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1 **Analysis of the structural changes of a pellet/powder bentonite**
2 **mixture upon wetting by X-ray computed microtomography**

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31 **Abstract:**

32 Pellet/powder bentonite mixture is one of the candidate materials for sealing plugs in deep
33 geological high-level radioactive waste disposal. This note presents an investigation on the
34 structure changes of this mixture occurring during the saturation process by means of X-ray
35 computed micro-tomography. The test was performed in an infiltration column (60 mm in
36 inner diameter and 120 mm in height). Water was supplied to the two ends of the column and
37 the changes of the sample morphology were observed during a period of 100 days of
38 hydration. Digital Volume Correlation (DVC) technique was used to determine the vertical
39 displacement field of the bentonite powder. A pressure transducer was used to measure the
40 axial swelling pressure during the hydration. The results show that the initial distribution of
41 powder in the inter-pellet pores was not homogeneous; the powder filled almost completely
42 the pores in the zones close to the two ends while air-filled inter-pellet voids were observed in
43 the middle of the column specimen. When water started to infiltrate inside the specimen from
44 both ends, the pellets and the powder grains started to swell (because of the swelling
45 properties of smectite, the principal mineral of bentonite) and filled the voids. That induced at
46 the same time increase of swelling pressure and downward movement of powder grains. The
47 results allowed a better understanding on the hydro-mechanical couplings, at the pellet scale,
48 in the pellet/powder bentonite mixture upon wetting.

49 *Keywords:* pellet/powder bentonite mixture; X-ray computed microtomography; initial
50 heterogeneity; Digital Volume Correlation; water saturation; homogenization.

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52

53 **1. Introduction**

54 In the context of deep geological high-level radioactive waste disposal, non-compacted
55 pellet/powder bentonite mixtures are considered as candidate sealing materials because of
56 their high swelling capacity and high radionuclide migration retardation properties, as well as
57 their operational advantages: they are easy to transport, install, and allow gaps between the
58 rock and the seal to be minimized. In a real repository this unsaturated mixture will be
59 installed to plug galleries. Then, water coming from the host rock will start saturating the
60 mixture under almost constant-volume conditions. The swollen mixture will fill different
61 voids and generate swelling pressure, ensuring the sealing of the storage system. Molinero
62 Guerra et al. (2017) used X-ray computed tomography (X-ray CT) to investigate the
63 microstructure of pellet/powder bentonite and found that this mixture was characterized by an
64 initial heterogeneous distribution of powder grains inside the inter-pellet pores. This initial
65 state could greatly influence the saturation process and the corresponding structure changes.
66 As a result, the overall hydro-mechanical behavior would change during the saturation
67 process. Therefore, to properly assess the safety of the underground radioactive waste
68 disposal, it is of paramount importance to well understand the microstructure change of the
69 mixture in the course of hydration.

70 In this context, the Institute of Radioprotection and Nuclear Safety (IRSN, France), as a part
71 of the overall research and development program that aims at providing scientific background
72 for disposal safety, launched the SEALEX (SEALing performance EXperiments) project,
73 within which this work was conducted. It consists of a series of full-scale experiments carried
74 out in IRSN's Underground Research Laboratory (URL – Tournemire, France) (Barnichon &
75 Deleruyelle, 2009; Mokni & Barnichon, 2016) and small-scale experiments conducted in the
76 laboratory (Wang et al., 2012; Saba et al., 2014). One of the aims of the full-scale experiments
77 was to test the long-term hydraulic performance of sealing systems in normal conditions for

78 different clay core compositions (pure bentonite or bentonite/sand mixtures) and
79 conditionings (pre-compacted blocks, mixture of pellets/powder, or in-situ compacted). In the
80 present work, a pellet/powder bentonite mixture with a proportion of 80/20 in dry mass was
81 investigated.

82 The hydro-mechanical behavior of engineered barriers composed of compacted
83 bentonite/sand mixtures or compacted pure bentonite has been extensively investigated (e.g.
84 Pusch 1982; Graham et al., 1989; Komine & Ogata, 1994; Dixon et al., 1996; Alonso et al.,
85 2005; Wang et al., 2013b, 2014; Saba et al., 2014; Sun et al., 2014). However, few studies
86 have been carried out on the mixture of pellet and powder of bentonite. Imbert & Villar
87 (2006) performed infiltration tests on a pellet/powder bentonite mixture and found that the
88 swelling pressure was analogous to that of a specimen of compacted powder at the same dry
89 density. Garcia-Siñeriz et al. (2015) studied the homogenization process of a material made of
90 bentonite pellets through a large-scale experiment on engineered barrier (EB). Upon
91 dismantling after 12 years of operation, a gradient of density and water content was still found
92 within the mixture because of the initial heterogeneity of the material. Van Geet et al. (2005)
93 investigated the hydration process of a pellet/powder bentonite mixture by X-ray CT. A
94 progressive decrease of the pellets density and an apparent homogenous sample after
95 saturation were observed. However, in the work of Van Geet et al. (2005), a cylindrical cell
96 containing only few pellets was used. As a result, the analyses mainly focused on the scale of
97 a single pellet.

