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X-ray imaging of immiscible fluid fingering patterns in a natural high porosity rock

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2 ABSTRACT

3 This paper presents the development of a laboratory scale apparatus and first experimental
4 results on the characterization of fingering patterns of immiscible fluids in a porous rock
5 (Fontainebleau sandstone), using three dimensional full-field measurements from x-ray
6 tomography. The few existing studies that have extended experimental investigation of immiscible
7 fluid flow from 2D to 3D have been primarily interested in the pore scale or performed on idealized
8 porous media. While the heterogeneities inherent to natural rocks are known to play an important
9 role on subsurface fluid flow regimes, a limited number of studies have approached the problem
10 of characterizing the time resolved 3D multiphase flow in these material, at the mesoscale. The
11 series of experiments reported in this paper has been performed at a low viscosity ratio, water
12 invasion into oil as the defending fluid, and different capillary numbers (1.8 orders of magnitude).
13 The results illustrate the qualitative transition in the flow regime, from capillary fingering to viscous
14 fingering. While a full quantitative characterization the geometrical features of the fingers will
15 require further technical refinements, a qualitative understanding can be already gathered from
16 the results presented herein.

17 **Keywords:** immiscible fluids, x-ray tomography, viscous fingering, capillary fingering, flow instability

1 INTRODUCTION

18 The study of immiscible fluid flow in porous media is of great scientific interest to assess fluid migration
19 in geological repositories such as sedimentary rock reservoirs. The interaction between the intrinsically
20 heterogeneous porous structure of the host material and the interface between percolating fluids plays an
21 important role in the possible development of unstable flow patterns, giving rise to localized favourable
22 flow paths and entrapment clusters at the mesoscale. The natural occurrence of immiscible fluid interactions
23 in oil-water reservoirs, as well as artificial injections of non-native fluids (e.g., multiphase carbon dioxide
24 and water, hydrogen and water) for recovery and storage, has led to an increasing amount of research aimed
25 at characterizing the site conditions which favours or prevent the development of unstable fluid fronts and
26 residual entrapment.

27 At the scale of the porous network, the study of immiscible fluid flow instabilities in laboratory settings
28 has been facilitated by the development of experimental systems combining fluid percolation cells and
29 full-field measurement techniques. Such apparatus were initially extended, with the addition of a solid
30 porous phase, to Hele-Shaw cells (Hele-Shaw, 1898), which were previously used to study 2D multiphase
31 flow between two parallel plates (e.g. Paterson, 1981). In a seminal work in the field, Lenormand and
32 Zarcone (1985) proposed a new method to characterize multiphase flow inside idealized 2D porous
33 media, using series of optical images acquired during the invasion process. Shortly after, in an extensive
34 experimental and numerical study, Lenormand et al. (1988) proposed that the spatial distribution of
35 immiscible fluid pairs during the invasion process in a 2D porous medium is primarily controlled by
36 two dimensionless numbers: the viscosity ratio $M = \mu_i/\mu_d$, where μ_i and μ_d are the dynamic viscosity
37 of the invading and defending fluids, respectively, and the capillary number $Ca = \mu_i\nu_i/\sigma$, where ν_i is
38 the velocity of the invading fluid and σ is the interfacial tension. Using the combination of these two
39 dimensionless numbers during different invasion percolation experiments, the authors identified regions in
40 a log $M - Ca$ phase diagram characterizing three main limit flow regimes: (1) stable front displacement, (2)
41 capillary fingering, and (3) viscous fingering. This diagram, with the identified flow regimes and transition
42 regions, is represented in Figure 1. Using similar Hele-Shaw type experimental set-ups (in 2D), subsequent
43 experimental campaigns have also identified the mechanisms behind the unstable flow regimes in idealized
44 porous media (Khosravian et al., 2015; Zhao et al., 2016), comparisons with Saffman-Taylor theoretical
45 solutions (Toussaint et al., 2005), fractal dimension analysis (Lenormand and Zarcone, 1985; Måløy et al.,
46 1985), as well as pore-continuum upscaling relations (Løvøll et al., 2011).

