glass tube multiplied by the length of the pack). Porosity dependency on packing procedure is demonstrated in **Figure 1**. The wetting and non-wetting fluids used are shown in **Table 1**. *Shaking*: 2 cm of sand/glass beads were poured into the glass tube using a funnel, and the glass tube was gently shaken. This was repeated until the porous medium reached 2 cm from the outlet. A thin layer of glass wool was then fixed on top of the sand (thickness 0.5-2 mm) and a rubber stopper was secured at the top of the column. A finite weight of sand/glass beads was used to pack a single glass tube: 238 g for sand (212-250 µm) and 230 g for glass beads. This procedure produced pack porosities within a range of 37-41%. When a wider grain size distribution was used, the porosity increased both in value and range, to 40-46%. The method described above was used. The amount of sand required to fill the tube varied more when 125-250 µm sand was used; ±4%, than for the narrow range sand (<1%). The poorer sorting of the sand is thus directly reflected in the porosity and weight measurements.

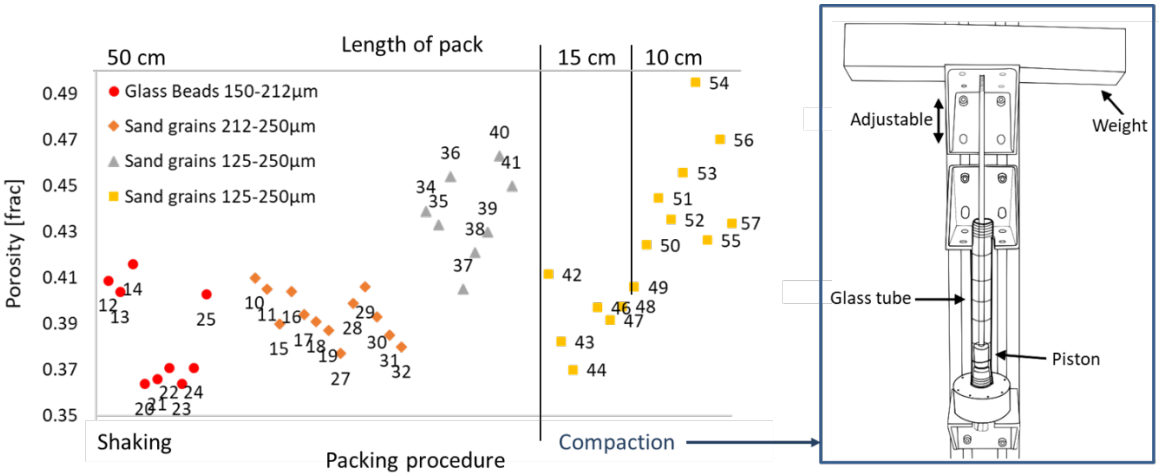

Figure 1: Porosity of the unconsolidated packs. The red dots represent glass beads, the orange dots represent sand grains with a narrow grain size distribution. The grey triangles (long sand packs) and yellow squares (shorter packs) represent sand with a wider grain size distribution, packed by different methods: shaking and compacting, respectively. Missing numbers are packs where air was used as the non-wetting phase.

The packs were initially fully saturated by the non-wetting fluid (air or mineral oils), and flooded, either before imbibition (by oil to measure absolute permeability) or after (by water, to measure incremental recovery and end point relative permeabilities). The packs that were made using the shaking procedure often did not maintain their structure during forced floods; redistribution of grains and formation of high-permeability streaks through the packs was observed in the glass tubes. An improved packing procedure was created, where the grains/beads were sufficiently compacted to maintain the pack structural integrity during forced floods. Irreducible water saturations could thus be established, and

the same pack could be used in several imbibition cycles. This procedure is called *compacting*, and the device used is shown in **Figure 1**. The packing device was constructed to add pressure to the packs during packing. A vertically adjustable hanger at the top was fitted with a vertical steel rod attached to a POM (polyoxymethylene) piston at the lower end. The piston diameter corresponded with the inner diameter of the glass tube. 1-1.5 cm of sand (so far only coarse sand, 125-250 μm has been tested) was poured into the glass tube. The piston was placed into the glass tube, on top of the sand, and a weight was added to the hanger to compact the sand into a pack (1 kg was used, and left on the hanger for 10 seconds). Between each new addition of sand, the piston was pulled out of the glass tube. When the glass tube was filled with sand, pressure was applied for 16-18 hours. The spread in porosity increased using the compaction method (38-49%), however, forced floods were performed without sand redistribution. The compaction method will be tested with more narrowly distributed sand grain sizes in following work. A combination of controlled shaking and controlled compaction should be considered in future work.

