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1 **Characterization of water retention, compressibility and swelling**  
2 **properties of a pellet/powder bentonite mixture**

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31 **Abstract:**

32 The water retention, compressibility and swelling properties of a pellet/powder bentonite  
33 mixture were investigated in the laboratory. The water retention capacity was determined under  
34 constant-volume condition using a specially designed cell allowing vapor exchange in all  
35 directions; suction controlled oedometer tests were carried out for the compressibility  
36 investigation; both constant-volume and swell-consolidation methods were applied for the  
37 swelling pressure determination. The vapor equilibrium technique was used for suction control.  
38 Comparable results were found for the water retention capacity of the pellet/powder mixture  
39 determined under constant-volume condition and one single pellet under free swelling condition  
40 for suctions higher than 4 MPa, suggesting that the suction was mainly controlled by the micro-  
41 pores inside the pellets/powder grains and the swelling of the clay particles was accommodated  
42 by the existing macro-pores. On the contrary, at lower suctions, the constant-volume conditions  
43 gave rise to a lower water retention capacity for the mixture, indicating the restricted clay  
44 particle swelling after the filling-up of the existing macro-pores. Oedometer compression  
45 curves revealed a different behavior for low and high suctions: at high suctions, the volume  
46 change behavior was governed by the pellets rearrangement combined with possible pellets  
47 crushing upon loading. By contrast, at low suctions, the mixture lost its initial granular structure  
48 during suction decrease; thus, the volume change behavior became similar to that of compacted  
49 bentonite. Compared to compacted bentonite, the pellet/powder mixture showed a lower yield  
50 stress. A lower value of swelling pressure was found with the constant-volume method,  
51 confirming the limitation of the swell-consolidation method in determining soil swelling  
52 pressure.

53

54 **Keywords:** pellet/powder bentonite mixture; laboratory testing; initial heterogeneity; water  
55 retention; swelling; compressibility

## 57 **1. Introduction**

58 The design of most deep geological repositories for high level radioactive wastes (HLW) is  
59 based on the multi-barrier concept which involves the natural host rock and engineered barriers  
60 (Sellin and Leupin, 2013). In this design concept, bentonite-based materials are used to  
61 construct buffers, backfills and seal plugs of disposal galleries and access shafts to ensure  
62 isolation of the waste from the environment. In particular, mixtures made up of bentonite  
63 powder and pellets are considered as a promising candidate sealing material because of its  
64 particular properties. In addition to their low permeability, high swelling capacity and high  
65 radionuclide migration retardation properties, the pellet/powder mixture exhibits operational  
66 advantages in terms of emplacement and avoids the formation of technological gaps between  
67 the host rock and the seal plug. Once installed in the repository, the pellet/powder mixture will  
68 undergo both hydraulic and mechanical loadings: infiltration of host rock pore water and  
69 mechanical confinement imposed by the surrounding rocks. In this context, to ensure the  
70 stability of the storage structure, thus the safety of the disposal system, it is of paramount  
71 importance to well understand the hydro-mechanical behavior of unsaturated bentonite  
72 pellet/powder mixture. For this purpose, the French Institute for Radiation Protection and  
73 Nuclear Safety (IRSN) launched the SEALEX project, which consisted of a series of in situ  
74 experiments in the Underground Research Laboratory (URL) in Tournemire, France (Mokni  
75 and Barnichon, 2016; Mokni, 2016). This project aimed at investigating the long-term hydraulic  
76 performance of sealing systems in normal and critical scenarios, with different sealing plug  
77 configurations (pre-compacted or field compacted sand/bentonite mixtures, pellet/powder  
78 bentonite mixtures). In parallel to the in situ experiments, mock-up tests were also carried out  
79 on compacted sand/bentonite blocks (Wang et al., 2012; Saba et al., 2014; Mokni, 2016). The  
80 swelling pressure of compacted sand/bentonite blocks was also determined by Wang et al.