47 The generalization of flow patterns from idealized 2D porous media to natural 3D porous rocks is
48 not trivial, as it depends on very different geometrical arrangement of the granular structure and flow
49 connectivity in the pore network. As such, advancements in x-ray computed tomography (CT) provide an
50 interesting avenue for the full-field characterization of multi-phase flow in such complex material. With the
51 ability to track the evolution of the fluid interfaces at resolved time increments and at the pore network
52 resolution, x-ray imaging is an essential advancement for studying flow patterns in 3D experimental setups.
53 Experimental studies using x-ray CT have mainly focused on the role of flow instabilities during the
54 invasion process in relatively small percolation domains for geomaterials (Prodanović et al., 2007; Spurin
55 et al., 2019), as well as glass bead packing (Culligan et al., 2006; Datta et al., 2013; Singh et al., 2017). The
56 high resolution at which full-field images were acquired in these studies have enabled to identify important
57 mechanisms in the fluid flow behavior at the pore scale, as well as the role of the granular media structures
58 in relation to the immiscible fluid interface. This highlighted the development of intermittent pathways, the
59 trapping of viscous phase and the role of fluid wetting in the invasion process. In all these studies, however,
60 the experimental setup, with relatively small percolation domains and regions of interests in the images,
61 does not enable the emergence of observable mesoscale fingers which could be compared to the results
62 previously obtained in experiments with 2D Hele-Shaw cells.

63 In a unique experimental campaign characterizing 3D mesoscale flow patterns, Hu et al. (2020) studied
64 the effects of different viscosity ratios and capillary numbers on the development of fingering structures
65 in a glass bead packing. In this study, the authors proposed, for the first time, an extension of the Ca-M
66 phase diagram in three dimensions and for a wide range of values, using different fluid pairs and injection
67 rates. While the boundaries of the phase diagram, identifying the three different flow regimes (stable front
68 propagation, capillary fingering and viscous fingering), were found to be in agreement with previously
69 reported 2D studies, the authors also noted a wider transition zone between the different regimes. It was also
70 reported that the fractal analysis for capillary fingering was in agreement with predictions from invasion
71 percolation theory (Dias and Wilkinson, 1986; Feder, 1988). The possibility to extend the findings obtained

72 for packings of spherical beads to natural geomaterials remains an open question. Recent numerical studies
73 where flow simulations were performed using the pore network geometry of natural rocks indicate that there
74 are noticeable differences in the transition of flow patterns and inhibition of fingering between the case of a
75 homogeneous distribution of porosity and the case of strongly heterogeneous porosity distribution, which
76 is typical of natural geomaterials (Liu et al., 2015; Tsuji et al., 2016). While other important experimental
77 campaigns were performed at larger scales on natural rocks (e.g., Riaz et al., 2007; Perrin and Benson,
78 2010; Berg et al., 2013), these previous studies were not focused on directly characterizing the development
79 and structure of individual fingers.

80 This paper presents a laboratory experimental system, which has been specifically designed to study
81 the evolution of immiscible fluid fingering in natural sandstone samples. Specifically, the purpose of
82 this work is to experimentally observe the development of fingering structures, by characterizing their
83 ramification and evolving geometry at different flow regimes. While a full quantitative characterization of
84 these geometrical features will require further technical refinements, a qualitative understanding can be
85 already gathered from the results presented herein. Therefore, the apparatus used for this study is made
86 compatible with x-ray CT imaging at the laboratory scale, over a flow domain which is around two orders
87 of magnitude larger than the average pore diameter of the tested rock specimens. The first observations of
88 fingering structures in high porosity Fontainebleau sandstone specimens, tested at three different capillary
89 numbers, are then presented as a preliminary and first outcome of this study.

90

2 MATERIALS AND METHODS

91 2.1 Materials

92 The natural porous rock specimens used in this study are extracted from a single block of Fontainebleau
93 sandstone from the Paris basin in France. This particular sandstone was selected for its high Quartz content
94 (98%), and therefore its inert chemical properties when interacting with percolating fluids. With a porosity
95 of 21%, the pore network consists of a heterogeneous distribution of cemented grains with a mean grain
96 size of $260\mu\text{m}$. Specimens of the raw sandstone material are prepared as 13 mm diameter by 26 mm height
97 cylindrical cores extracted at a single orientation and all from the same region of the initial block.