Table 1: Wetting and non-wetting fluids used. The wetting fluids were based on DI water. The non-wetting oils were filtered through silica sand and glass wool before use, to remove polar components. Air was used non-filtered.

Wetting/Non-wetting	5 wt% NaCl Brine	5000ppm HPAM	70 wt% Glycol	Air	Decane	Marcol 82	DTE FM 32	DTE FM 68
Viscosity [10^{-3}Pa*s]	1.15	28.00	28.00	0.017	0.96	33.00	73.00	197.00
Density [g/cm^3]	1.03	1.03	1.20	0.0013	0.73	0.84	0.85	0.86

B- BOUNDARY CONDITIONS

The high permeability and low capillarity of the sand packs make them susceptible to laboratory effects that are not usually influential of flow.

Spontaneous imbibition experiments were performed in our sand packs applying TEOFSI boundary conditions: Two Ends Open- Free Spontaneous Imbibition. This boundary condition is frequently described as having one end of the porous medium (termed the "inlet") in contact with the wetting phase (here: brine, glycerol or polymer solutions) and the other end (termed the "outlet") in constant contact with non-wetting fluid (here: air or oil). However, when unconsolidated media are used, filters are present at the inlet and outlet end faces to keep the grains or beads in place during spontaneous imbibition. A filter placed at the boundary between the wetting/non-wetting fluid and the porous medium introduces new boundary conditions that could influence the spontaneous imbibition process. This section describes the impact of boundary conditions associated with the filters.

The filters placed at the ends of the packs can be considered as capillary filters, with unique spontaneous imbibition behavior dictated by the filter properties. When applicable, the filter properties should correspond to the properties of the pack. In particular: the capillary back pressure (CBP) at the end face in contact with the wetting fluid, will largely control counter-current non-wetting fluid production. Particularly, non-

wetting fluid can only be produced at the inlet side when its pressure exceeds the CBP (Foley, et al. [10]). Filter CBP exceeding the CBP descriptive of the pack itself can therefore introduce a significant uncertainty in our interpretation of results; where the amount of counter-current production can incorrectly be attributed to other parameters. The filters tested for the inlet end are glass micro-porous discs or cellulose-based filters (paper or membrane), and for the outlet: glass wool or metal mesh. The filters were used with different end piece designs, further described in section C. *Glass micro-porous discs* with pore sizes of 16-40 µm or 40-100 µm were initially used. The discs were manufactured together with the glass imbibition tubes and embodied in the inlet end, as shown in **Figure 2**. Imbibition tubes and glass micro-porous discs were hand made by Mellum AS, Friedel Glassblåseri.

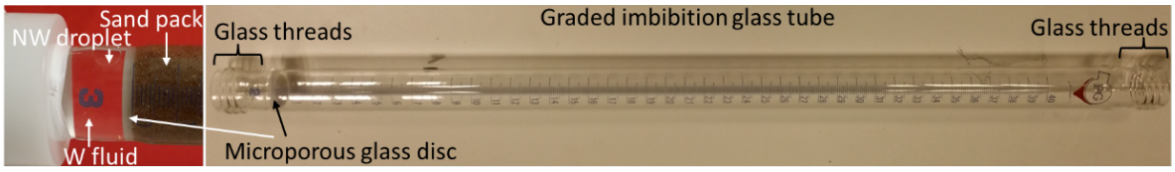

Figure 2: Graded imbibition glass tube with microporous glass disc imbedded in the inlet end. Left: close-up of inlet end. Right: 0.5m long imbibition glass tube.