98 The present work was thus conducted to investigate the structural changes of a pellet/powder
99 bentonite mixture with a large number of pellets by X-ray CT. Firstly, the sealing capacity of
100 the mixture and its structural evolution during saturation were studied. Secondly, Digital
101 Volume Correlation (DVC) technique was applied to calculate the displacement field within
102 the sample. Finally, the evolution of the axial swelling pressure while wetting was

103 investigated. The obtained results provide helpful elements to assess the hydromechanical
104 performance of seals/plugs made up of this kind of bentonite mixtures.

105 **2. Material and methodology**

106 **2.1. Pellet/powder MX80 bentonite mixture**

107 The soil studied was a mixture of pellet/powder MX80 bentonite (from Wyoming, USA) at a
108 proportion of 80/20 in dry mass. It was provided by the Laviosa-MPC Company under the
109 commercial name Expangel SP7 for pellets and SP30 for the powder, both were produced
110 from the same bentonite. The bentonite had a smectite content of 80%, other minerals being
111 quartz, calcite and pyrite. The CEC was 98 meq/100 g and the major exchangeable cations
112 were: sodium (52 cmol(+)/kg), calcium (37 cmol(+)/kg), magnesium (10 cmol(+)/kg). The
113 liquid limit was 560%, the plastic limit was 62% and the unit mass of the solid particles was
114 2.77 Mg/m^3 (Saba et al. 2014).

115 Cylindro-spherical pellets of bentonite were produced by compacting commercial bentonite
116 powder in a mold of 7 mm in diameter and 7 mm in height. The initial suction ($s = 135 \pm 3$
117 MPa) was measured in the laboratory with a chilled mirror dew point tensiometer (Decagon
118 WP4C), corresponding to an initial water content $w = 5\% - 7\%$. The initial void ratio of the
119 pellets was $e = 0.306 - 0.386$ (corresponding to a dry density of $2.00 - 2.12 \text{ Mg/m}^3$) and the
120 degree of saturation was $S_r = 55\% - 66\%$. The bentonite powder was produced by crushing
121 pellets. An initial water content of 3% (smaller than that of the pellets) was found in the
122 laboratory, corresponding to an initial suction $s = 191 \text{ MPa}$ (equally measured by a chilled
123 mirror dew point tensiometer).

124 The saturation fluid was obtained by mixing the chemical components presented in Table 1
125 with distilled water until full dissolution. The resulting synthetic water had the same chemical
126 composition as the pore water of the Callovo-Oxfordian claystone (a possible geological host

127 rock for radioactive waste disposal in France) from the underground research laboratory in
128 Bure (Wang et al., 2013a). The table shows a total mass of 5.677 g of salt per liter of solution,
129 that corresponded to a salinity (mass of salt per mass of solution) lower than 1%.

130 **2.2. Experimental set-up**

131 A special set-up was designed to carry out X-ray CT observations on the pellet/powder
132 mixture while wetting. The layout is presented in Figure 1a. It consists of a transparent
133 PMMA (PolyMethyl MethAcrylate) cell of 60 mm in inner diameter and 120 mm in height
134 (corresponding to 1/10 scale of the SEALEX full-scale experiment). In the full-scale
135 experiment, a horizontal borehole, with a 0.60-m diameter and 5.40-m length was excavated
136 in the Tournemire URL, located in a Mesozoic sedimentary basin on the western edge of the
137 French Causses. The clay-based core, which represented the engineered clay barrier, had a
138 total length of 1.20 m. The laboratory specimen was installed between two porous stones and
139 filter papers, placed at its top and bottom. The constant-volume condition for the
140 pellet/powder bentonite mixture was ensured by the rigid PMMA cell and a top piston which
141 could prevent axial swelling. The mixture was saturated from both sides (top and bottom),
142 simulating the SEALEX in situ experiments (in the full-scale experiment, the clay-based core
143 was saturated with water from both ends with a small pressure of 10 kPa). The axial swelling
144 pressure was monitored during the test with a force transducer. The base and top were both
145 equipped with a water inlet and an air outlet. Figure 1b shows the PMMA cell positioned
146 inside the X-ray microtomograph. The cell was connected to two reservoirs (located at the top
147 and bottom of the sample) allowing water infiltration. The visualized parameter from X-ray
148 CT observations was the linear attenuation coefficient, which was represented as a grey level.
149 This parameter depended on density, the atomic number and the used X-ray energy.