81 (2013a). The results confirmed the dominating role of the block dry density, identified by many  
82 researchers (Börgesson et al., 1996; Dixon et al., 1996; Lloret et al., 2003; Villar, 2005; Gens  
83 et al., 2011; Villar et al., 2012; Schanz and Al-Badran, 2014). Further examination showed that  
84 sand in the mixture did not contribute to the swelling pressure; it was the dry density of  
85 bentonite contained in the mixture that defined the final swelling pressure of the sand/bentonite  
86 mixture (Wang et al., 2012; Saba et al., 2014), in agreement with the results obtained by  
87 previous researchers (e.g. Villar and Rivas, 1994; Karnland et al., 2008). The well-established  
88 relationship between final swelling pressure and bentonite dry density constituted a helpful tool  
89 in interpreting the field observation in the SEALEX project (Wang et al., 2013a).

90 As opposed to the compacted bentonite-based blocks, the pellet/powder bentonite mixture has  
91 been scarcely investigated in the laboratory. Hoffmann et al. (2007) performed different tests  
92 on mixtures of different-size pellets of Febex bentonite at different dry densities considered  
93 within the Engineered Barrier (EB) project. An important finding was the progressive decrease  
94 of water permeability induced by the clogging of the large inter-pellet pores due to granule  
95 swelling and the development of both swelling strain and pressure upon wetting. Imbert and  
96 Villar (2006) investigated the hydro-mechanical behavior of a 50/50 FoCa bentonite  
97 pellet/powder mixture by a series of infiltration tests. They concluded that the mixture became  
98 homogeneous with a swelling pressure equal to that of a bentonite powder compacted at the  
99 same dry density once full saturation was reached.

100 In this study, the water retention capacity and the swelling and compressibility properties of a  
101 80/20 pellet/powder MX80 bentonite mixture were investigated. Firstly, the water retention  
102 curve (WRC) was determined by following a wetting path under constant-volume conditions  
103 using a specially designed cell with vapor exchange allowed in all directions. This curve was  
104 compared to that of a single pellet of bentonite swelling under free swelling conditions.  
105 Secondly, a series of suction controlled oedometer tests were performed to investigate the

106 compressibility properties of the mixture. The swelling behavior of the mixture after following  
107 a wetting-drying path was also studied in oedometer. Finally, the swelling pressure was  
108 investigated using both constant-volume and swell-consolidation methods. Emphasis was put  
109 on the size of samples due to the strong heterogeneous nature of the material with large pellets.  
110 The results obtained allowed the main hydro-mechanical properties of the bentonite  
111 pellets/powder mixture used in the SEALEX project to be characterized, providing relevant  
112 parameters for the numerical analysis of the field infiltration experiments involving this  
113 mixture.

## 114 **2. Materials and methods**

### 115 **2.1. Materials**

116 The investigated material was a mixture of pellet/powder of MX80 bentonite at a proportion of  
117 80/20 in dry mass. The MX80 bentonite came from Wyoming, USA, and had a smectite content  
118 of 80%, other non-clayey inclusions being quartz, calcite and pyrite. The cation exchange  
119 capacity (CEC) was 98 meq/100g, with Na<sup>+</sup> as major exchangeable cation, with a value of 52  
120 meq/100g (1.2 meq/100g for K<sup>+</sup>, 10 meq/100g for Mg<sup>2+</sup> and 37 meq/100g for Ca<sup>2+</sup>). The liquid  
121 limit was 560%, the plastic limit was 62% and the unit mass of solid was 2.77 Mg/m<sup>3</sup> (Saba et  
122 al., 2014).

123 Pellets were produced in the Laviosa-MPC company by instantaneously compacting MX80  
124 bentonite in a mould of 7 mm in diameter and 7 mm in height. A zoom on a single pellet is  
125 shown in Figure 1. The fabrication water content was  $w = 6.0 \pm 1.0\%$  and the dry density was  
126  $\rho_d = 2.06 \pm 0.06 \text{ Mg/m}^3$ , corresponding to a void ratio  $e = 0.30 \pm 0.07$ . The pellets were stored  
127 in the laboratory in a hermetic plastic box at 20°C. The initial suction ( $s = 135 \pm 3 \text{ MPa}$ ) was  
128 measured in the laboratory with a chilled mirror dew point hygrometer (Decagon WP4C), at an  
129 initial water content  $w = 7.2 \pm 1.0\%$  (determined after oven-drying at 105°C), slightly higher

130 than the fabrication one due to the water adoption from air after fabrication (Molinero Guerra  
131 et al., 2017). Note that the WP4C measures corresponded to total suction, including the osmotic  
132 suction.