98 The two immiscible fluids selected for this study are a high viscosity oil and demineralized water doped
99 with a tracing agent. The oil, Total Azolla ZS 150, is the resident fluid initially saturating the porous
100 medium and therefore acts as the defending fluid during the invasion process. At 20°C it has a dynamic
101 viscosity of $330\text{ mPa}\cdot\text{s}$. To enhance the x-ray attenuation contrast of the invading fluid, a 20% mass ratio of
102 sodium iodide, NaI, is mixed into the demineralized water. At this concentration and a temperature of 20°C ,
103 the empirical relation for aqueous solutions developed by Laliberté (2007) suggests a dynamic viscosity
104 of $1.22\text{ mPa}\cdot\text{s}$. Using the pendant drop method for this fluid pair, the interfacial tension is measured
105 experimentally to be 40.7 mN/m (Stauffer, 1965).

106

107 2.2 Experimental apparatus and protocol

108 The experimental device is mainly based on a triaxial apparatus designed to optimize the quality of x-ray
109 CT acquisition under various experimental fluid flow and mechanical stress conditions. It serves in this
110 case as a core holder for the sandstone specimen, which is isolated from the confining chamber by a soft
111 membrane. The internal design of the triaxial cell, with an axial piston passing through it, enables to fully
112 decouple radial and axial stresses. While the current system has been used at relatively low confining
113 pressures (0.7 MPa), the apparatus is designed to sustain up to 30 MPa . It is therefore a versatile system

114 which can extend the scope of this campaign to higher pore pressures and coupled hydro-mechanical
115 experiments. A schematic view of the apparatus, with the fluid flow inlet connected to a high precision
116 syringe pump, is shown in Figure 2.

117 For the test preparation, the sandstone specimen is first gradually saturated inside a vacuum chamber
118 with oil heated at 50°C to reduce its viscosity and reach a homogeneous saturation level. After 24h inside
119 the vacuum chamber, the lateral side of the cylindrical specimen is then wrapped with a layer of Teflon tape
120 to enhance the smoothness of the contact with the sandstone surface. It is then installed inside an oversized
121 heat shrink membrane pre-installed on the bottom platen of the triaxial cell and filled with oil to avoid
122 air entrapment. The sodium iodide saturated water, filling the syringe pump and tubing connector, is then
123 injected until it reaches the inlet of the bottom porous stone. Afterwards, the top cap is installed and the
124 membrane shrunk to the required size, ensuring a tight fit around the specimen, the porous stones and the
125 stainless steel tubes. The triaxial cell is then closed and the confining chamber is gradually pressurized to
126 0.7MPa.

127 Each test starts with the simultaneous initiation of fluid injection, at a prescribed and constant flow rate,
128 and the acquisition of x-ray CT scans. The sodium iodide saturated water therefore initially percolates
129 through the bottom porous stone and reaches the specimen to invade the pore space in an average upward
130 movement. The injection and image acquisition proceeds continuously until a clear percolation path is
131 identified in the sequence of x-ray radiographs and the more attenuating invading fluid is seen to reach the
132 top porous stone. Since the injection rate varies greatly from one experiment to the other, by more than an
133 order of magnitude, a careful calibration of the image acquisition is required.

134

135 2.3 X-ray image acquisition

136 The experimental setup described above is installed inside the x-ray scanner at laboratoire 3SR in
137 Grenoble (Viggiani et al., 2014). The acquisition parameters for this series of experiments were selected
138 to optimize the acquisition time, at a resolution and signal-to-noise ratio allowing a clear segmentation
139 of the different fluid phases. The acquisition rate is three x-ray projections per second for a total of 1200
140 projections per reconstructed CT scan. At this speed, a tomography can be reconstructed over a 400 second
141 time period for a complete 360° rotation. It is therefore relevant to represent the field measurements as a
142 reconstructed volume when the time scale at which the fluid patterns develop are sufficiently slow compared
143 to the acquisition time. However, at fast injection rates, when the breakthrough occurs at or below the time
144 scale of a single CT scan, the filtered back-projection reconstruction method leads to partial volume effects
145 and streak artifacts. In the present study, due to current limitations of the laboratory x-ray scanner, the data
146 from fast injection experiments are represented as the difference between an instantaneous projection and a
147 reference state at the same angle. Although this representation does not enable a quantitative analysis of
148 the 3D finger geometry, the high contrast of the injected fluid is sufficient to qualitatively assess the shape
149 of the developing fluid flow patterns.