150 **2.3. Sample preparation and test protocol**

151 The pellet/powder mixture was prepared by following the first protocol proposed in Molinero
152 Guerra et al. (2017) to obtain a homogeneous pellet/powder distribution within the sample at
153 the target dry density (1.49 Mg/m^3). It consists in filling the cell by packets corresponding to
154 one layer of pellets spread over its base and by adding the corresponding amount of powder
155 (considering the proportion 80-pellet/20-powder in dry mass). The density of the mixture was
156 checked with the mass of the bentonite (with known water content) used to fill the cell (with
157 known volume). Moreover, some reference elements of 1500 PMMA spheres of 1.6 mm
158 diameter (density $1.18\text{-}1.19 \text{ Mg/m}^3$) were randomly distributed within the sample during the
159 preparation process. The purpose was to carry out quantitative analyses of bentonite powder
160 displacement by DVC technique. DVC is a full-field measurement technique that basically
161 consists in comparing two pictures at two different stages and calculating a displacement
162 field. A detailed description of this technique can be found in Sutton et al. (2009) and Bornert
163 et al. (2012).

164 The general experiment scheme consisted in scanning the pellet/powder mixture at different
165 times during the saturation process. This resulted in a kind of incremental scans occurring
166 during the hydration process: several X-ray CT observations were carried out at different
167 times until an apparent homogeneous mixture was observed. Each scan took 19 h. Prior to
168 each X-ray CT scan, the whole system shown in Figure 1a was moved and installed inside the
169 microtomograph.

170 The first step of the test consisted in opening the water inlet valves. Air inside the system was
171 evacuated by opening the air outlet valve until no air bubble could be observed anymore in
172 the outlets. Water was supplied through both the top and bottom of the sample, as in the
173 SEALEX full-scale experiments, during the total time of the test with no pressure. Axial
174 swelling pressure was recorded automatically by a force transducer and a numerical data
175 logger.

176 **3. Results**

177 **3.1. Qualitative analyses**

178 The behavior of the pellet/powder mixture was firstly investigated by carrying out a
179 qualitative analysis of the X-ray CT observations. Figure 2 shows a vertical section obtained
180 at different times.

181 Initially, the mixture had a heterogeneous distribution of pellets and powder in spite of a
182 careful preparation procedure. Air-filled inter-pellet voids were dominant in the zone between
183 the bottom and 90-mm height. Above this zone, bentonite powder was more dominant in the
184 inter-pellet voids. The top of the mixture was characterized by the presence of larger air-filled
185 voids between the mixture and the porous stone, most probably due to segregation in the
186 fabrication process. In-depth characterization of a mixture composed of pellet/powder
187 bentonite can be found in Molinero Guerra et al. (2017).

188 After 2 days of hydration, the voids on the top of the specimen were completely sealed due to
189 their vicinity to the hydration front, so the material at this level swelled quickly. The swelling
190 properties of the material corresponded to the swelling properties of smectite, which was the
191 principal mineral of bentonite. It can be deduced from sections corresponding to 2 days and 4
192 days after hydration that the evolution of the top hydration front was quicker than the bottom
193 one. Afterwards the evolution of both hydration fronts was similar. The initial structure lost
194 its granular nature while wetting; air-filled inter-pellet voids were still visible after 56 days of
195 hydration, even though the pellets located at the furthest position from both hydration fronts
196 had already swollen at this time. An apparently global homogeneous sample was observed
197 after 100 days of hydration. At this time, almost all the air-filled inter-pellet voids had been
198 completely sealed. In this figure, a glass sphere with a diameter of 10 mm can be seen; this

199 element was introduced to the specimen during its preparation as a reference material for X-
200 ray CT analysis.

201 To better understand the transition between the initial state and the final one, an enlargement
202 of a horizontal section at 60 mm from the bottom hydration front is presented in Figure 3.
203 Initially, high-density pellets could be observed with several powder grains between them.
204 After 11 days of hydration, some cracks developed within the pellets due to the decrease of
205 suction. As was observed by Molinero Guerra et al. (2017), cracks were already part of the
206 initial state of the pellet; they appeared during the storage in the laboratory combined to the
207 fabrication process of a single pellet. Those cracks could not be observed in this work, due to
208 the insufficient resolution of the X-ray CT observations (50 $\mu\text{m}/\text{voxel}$) as compared to the
209 resolution that was used to study a single pellet (4.4 $\mu\text{m}/\text{voxel}$); however, it is most probable
210 that they were already present at the initial state and opened while wetting. Wetting induced
211 swelling of smectite particles, enhanced these cracks and made them visible after 11 days of
212 hydration. After 27 days, more cracks were observed, as well as the decrease of the inter-
213 pellet voids due to swelling. Then, pellets started losing their granular structure. After 56
214 days, pellets could still be identified even if they had almost lost their initial granular
215 structure; some air-filled inter-pellet voids were observed. Therefore, the evolution of the
216 structure of a single pellet was characterized by the development of cracks combined to the
217 swelling of its components.