133 The MX80 bentonite powder was produced by crushing pellets just after their fabrication. The  
134 maximum size of grains was 2 mm and the portion of the grains smaller than 80  $\mu\text{m}$  was 5%.  
135 An initial water content of  $3.2 \pm 1.0\%$  was found in the laboratory by oven-drying at  $105^\circ\text{C}$  for  
136 24 h, corresponding to an initial suction  $s = 191$  MPa (measured with WP4C). This water  
137 content was quite low compared to the fabrication water content ( $6.0 \pm 1.0\%$ ), suggesting loss  
138 of some water during the pellets crushing operation. More details about the initial state of the  
139 material can be found in Molinero Guerra et al. (2017).

## 140 **2.1. Sample preparation**

141 All the results presented in this study were obtained by performing laboratory tests on the  
142 pellet/powder bentonite mixture. The mixture was prepared by following the first protocol  
143 presented in Molinero Guerra et al. (2017) to obtain a relatively homogeneous pellet/powder  
144 distribution. It consisted in filling the cell by packets corresponding to one layer of pellets  
145 spread over the base of the cylinder, with the corresponding amount of powder, respecting the  
146 proportion of 80-pellets/20-powder. The global dry initial density of the mixture thus prepared  
147 was  $1.49$  Mg/m<sup>3</sup>.

148 In order to better understand the initial pellet/powder distribution, microfocus X-ray computed  
149 tomography observations were made on the mixture at its initial state. A vertical slice is  
150 presented in Figure 2. The dimensions of the observed sample were 120 mm in height and 60  
151 mm in diameter. It appears that even though a special protocol was followed, it was difficult to  
152 perfectly control the homogeneity of the sample. Indeed, the grains of powder were not  
153 uniformly distributed within the sample, and inter-pellet voids could be clearly identified. The

154 sample, placed between two porous stones, presented larger inter-pellet voids close to the top  
155 porous stone and no grains of powder were observed in this zone. Therefore, this material was  
156 characterized by a strong heterogeneity at its initial state, which is an important aspect to take  
157 into account while further analyzing the global hydro-mechanical behavior of the sample. This  
158 initial heterogeneity was investigated in depth by Molinero Guerra et al. (2017).

159 The samples used for the present study were prepared in the same manner, but with a height of  
160 35 mm. This height corresponded to 5 times the height of a single pellet and it was believed  
161 that such dimensions constituted a representative volume of the pellet/powder mixture. This  
162 ratio of sample size to maximum grain size was also adopted by Duong et al. (2013) when  
163 investigating the mechanical behavior of a mixture of coarse and fine soils.

## 164 **2.2. Experimental methods**

### 165 *2.2.1. Water retention properties*

166 The water retention curve (WRC) of the pellet/powder bentonite mixture under constant-  
167 volume conditions was determined on samples of 50 mm in diameter. A special device  
168 presented in Figure 3 was designed for this purpose. The pellet/powder mixture was placed into  
169 a rigid stainless steel cell that was designed to allow vapor exchanges not only through the two  
170 metallic porous disks on both top and bottom of the sample, but also through a cylindrical  
171 porous disk on the lateral surface of the sample. This design was expected to significantly  
172 accelerate the hydration of the sample by vapor equilibrium technique, essential for low-  
173 permeability materials like the studied swelling MX80 bentonite. The mass of sample was  
174 regularly measured. In a standard fashion, equilibrium was considered once the mass changes  
175 became insignificant. For each pellet/powder mixture, once equilibrium was reached, three  
176 measures were performed for both the water content by oven drying and the suction with WP4C.  
177 The mean values were considered in the further analyses.