3 RESULTS

150 In a first set of experiments, a series of three invasion percolation processes have been performed with x-ray
151 imaging. For this series of experiments, with sodium iodide enriched water injected into the defending oil,
152 the log value of the viscosity ratio, $\log(M)$, is -2.43. Under these conditions, the three selected injection
153 rates of 0.3, 0.05 and 0.005 ml/min also result in log values of the capillary numbers, $\log(Ca)$, of -5.90,
154 -6.67 and -7.67 respectively. These values are represented in the phase diagram of Figure 1, where the
155 three experiments are located in the transition region from capillary fingering, at the slowest injection

156 rate, to viscous fingering, at the fastest injection rate. In this transition region, the change in capillary
157 number by 1.77 orders of magnitude results in significantly different fluid flow patterns, visible in x-ray
158 radiographs. Figure 3 shows this stark contrast in the x-ray radiographs for an intermediate stage during
159 finger development and at the breakthrough time, i.e., when the injected fluid reaches the specimen outlet.
160 In this representation, the attenuation of the injected fluid is highlighted by subtracting the image at the
161 initial state (attenuation contribution of the apparatus and granular structure).

162 For the higher capillary number, $\log(\text{Ca}) = -5.90$, the invasion process occurs rapidly, at a breakthrough
163 time of 15 seconds, with a low invasion volume of the pore space. In this case, a single finger-like structure
164 appears to develop as the main invasion pathway, at a high aspect ratio extending vertically through
165 the sample with limited secondary invasion pathways visible at the bottom of the sample. While radial
166 expansion of the main finger remains limited, it is visible around specific regions of the main pathway
167 between the pair of radiographs in Figure 3a, indicating secondary radial growth well below the finger tip.
168 However, this radial expansion does not seem to mature into new invasion fronts within the time period
169 before breakthrough. Conversely, for the experiment at $\log(\text{Ca}) = -6.67$, the invasion fluid is seen to occupy a
170 larger volume of the pore space, before the breakthrough, which occurs after 10 minutes. In the radiographs,
171 two large fingers connected to the inlet can be seen growing simultaneously in the specimen. Although it is
172 not possible to assess their three dimensional aspects in this representation, a substantially larger radial
173 extension of the fingers along their length is visible, compared to the experiment at the higher capillary
174 number. At the tip of the invasion front, smaller structures of limited vertical length are also observed,
175 suggesting that the advancing front is not a homogeneous compact plume but is rather populated by smaller
176 scale substructures. In the experiment at $\log(\text{Ca}) = -7.67$, the invasion process occurs at a much slower rate,
177 as reflected by a breakthrough time of 1.2 hours. The invasion front is seen to propagate as a self-similar
178 structure with both a vertical compact front and a radial expansion around a single finger.

179 Since the invasion process is much slower during the experiment performed at the lowest capillary
180 number, a series of CT scans could be reconstructed during the development of the fingering pattern. Figure
181 4 shows a time series, as a function of the breakthrough time t^* , during the injection of the invading fluid
182 forming a compact structure. The volume increments between each time step are highlighted in the Figure.
183 Since the increments shown are also spaced in time, it can be seen that the main finger initially grows
184 rapidly from the sample inlet. As the interface between the resident oil and injected water increases and,
185 therefore, the radial flow becomes greater, the vertical growth slows down over time. It is also interesting
186 to note the presence of a constriction near the specimen inlet before an expansion into a larger plume,
187 continuously widening as the front progresses vertically through the sample. A secondary finger can be
188 seen to emerge after the breakthrough occurs.

4 DISCUSSION AND CONCLUSIONS

189 For the series of three experiments presented in this preliminary study, the flow pattern is seen to be
190 influenced by the capillary number, resulting in a transition in the development of fingering structures.