The discs worked well during spontaneous imbibition of water/glycol solutions into packs saturated by air. Negligible counter-current production is expected in such experiments, due to the significant viscosity ratio. During water imbibition into oil saturated packs, however, two issues were raised, and the use of discs was disbanded: first, counter-currently produced oil accumulated near the inlet end (see **Figure 2** and section C). Second, having oil in the filters affected the imbibition ability of the filter in subsequent experiments, possibly due to an aging effect, that we were not able to reverse. The filters did not allow spontaneous imbibition to occur evenly across the end face after exposure to oil. Imbibition in limited regions at the surface significantly influence the front development during the onset of spontaneous imbibition, and the imbibition process can no longer be described by established differential equations (Føyen, Fernø et al. [11]). We therefore decided to use replaceable filters, where the initial filter conditions after packing could be assumed to be reproducible between different packs. Three different *cellulose-based filters* were then tested for use at the inlet end: paper A (20 µm), paper B (5 µm) and a membrane filter (5 µm). Paper A was soft (tissue-like) and provided good contact between the sand and the imbibing phase. Imbibition initiated upon contact between the filter and wetting fluid, and imbibition across the filter was even. Tearing in the filters could however occur during some of the spontaneous imbibition experiments, due to the filter softness. A torn filter could cause excelling imbibition rates or counter-current imbibition production, and the pack could not be used further. Paper B, an Ashless Quantitative Filter, Grad 589/2 manufactured by Whatman, was stiffer and more resilient than paper A. The contact between the sand grains and the imbibing fluid could therefore be slightly limited. Filter integrity must, however, be maintained in order to use the same pack through several cycles, and this filter is the top choice in current experiments. A third cellulose-based filter was also tested. Filter C, a Track-Etched

Cyclopore membrane filter manufactured by Whatman, was tested and shown to be oil-wet. When this filter was used at the end of porous packs, the imbibition process did not initiate itself, because the membrane filter constituted a barrier between the water-based fluid and strongly water-wet sand or glass bead pack. This is yet another example of how the boundary conditions in this setup can yield experimental observations that do not reflect the pack itself, but the filter properties. Spontaneous imbibition could be initiated by forcedly injecting the imbibing fluid through the filter. Imbibition progressed after wetting-phase continuity was established through the filter, and appeared to be controlled by the porous medium. Before choosing a filter for the sand pack, a qualitative filter wetting preference test is recommended. A simple test is performed by placing a small droplet of oil/water on top of the filter and observe its behavior and uptake. We placed a water droplet on top of the membrane filter and observed that it did not spread, but curved on the surface, i.e. the contact angle was $>90^{o}$. When the filter was slightly tilted, the water droplet rolled off the filter. Although qualitative, this indicates that spontaneous imbibition was highly unlikely in this filter when it was placed at the end of a porous pack.

In our experimental work, both the wetting phase and non-wetting phase viscosities have been varied. The water phase viscosity has been increased using polymers. Glycerol has also previously been used to increase the viscosity of the aqueous phase in spontaneous imbibition experiments (Kyte and Rapoport [12], Fischer and Morrow [13]). **Figure 3** shows polymer spontaneous imbibition into different sand packs (viscosity ratio $\mu_{nw}/\mu_w=0.03$). Polymer imbibition was first tested using Paper A and a long sand pack. A decreasing imbibition rate was observed, which levelled out at a low recovery factor. A hypothesis for this was that the filter was clogged by polymer. We removed the filter and observed a significant production of sand. During this time, spontaneous imbibition did not occur. When sand production stopped, polymer spontaneous imbibition recurred. Two new sand packs were subjected to polymer SI without a filter at the inlet. Outflow of sand was observed, during which oil was not produced. Polymer imbibition continued when the sand was stationary. Two short sand packs (10 cm) were also used, where Paper B (5 μm Cyclopore membrane filter) was used at the inlet, and the polymer was filtered through a corresponding filter before SI was initiated. The SI process occurred without sand production or clogging problems in these two packs. We can, however, not guarantee that the filters would not clog when using e.g. longer packs. This will be further investigated in future work.