218 Van Geet et al. (2005) investigated the homogenization process of a pellet/powder mixture by
219 performing an analysis of the X-ray CT results. They noticed the decrease of the pellet density
220 while wetting and the final homogenization at complete saturation. In the present work, a
221 vertical section was investigated in order to ensure the total homogenization at the studied
222 resolution along the specimen's height, as well as the sealing of all air-filled voids within the
223 mixture.

224 **3.2. Quantitative analyses**

225 **3.2.1. Evolution of the axial swelling pressure**

226 The evolution of the axial swelling pressure during wetting was monitored during the test
227 thanks to a force transducer installed at the bottom of the sample (see Figure 1). Results are
228 presented in Figure 4 for the 160 days of hydration. Due to the design of the cell, this value
229 corresponded to the average force transmitted to the porous stone at the bottom of the sample.
230 A plateau of 3 MPa was reached after 100 days of hydration. In the work of Molinero-Guerra
231 et al. (2018), an infiltration column test was performed on the same material with the same
232 conditions. The results showed the same kinetics of the axial swelling pressure (it increased
233 and reached a plateau after 100 days, but at 2.5 MPa).

234 After Wang et al. (2012, 2013c), the swelling pressure of pure bentonite, mixture of
235 bentonite/sand, and mixture of bentonite/claystone can be correlated with the dry density of
236 bentonite at the final (fully-saturated) state. A unique equation was proposed to predict the
237 swelling pressure of MX80 bentonite-based materials from the dry density of bentonite. In
238 addition, these works evidenced that the swelling pressure obtained by using synthetic
239 Callovo-Oxfordian claystone water was similar to that obtained by using distilled water due to
240 the low salinity of this synthetic water. Based on the equation proposed by Wang et al. (2012),
241 for a dry density of the mixture of 1.49 Mg/m^3 the axial swelling pressure value should equal
242 to 4.15 MPa. This value is higher than those observed in infiltration columns in the present
243 work and in the work of Molinero-Guerra et al. (2018). It is possible that the sample was not
244 completely saturated and the plateau observed did not correspond to the final swelling
245 pressure value.

246 **3.2.2. Investigation of the displacement field of the bentonite powder**

247 DVC technique was used to calculate the displacement field of PMMA spheres added to the
248 sample during the fabrication process. Figure 5 presents the results, which correspond to
249 vertical displacements occurring during the first 100 days of hydration (positive value
250 corresponds to an upward displacement; μ and σ represent average and standard deviation
251 values of the displacement, respectively). For each location (height), the displacements of
252 spheres located at ± 10 mm in the vertical direction were considered, and finally the mean
253 value was calculated.

254 The results show a sudden displacement of 0.2 – 0.4 mm of the PMMA spheres towards the
255 bottom after 2 days of hydration for the locations at 30, 50, 70 and 110 mm. Actually, these
256 locations correspond to the zone where air-filled inter-pellet voids were dominant (Figure 2).
257 Hydration induced swelling of the material at both ends, increasing the swelling pressure
258 (Figure 4), and provoked pellets re-arrangement. The movement of pellets would then induce
259 the movement of bentonite powder and PMMA spheres located in the inter-pellet voids. As
260 air-filled inter-pellet voids were dominant in these zones, bentonite powder and PMMA
261 spheres would tend to move downward because of gravity. This phenomenon was less visible
262 at 10 mm from the bottom because the bottom partly restricted this movement. At 90 mm, a
263 heave was observed during the first 2 days of hydration. First, the downward displacement of
264 PMMA spheres should have been limited in this zone because bentonite powder was
265 relatively dominant in the inter-pellet voids (see Figure 2). Second, material in this zone
266 would have tended to heave to fill the initial gap at the top during these first two days.

267 The above phenomena continued generally with the same trends until 11 days of hydration.
268 After this date, the movement of the PMMA sphere was negligible until 49 days of hydration.
269 Between 49 days and 56 days of hydration, a sudden downwards movement was found at all
270 the levels (except at 10 mm from the bottom). This movement might be explained by the
271 movement of the cell during its installation; the cell (the whole system shown in Figure 1a)

272 was removed from the microtomograph and put back to its position only during the duration
273 of the scans. This phenomenon might be equally used to explain the general downward
274 displacement between 56 days and 100 days of hydration.