178 In parallel, the water retention curve under free swelling conditions for a single pellet of  
179 bentonite was obtained. To this end, pellets were hydrated under several values of suction from  
180 their initial state by vapor equilibrium technique. For the pellet/powder mixture, zero suction  
181 was applied by injecting deionized water into the cell under a low pressure corresponding to a  
182 water column height of about 200 mm.

### 183 2.2.2. *Compressibility properties – suction controlled oedometer tests*

184 Controlled suction oedometer compression tests were performed on samples of 35 mm in height  
185 and 50 mm in diameter by circulating vapor at controlled relative humidity through the base of  
186 the sample as shown in Figure 4 (tests SCO1, SCO2 and SCO3 presented in Table 1). In order  
187 to investigate the mechanical behavior of the mixture in a relatively large range of load, a high  
188 pressure oedometer was used allowing application of vertical stresses as high as 50 MPa  
189 (Marcial et al., 2002). This oedometer was previously calibrated using a force transducer in  
190 order to ensure the accuracy of the imposed pressures. As opposed to other suction values, zero  
191 suction was imposed by circulating deionized water. Vertical displacement was monitored  
192 using a digital micrometer (accuracy  $\pm 0.001$  mm). The temperature in the laboratory was  
193 controlled at  $20 \pm 0.5^\circ\text{C}$  during the experiments.

194 Time was allowed until swelling stabilized under the imposed value of suction and a low  
195 vertical stress of 0.1 MPa. The stabilization was considered when the vertical displacement was  
196 lower than 0.01 mm for, at least, 8 h. It is worth noting this criterion was defined based on the  
197 French Standard AFNOR (1995) for oedometer compression test. The real swelling  
198 stabilization might take much longer time. Three tests were carried out under three different  
199 suctions (0 MPa, 9 MPa and 138 MPa). The test at 138 MPa suction (SCO1) was performed  
200 without imposing the suction, assuming that the suction changes during loading-unloading were  
201 negligible. In this case, the test was performed at constant water content. This assumption was  
202 supported by the results of Gens et al. (1995) who measured the suctions of a soil compacted

203 under three different stresses and observed that on the dry side of optimum the soil suction was  
204 almost the same, suggesting that inter-aggregate macro-pores were dry. Similarly, in this study,  
205 the macro-pores of the pellets/powder mixture could be considered as dry at a suction as high  
206 as 138 MPa. Thus, it is assumed that compression decreased the macro-pores but not the suction  
207 of the mixture.

208 Loadings were performed in steps from 0.1 MPa. A new load equal to the double of the previous  
209 one was applied (except the last loading step for sample SCO1) when the change of the vertical  
210 displacement became insignificant ( $< 0.01$  mm for 8 h according to AFNOR, 1995). The  
211 maximums loads were 12.8 MPa for SCO2, SCO3 and SCO4 and 18 MPa for SCO1 because  
212 of the higher stiffness of this latter due to its higher suction. After the loading process,  
213 unloadings were performed in steps down to different load values.

### 214 2.2.3. *Swelling properties*

215 Three tests, SCO3, SCO4 and CV1 presented in Table 1, were performed in order to study the  
216 swelling properties of the pellet/powder bentonite mixture. Both the constant-volume and  
217 swell-consolidation methods were applied to determine the swelling pressure. To apply the  
218 constant-volume method, a constant volume cell was used, which included three parts: (i) the  
219 bottom part which contained a porous stone and a drainage system; (ii) the middle cell (70 mm  
220 in inner diameter) which prevented any radial swelling, with two air outlets; (iii) the top part  
221 including a total pressure sensor for monitoring the axial swelling pressure. The water inlet was  
222 connected to a water reservoir placed at the same height of the cell, flooding the pellet/powder  
223 mixture from the bottom. The saturation water used was a synthetic water (see Table 2 for the  
224 receipt of preparation), which has the same chemical composition as the pore water of the  
225 Callovo-Oxfordian claystone in the underground research laboratory of the French agency for  
226 radioactive waste management (ANDRA) in Bure, France. In the swell-consolidation method,  
227 the sample was hydrated under a low vertical load of 0.1 MPa until full swelling was achieved.