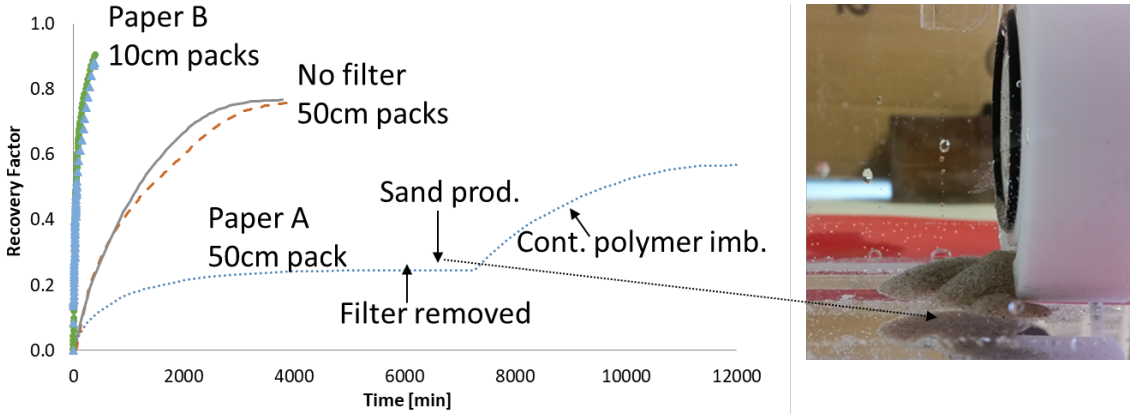

Figure 3: spontaneous imbibition in polymer solution. When the polymer was not filtered before use, the inlet filter clogged and prevented further spontaneous imbibition. When SI was performed without a filter at the inlet, sand was produced, which introduces a significant uncertainty in the experiments.

Limited, or non-existing, capillary inflow of the wetting fluid can be summarized as the main challenge when selecting inlet filters. Using TEOFSI boundary conditions, the outlet end only needs to facilitate outflow of non-wetting fluid. Initially, glass wool (silanized, ordered from Sigma Aldrich) was used at the outlet end, between the pack and the outlet end piece (in this case a rubber stopper). Recent numerical work has shown that compressible filters, such as glass wool, introduce an uncertain, and sometimes significant, resistance to flow (Andersen, Brattekås et al. [9]). If the outlet filter had sufficient resistance, this too could have an impact on the amount of oil produced counter-currently. Although the glass wool worked well as a screen, to maintain the pack in place, we would not recommend this solution, as it increases the uncertainty in the measured results. In current experiments, a fine metal mesh is facilitated between the pack and outlet end. Quantitative tests show that the metal mesh tend towards oil-favorable conditions, which is desirable for the outlet end of a water-wet pack. Standard stainless-steel metal mesh was used at the inlet and outlet (available from most hard ware stores). The recommended mesh aperture will depend on the experimental configuration. At the outlet, mesh apertures < sand grain size have been used to avoid sand leakage. We have used a mesh aperture of~ 100 µm. At the inlet, the mesh is mostly used to support the paper filter and can have a higher aperture. We have used a mesh aperture of ~1 mm at the inlet.

C- THE EXPERIMENTAL CONFIGURATION

This section describes the external influencers of SI in porous packs: the end pieces and experimental setups. We will here define an "end piece" as the piece of equipment present outside of the sand pack and filters. As briefly discussed in section B, the design of the end piece can cause wetting phase discontinuity at the inlet end due to accumulation of the non-wetting phase (**Figure 2**). The "experimental setup" refers to the inflow/outflow of wetting and non-wetting fluid, and measurement of non-wetting fluid production. The end pieces and experimental setups must be discussed in relevance to each other. In this work, four different experimental setup/end piece configurations were used (**Figure 4**) and are described below. *Complete immersion* of the imbibition tube is

advantageous and ensures equal hydrostatic pressures at the inlet/outlet, however, this configuration was challenging in our work. This configuration was first used during SI in glass tubes facilitating micro-porous glass discs, and a large distance between the sand pack/filter and the free wetting phase (see **Figure 2**). Counter-currently produced oil accumulated in the glass tube close to the inlet filter, which caused two main challenges: 1) counter-current oil production could not be measured versus time, and 2) the accumulated oil volume blocked wetting-phase inflow at the end face, leading to limited imbibition wetting phase regions. The inlet end piece was changed to facilitate paper filters, and the distance between the filter/pack and free volume where counter-current production could be measured was decreased. Artifacts were still observed during the experiments, that were associated with an additional resistance at the outlet. We believe that this was caused by the glass wool and the associated challenges further described in section B, as the packs were prepared using the shaking procedure, where glass wool and a rubber stopper secured the outlet. This was, however, not investigated at the time. Initiating the experiments could also be challenging, because SI could often start before the pack was correctly aligned in the bath. The configuration was changed to *separate inlet/outlet A* to overcome the challenges and improve the initiation and inlet configuration. The inlet was connected directly to a small water tank, with the water level just above the tube. This ensured a controlled initiation of the imbibition process (where the imbibing fluid could be poured gently into the tank with the sand pack already in the correct location). This setup also reduced the impact of the additional outlet resistance, which we now know was caused by glass wool. Counter-current production was, however, not easily measured, and the configuration was slightly altered to *separate inlet/outlet B*, which allowed direct measurement of counter-current production without delay. The tube was submerged into the wetting bath, and the outlet tubing was connected to air through a fitting implemented in the wall of the bath. The use of separate inlet/outlet configurations were abandoned after calculating front capillary pressure. The method described by Haugen, Fernø et al. [2] was used, revealing that the calculated front capillary pressure was in the same order of magnitude as the hydrostatic pressure at the inlet (caused by the water column). Thus, the inflow of wetting fluid is not only governed by the capillary pressure, but also significantly influenced by hydrostatic pressure. When investigating spontaneous imbibition in low-capillarity packs like these, such calculations should be performed for every setup before use, to make sure we are able to discriminate between the front capillary pressure and hydrostatic pressure. The forth configuration we have used removed the hydrostatic pressure influence and is called the *continuously flushing inlet*. This configuration is similar to the setup used by Meng et al. (2015) in some aspects, but was further modified to collect counter-currently produced non-wetting phase. This is a necessary addition to the experimental setup when the CBP in the inlet filter is reduced in respect to the pack CBP. The wetting fluid was circulated through a void space in the inlet by a pump, where the void space was also exposed to the inlet end face of the sand pack. Counter-currently produced non-wetting fluid was transported out of the inlet and accumulated in a measuring tube, before the wetting fluid was circulated back to the pump. It is possible to introduce a viscous pressure to the configuration when the continuously flushing inlet is used. Using a COMSOL model of our particular end