275 **4. Conclusions**

276 The wetting-induced structure changes of a pellet/powder bentonite mixture, one of the
277 candidate materials for sealing plugs in deep geological high-level radioactive waste disposal,
278 were investigated in this work by X-ray CT observations.

279 The results show that the initial mixture was a heterogeneous granular material since the
280 distribution of pellets and powder was not spatially uniform. While wetting the top and
281 bottom sides, water infiltrated quickly into the inter-pellet pores in the vicinity of both
282 hydration fronts. That induced rapid swelling of pellets and powder of bentonite due to the
283 absorption of water by smectite, the principal mineral of bentonite. Firstly, X-ray CT
284 observations showed that the initial granular structure was quickly lost in these zones; pellets
285 lost their structure and the mixture became an apparently homogeneous material, with no
286 more air-filled inter-pellet voids. For the rest of the sample, this process depended on the
287 evolution of the hydration front. Secondly, at the same time, swelling of pellets induced their
288 re-arrangement which caused downward movement of the bentonite powder, induced by
289 gravity, in the zones having large air-filled inter-pellet voids. This phenomenon enhanced the
290 heterogeneous spatial distribution of bentonite powder inside the mixture. Finally, swelling of
291 pellets and the powder grains induced a quick increase of axial swelling pressure that reached
292 3 MPa after 100 days. This value was lower than the estimated swelling pressure of the same
293 material having identical average dry density after full saturation. This result suggested that
294 the mixture was not fully saturated after 100 days of hydration even if the X-ray CT
295 observations showed a relatively homogeneous structure at this time.

296 The results in this study allowed visualizing water infiltration inside the pellet/powder
297 bentonite mixture during hydration. That would contribute to a better understanding on the
298 hydro-mechanical couplings in this material, when it is installed in the real repository.

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378 **Tables**

379 Table 1. Chemical composition of the synthetic water.

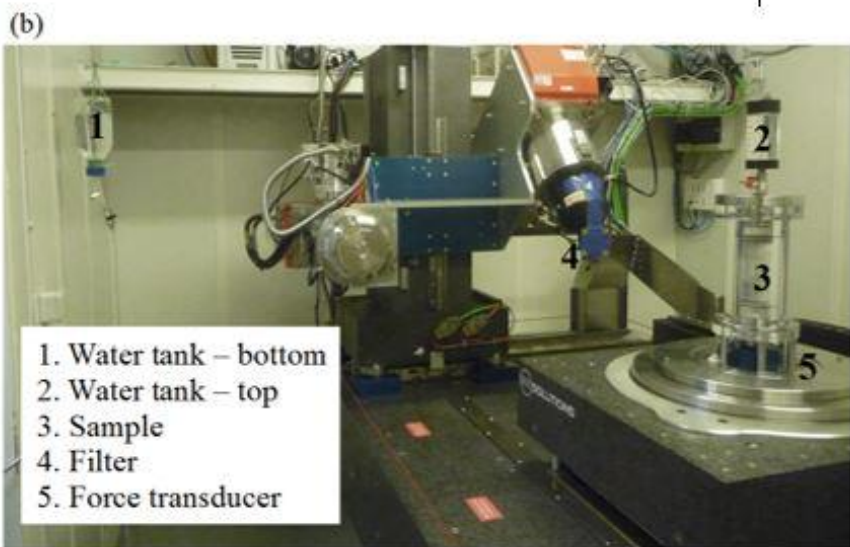
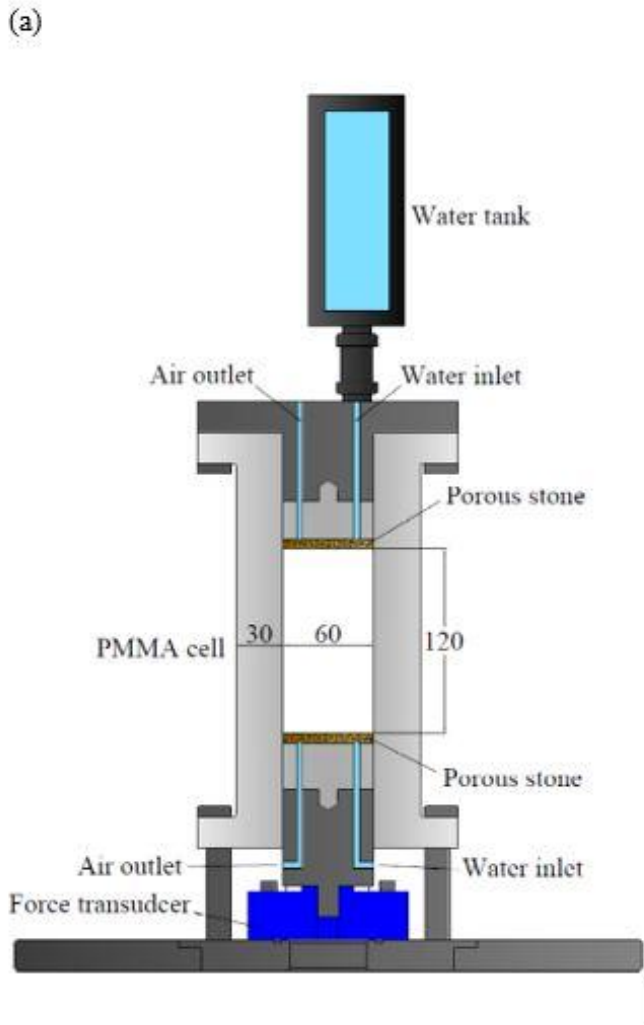
| Components | NaHCO ₃ | Na ₂ SO ₄ | NaCl | KCl | CaCl ₂ 2H ₂ O | MgCl ₂ ·6H ₂ O | SrCl ₂ ·6H ₂ O | Total |
|-----------------------------------|--------------------|---------------------------------|-------|-------|--|--------------------------------------|--------------------------------------|-------|
| Mass (g) per liter of solution | 0.28 | 2.216 | 0.615 | 0.075 | 1.082 | 1.356 | 0.053 | 5.677 |