228 Then, it was compressed following a standard consolidation test procedure. The pressure  
229 needed to compress the specimen back to its initial volume was defined as the swelling pressure  
230 of the material (Basma et al., 1995; Abdullah and Mhaidib, 1998, 1999; Agus, 2005, Wang et  
231 al. 2012).

### 232 **3. Experimental results**

#### 233 *3.1. Water retention curve*

234 Figure 5 displays the evolution of water content with time for the pellet/powder mixture  
235 equilibrated at different suctions. The special cell designed allowed the samples to reach water  
236 content stabilisation quite quickly. This was because with the specially designed cell (Figure  
237 3), the sample was completely surrounded by porous stones, which accelerated the vapor  
238 exchange process. For all imposed suctions, the water content was stabilized after 30 days of  
239 hydration, except for 4 MPa and 82 MPa. For  $s = 4$  MPa, some technical problems occurred  
240 during the test, and a constant water content value was found after 80 days. It is suspected that,  
241 at this low suction, the hydration system failed after a certain time due to water condensation  
242 in the pipes – cleaning intervention was necessary to solve the problem. The slight temperature  
243 fluctuations of  $\pm 0.5^\circ\text{C}$  in the laboratory during the experiments could cause this condensation.  
244 For suction  $s = 82$  MPa, it seems that water content was constant after 30 days; nevertheless, it  
245 continued increasing slightly up to 90 days. For this value of suction, several problems were  
246 found during wetting in the hydration system (blockage of the vapor circulation system) and  
247 regular interventions were needed. It could be considered that this technique ran well for  
248 suctions higher than or equal to 9 MPa. For lower suctions, problems related to condensation  
249 in the pipes could be significant in the relatively long tests (more than 80 days). These problems  
250 are to be solved in further studies, by placing the main system in water with temperature control  
251 for example.

252 The water retention curves under constant (pellet/powder mixture) and free swelling conditions  
253 (single pellet) for a wetting path are presented in Figure 6 in terms of water content versus  
254 suction. It appeared that pellet and mixture had comparable water retention capacities when  
255 suction was higher than 4 MPa, suggesting that the suction was governed by the micro-pores  
256 within the pellets or powder grains. In addition, in the case of the mixture, the existing macro-  
257 pores can accommodate the swelling of the clay particles. This is in agreement with the results  
258 obtained by Yahia-Aissa et al. (2001) on compacted Foca 7 clay. It seems that the mixture had  
259 a lower water retention capacity under constant-volume conditions for suctions lower than 4  
260 MPa, since its water content at zero suction was 34.3%. This indicated the restriction effect on  
261 the clay particles swelling, thus the water retention capacity. More data are needed in this range  
262 of suction to confirm this conclusion. Figure 7 presents the suctions measured by WP4C versus  
263 the imposed values by either vapor equilibrium technique (for non-zero suctions) or injecting  
264 deionized water (for zero suction) for the pellet/powder mixture.

265 Some differences were found between the measured suction after equilibrium and the imposed  
266 value by vapor equilibrium technique, in particular for zero suction. This could be due to the  
267 stress release while dismantling the specimen, which led the suction to increase within the  
268 mixture. This effect appeared more pronounced in the case of zero suction due to the highest  
269 swelling pressure developed, which enhanced the stress release effect.

270 Pintado et al. (2009) concluded that, with the vapor equilibrium technique, it was difficult to  
271 reach the same suction distribution inside the sample. From Figure 7, it appears that the suction  
272 imposed and the suction measured were almost the same, indicating that in our case the uniform  
273 suction distribution was reached thanks to the additional air flow through the lateral surface.  
274 This shows the performance of the designed device.

275 3.2. *Controlled suction compression tests (SCO1, SCO2 and SCO3)*

276 Figure 8 presents the evolution of volumetric strain with time for tests SCO1, SCO2 and SCO3.  
277 The test at 138 MPa of suction was performed without imposing suction. It was carried out at  
278 constant water content conditions. As mentioned before, the suction can be reasonably  
279 considered as constant in this test.