piece (incorporating the geometrical shape and dimension of the inlet design), this pressure was estimated to 0.065 Pa when a 1 cP, 1 g/cm^3 fluid was used, at volumetric circulation rates of up to 100 ml/h. Similar estimations should be performed for higher-viscosity fluids before viscous forces can be neglected in other experiments. Another benefit of this configuration is the ability to perform forced floods. By closing a valve in the inlet end piece, circulation ceases and the fluids are directed through the sand pack. A perfectly aligned setup is, however, required: because of the low capillarity of the high permeability packs, they are extremely sensitive to their surroundings and the impact from viscous forces and gravity.

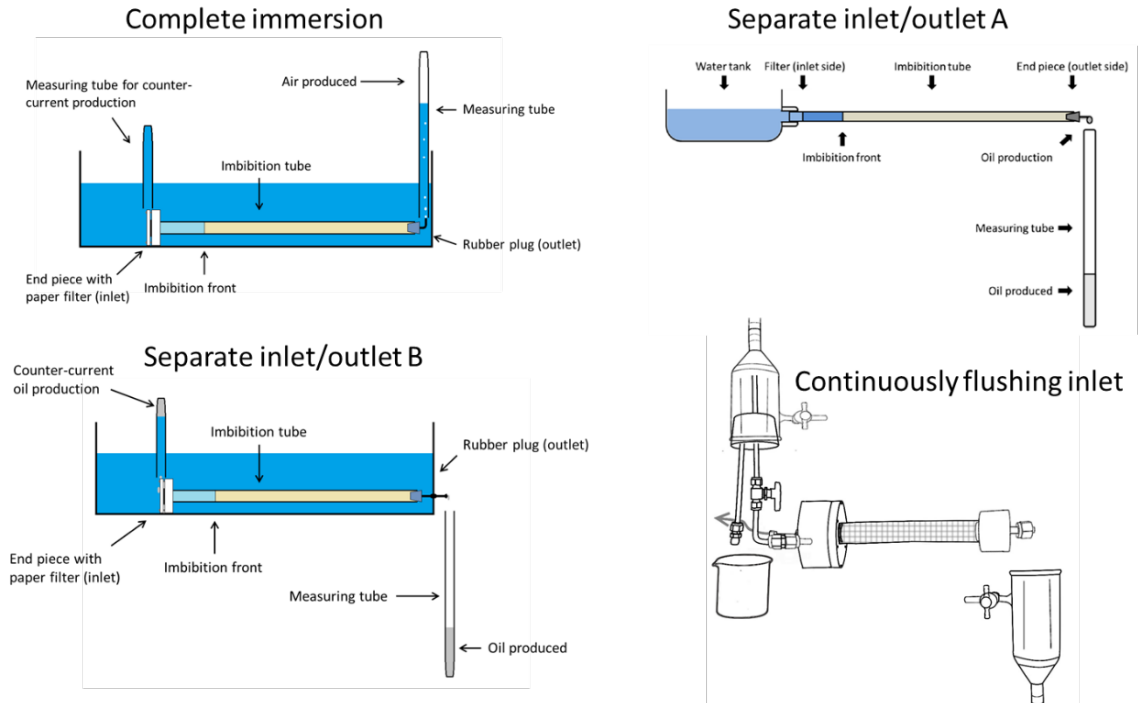

Figure 4: the different experimental setup/end piece configurations used in this work.