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382 **Figures**

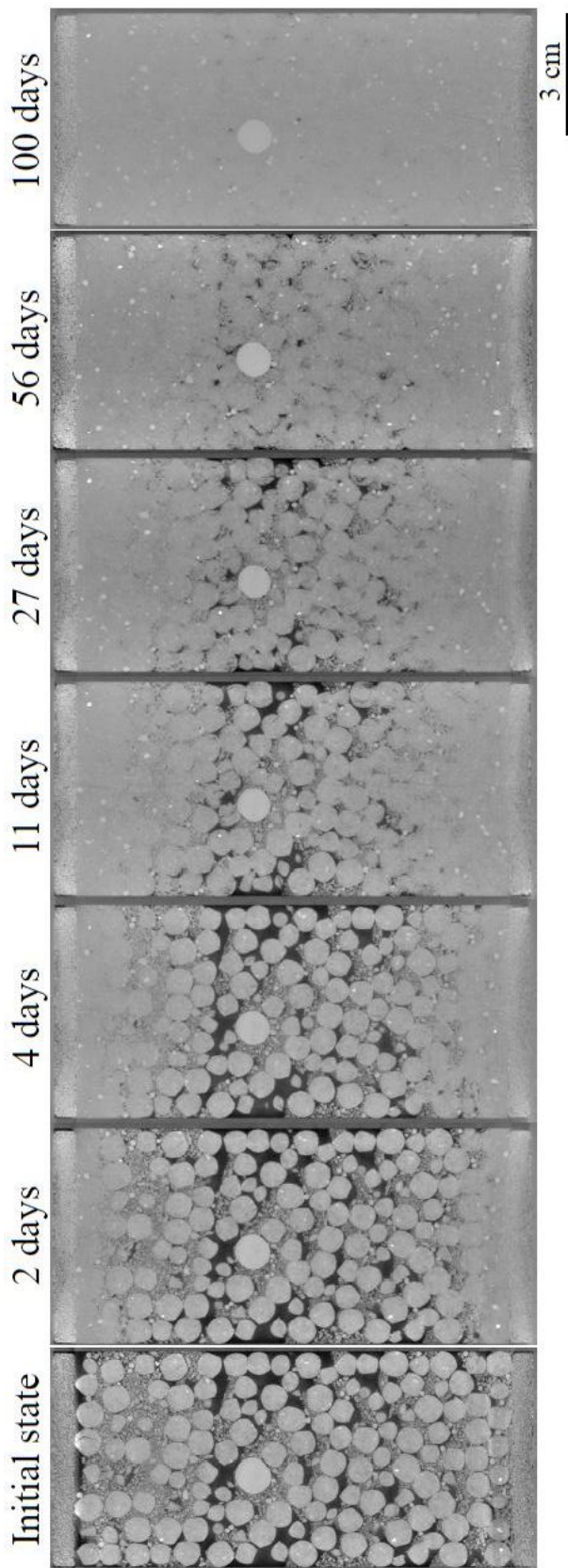
383 Figure 1. Photography of the experimental set-up inside the X-ray microtomograph device (b)
384 and layout of the cell (a).