191 For the high $\log(\text{Ca})$ experiment, the invasion process is dominated by viscous fingering, creating
192 thin and rapidly growing pathway of the invading fluid in the direction parallel to the imposed vertical
193 pressure gradient. The observed features of the finger-like structures, with branches developing at a width
194 approaching the pore size length scale of the studied sandstone, are similar to reported fingers in analog
195 2D porous media (Lenormand et al., 1988). This particular flow pattern results from the heterogeneities
196 naturally present in randomly organized granular arrangements, determining the length scale of the invasion
197 process (Feder, 1988). This random distribution of pore size and connectivity is also known to promote

198 a fractal growth of the fingers, compared to an organized distribution over the flow domain (Chen and
199 Wilkinson, 1985). Conversely, the propagation of viscous fingers in natural settings can be altered by sample
200 scale heterogeneities, such as layering and preferential pathways, promoting channeling of the invading
201 fluid (Brock and Orr, 1991; De Wit and Homsy, 1997). In the present study, the relatively homogeneous
202 pore size distribution and the absence of sample scale structural features in the host Fontainebleau sandstone
203 cores suggest that the mechanisms driving finger growth is primarily due to viscous forces and pore scale
204 heterogeneities, promoting possible fractal substructures in secondary branches evidenced by a moderate
205 lateral expansion of the main finger.

206 For this experiment at the highest capillary number, no evidence of flow intermittency is observed. This
207 contrasts with other studies performed in 3D porous media where multiphase flow is primarily driven by
208 viscous forces (Rücker et al., 2015; Spurin et al., 2019; Hu et al., 2020). The difference might be due to
209 the influence of boundary interactions, between the growing fingers and the membrane, thus limiting the
210 number of connected pores around the main pathway. The geometrical aspects of the pores and pore throats
211 can also influence the probability of snap-off events, where flow intermittency tend to occur between
212 intermediate to small pores (Spurin et al., 2019). It is possible that the pore structure of the rock used in
213 this case does not lead to the development of such event. However, a refined analysis at the smaller pore
214 scale would be necessary to investigate further its possible occurrence.

215 The experiment at intermediate $\log(Ca)$ captures effectively a transition towards capillary fingering. In
216 this case, the capillary and viscous forces become comparable and a competition between the two forces
217 driving the invasion process is seen to influence the aspect of the flow pattern. The increase in pore space
218 saturation shows a stabilization of the invasion front with a transition towards capillary fingering. This
219 stabilization of the front occurs as a result of capillary effects, which slow down the growth of viscous
220 instabilities over short length scales (Daripa and Paşa, 2008; Berg and Ott, 2012). For the low capillary
221 number experiment, observable features of the invading fluid pattern, such as wider and more compact
222 invasion front with increased radial growth, are associated with the capillary fingering dominated regime.
223 The clearly distinguishable contrast in morphology between experiments can be related to the fraction of
224 the resident fluid that has been displaced from the initial pore space volume.

225 The experimental setup presented in this paper demonstrates the existence of similarities in fluid flow
226 patterns between the investigated natural heterogeneous rock and previously studied 2D porous media.
227 Therefore, the results of this study provides a first insight into the immiscible fluid flow characteristics
228 at the mesoscale in such material and indicates a range of values expected for a transition to occur in the
229 invasion process. It is evident, however, that the bottleneck to further investigate the invasion process in
230 the viscous fingering regime lies in the speed of image acquisition. To extend the analysis to quantitative
231 measurements, a similar experimental method should be used in conjunction with faster x-ray tomography,
232 such as image beamlines at synchrotron facilities. Alternatively, recently developed methods in 3D volumes
233 reconstructions with a limited number of projections could be adapted to enhance the acquisition time
234 resolution.

CONFLICT OF INTEREST STATEMENT

235 The authors declare that the research was conducted in the absence of any commercial or financial
236 relationships that could be construed as a potential conflict of interest.

AUTHOR CONTRIBUTIONS

237 All authors listed have made a substantial, direct and intellectual contribution to the work, and approved it
238 for publication.