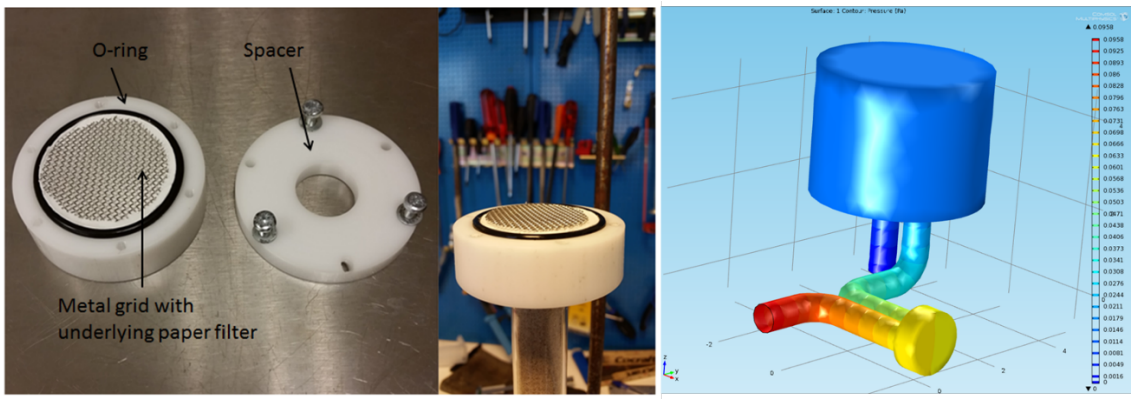

Figure 5: Left: Most frequently used end piece configuration. The packs were packed on top of paper filters with a coarse metal mesh under for mechanical support. Right: COMSOL model of inlet end piece. Warmer colors indicate a higher pressure. The values indicate the pressure with respect to the pressure at the end of the tubing

CONCLUSION

During our experimental work, we have found that high permeability, low capillarity packs of unconsolidated media are extremely sensitive to their surroundings. Spontaneous imbibition relies on capillary inflow of the wetting fluid, and the process is characteristic of the capillary driving force (the porous medium) and boundary conditions in addition to the fluids used. Using unconsolidated porous media, special care must be taken to avoid significant influence from laboratory properties. The main check points to consider when performing such experiments are summarized in **Table 2** below.

Table 2: Do's and don'ts when developing a system to investigate spontaneous imbibition in unconsolidated porous media.

DO'S	DON'TS
Consider the filter.	
The filter properties should be as close to the porous pack properties as possible, especially the CBP.	Do not use a compressible filter; this will introduce an uncertain resistance to flow, which can also influence counter-current prouction.
Use an inlet filter with a wettability corresponding to the fluid in contact with the filter (water-wet at inlet and oil-wet at outlet for a water-wet pack)	Do not proceed with a decision on which filter to use without qualitatively testing the filter wettability.
Use a filter that can maintain its structural integrity during several flooding cycles (if that is desired).	Do not use filters that are arbitrary. The filters should have the same parameters evenly distibuted over its surface, and it should be possible to use equal filters for several packs.
The filter should be thin and soft enough to enable good capillary contact between the sand and imbibing fluid.	
Perform spontaneous imbibiton tests with the wetting fluid evenly distributed across the inlet filter/non-wetting fluid evenly distiributed across the outlet filter.	Do not let the counter-currently produced fluids accumulate near the inlet filter.
Consider the fluids.	
Filter the imbibing fluid through a filter with a similar pore sixe as the inlet filter.	Don't expect negligible influence from fluids on e.g. filter wettability.
Consider the force	
Sand packs are extremely sensitive to their surroundings due to their low capillarity. Consider and calculate the viscous and hydrostatic forces included in your setup.	Don't perform an experiment without levelling the sand pack.
Consider the porous pack	
Make sure the packs are sufficiently compacted to maintain their structural integrity during forced floods (if desired) and through several cycles of spontaneous imbibition.	
Use a controlled packing procedure, where the pressure applied is controlled by the device, not by the person controlling it.	

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