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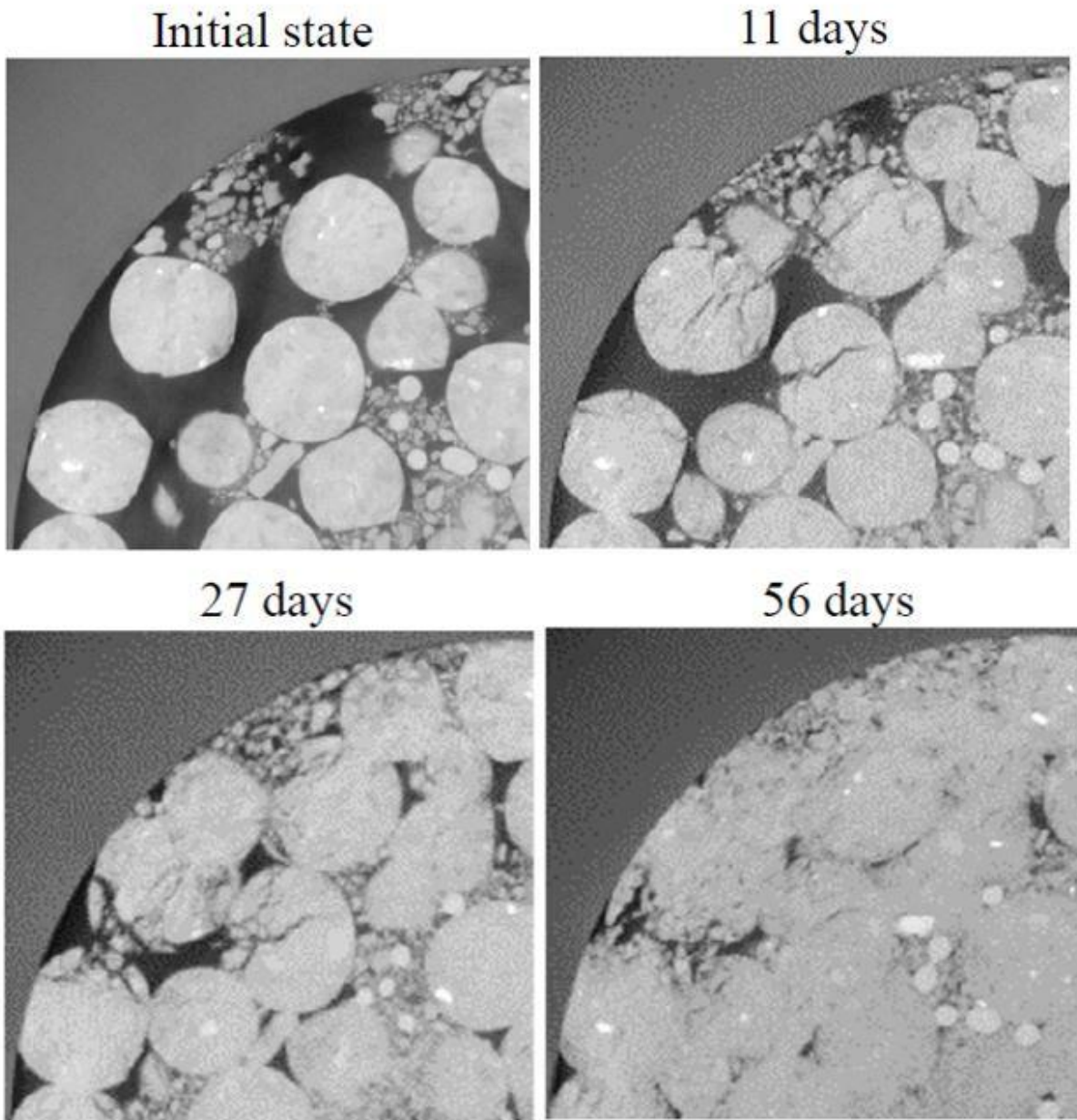
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Figure 2. Vertical sections of the sample at different times obtained by X-ray CT.