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FIGURE CAPTIONS

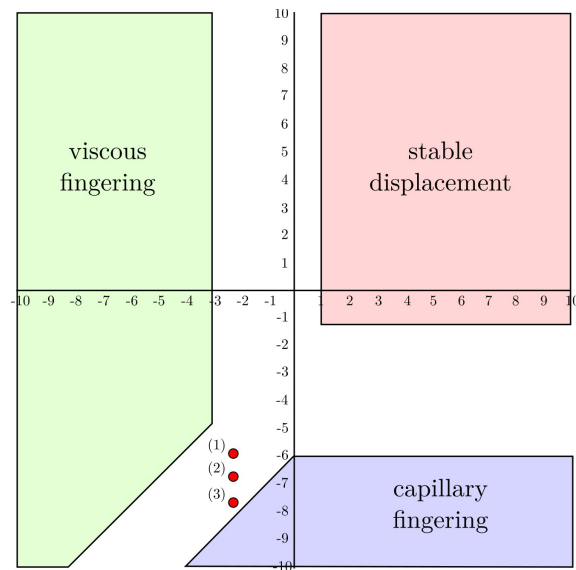


Figure 1. Phase diagram on a $\log(M)$ - $\log(Ca)$ scale and limits of the three fluid flow regimes. The three experiments at a $\log(Ca)$ of -5.90 (1), -6.67 (2) and -7.67 (3) are identified.

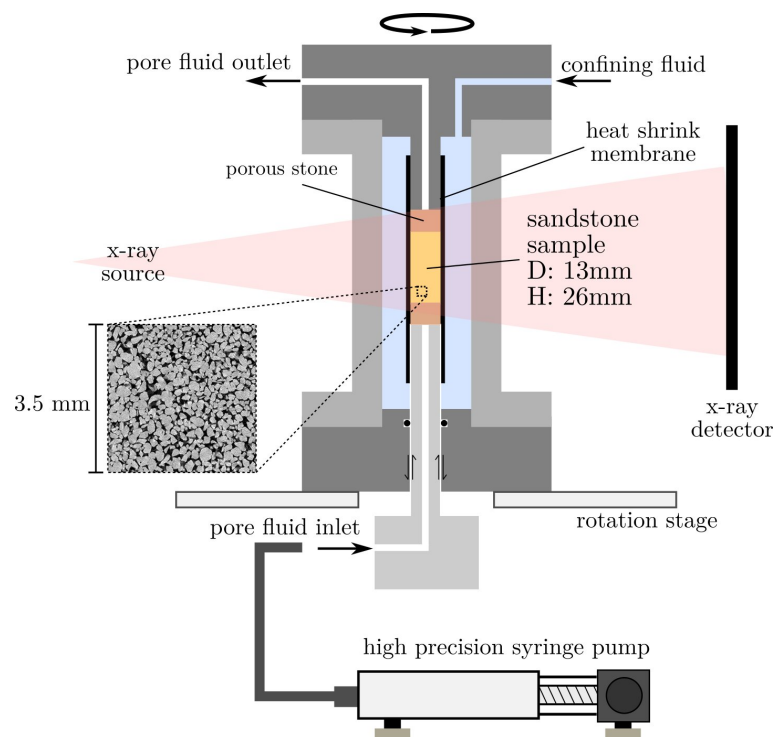


Figure 2. Schematic view of the different components of the fluid injection triaxial system and syringe pump. This configuration enables radiography and tomography imaging of the whole sample. A representative view of the pore structure is shown in the magnified inset.