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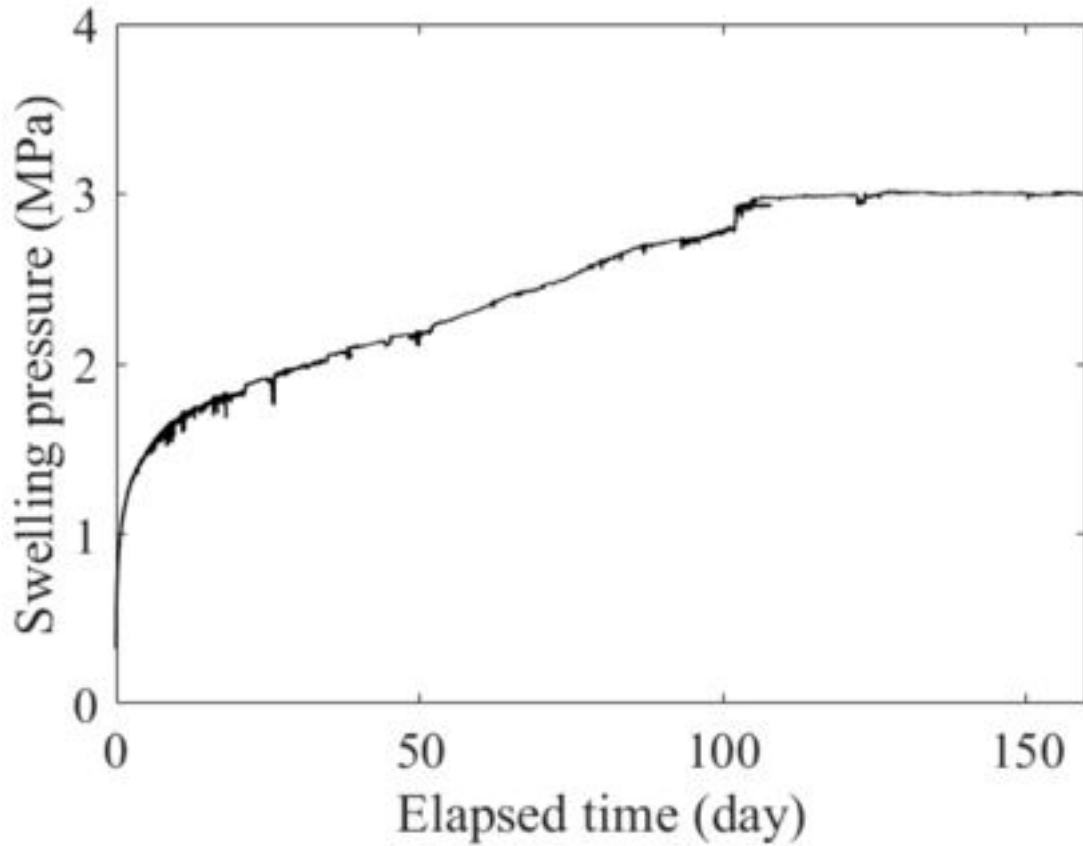
Figure 3. Enlargements of a horizontal section at 60 mm from the bottom obtained by X-ray CT.



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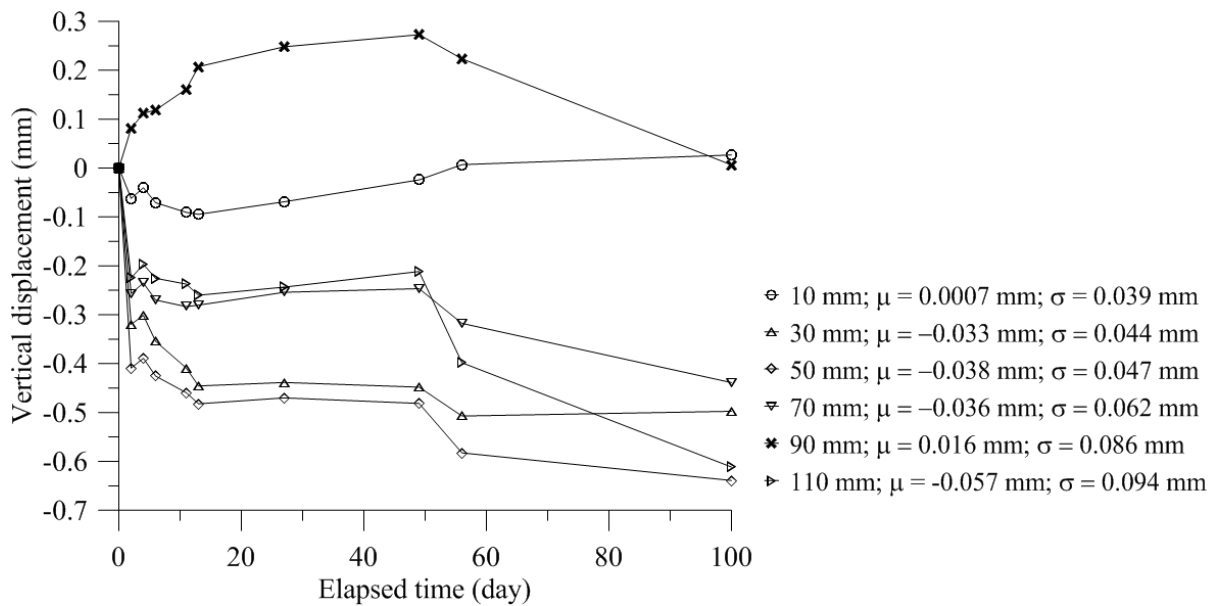
Figure 4. Evolution of the axial swelling pressure of the mixture.



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404 Figure 5. Vertical displacements obtained by Digital Volume Correlation (DVC) Technique
405 based on the PMMA spheres network while wetting.



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