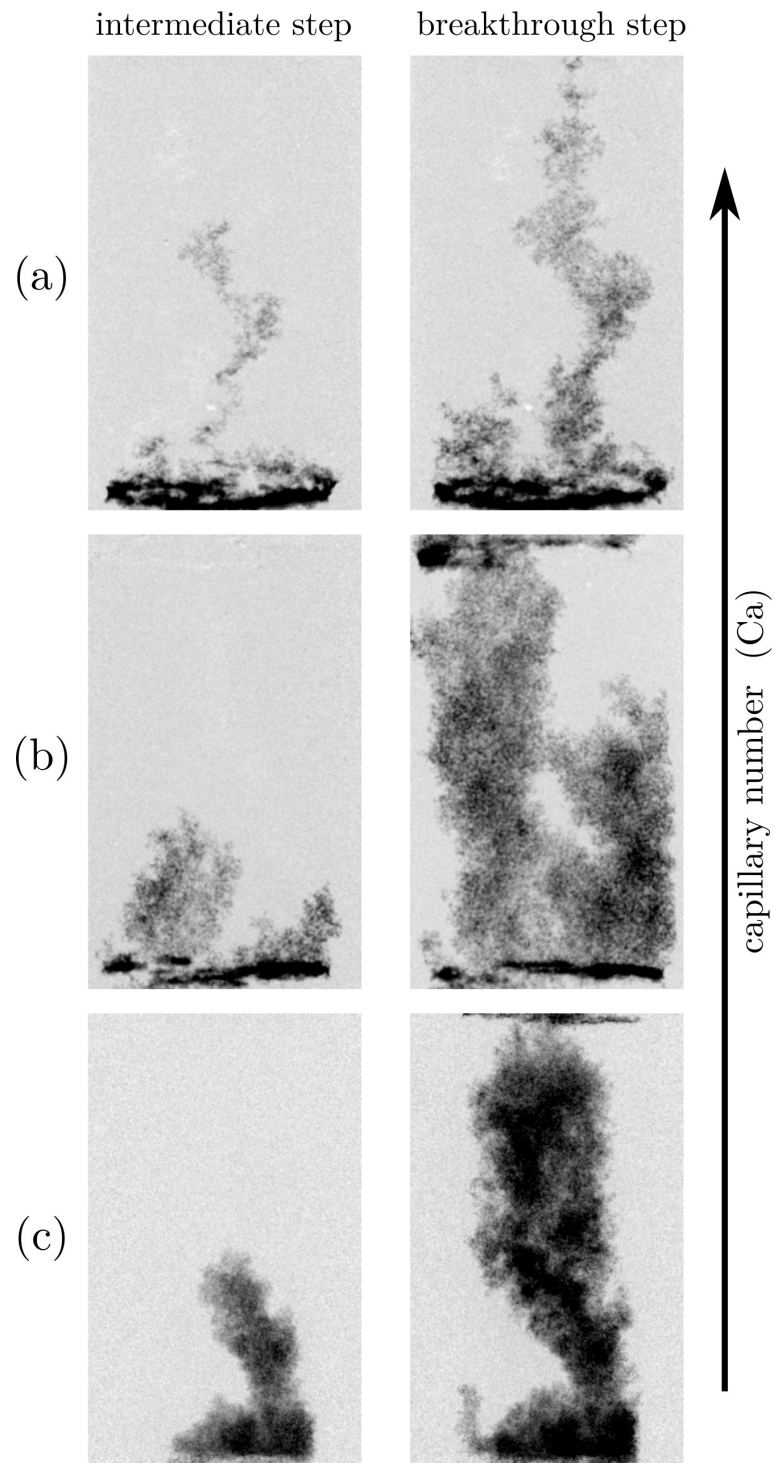


Figure 3. Radiography representation of finger patterns at an intermediate and breakthrough step during injection of the invading (water) and defending (oil) fluids. Experiments at three capillary numbers: $\log(\text{Ca})=-5.90$ (a) $\log(\text{Ca})=-6.67$ (b) and $\log(\text{Ca})=-7.67$ (c).

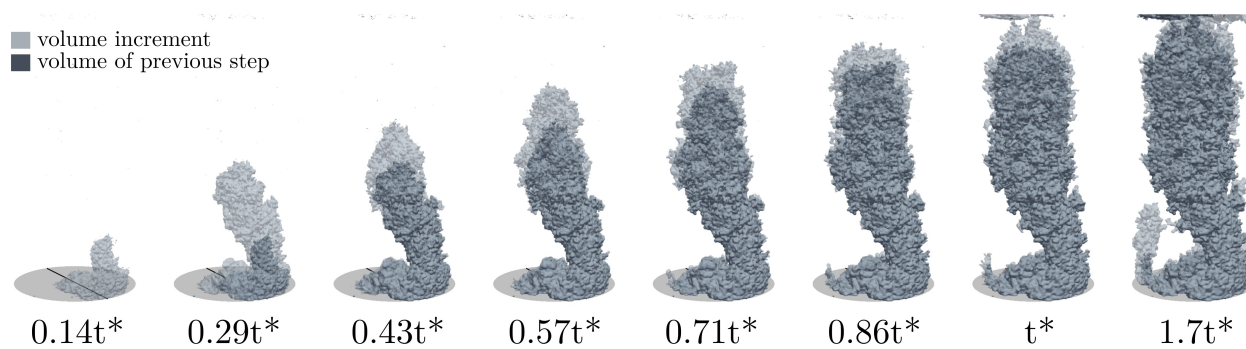


Figure 4. X-ray tomography reconstruction of the finger pattern for a low capillary number ($Ca=-7.67$, same as Figure 3c). The slow process enables to capture the development of capillary fingering in 3D.