280 In test SCO3, the pellet/powder mixture was hydrated under 0.1 MPa by injecting deionized  
281 water to impose zero suction. Equilibrium was reached after 1272 h (53 days), with a value of  
282 21% of volumetric strain, corresponding to a void ratio of  $e = 1.25$ . The volumetric strain at  
283 equilibrium was higher than the value obtained by Wang et al. (2012) for compacted  
284 bentonite/sand mixture: 18% ( $e = 0.97$ ). This could be explained by the higher swelling capacity  
285 of the pellet/powder mixture without sand. In test SCO2 for 9 MPa suction applied by vapor  
286 equilibrium technique, equilibrium was achieved after 1032 h (43 days), with a volumetric  
287 strain of 9% ( $e = 1.02$ ). A constant-volumetric strain was reached between 320 h and 430 h.  
288 This is due to a problem in the hydration system that needed a cleaning intervention. After  
289 430 h, the specimen continued swelling until reaching equilibrium. In test SCO1, no suction  
290 was imposed as 138 MPa corresponded to the initial state of the pellet/powder mixture; a period  
291 of 5 days was considered prior to loading in this case.

292 Once equilibrium was reached, the samples were submitted to loading/unloading at constant  
293 suctions. For SCO3 (zero suction), on the whole, the evolution of volumetric strain in each  
294 loading step showed typical consolidation features of fine-grained soils, in particular under high  
295 loads. On the contrary, for SCO1 (138 suction) and SCO2 (9 MPa suction), the evolution curves  
296 were rather characterized by the presence of almost instantaneous volumetric strains after each  
297 loading. It was thus suspected that when suction was higher than 9 MPa, it was rather the  
298 pellets/grains arrangement or crushing that governed the volume change behavior of the  
299 mixture.

300 Test SCO4 was designed to study the volume change behavior upon wetting/drying in  
301 oedometer. In this case, the pellet/powder mixture was submitted to a wetting-drying cycle at a  
302 constant vertical stress of 0.1 MPa. In the first phase, the specimen was hydrated by injecting  
303 deionized water during 1248 h (52 days) to reach equilibrium at zero suction (comparable to  
304 the 1272 h for test SCO3). A volumetric strain of 23% ( $e = 1.28$ ) was found at equilibrium.  
305 Then, a suction of 262 MPa was imposed to the mixture. The volumetric strain decreased  
306 drastically, reaching a value of 5% ( $e = 0.87$ ) after 1375 h (57.3 days). A final value of 3% ( $e$   
307  $= 0.84$ ) was observed after 2311 h (96.3 days).

308 Once equilibrated at the imposed suctions, the pellet/powder mixtures were submitted to  
309 compression at constant suctions. The results are displayed in Figure 9 in terms of void ratio  
310 with respect to vertical stress. The initial void ratio values were different due to the different  
311 swellings under different suctions. In a standard fashion, the compression curves were  
312 characterized by an initial linear part with low compressibility (that could be considered as the  
313 pseudo-elastic domain) followed by a second part with higher compressibility (that could be  
314 considered as the plastic domain). Finally, the third part, corresponding to the unloading,  
315 exhibited the same trend as the elastic domain, as only elastic deformations were expected. For  
316 test SCO3 where zero suction was applied, the unloading path could also be considered as bi-  
317 linear.

318 The swelling pressure of the pellet/powder mixture could be determined by the swell-  
319 consolidation method using the results from test SCO3. As it was explained previously, the  
320 swelling pressure of the material corresponded to the vertical stress needed to compress the  
321 sample to its initial volume. The obtained value was  $\sigma_s = 7.1$  MPa ( $e = 0.859$ ).

### 322 3.3. Swelling under constant-volume conditions (CVI)

323 The swelling behavior of the pellet/powder bentonite mixture under constant-volume  
324 conditions was investigated by following the evolution of the axial swelling pressure during  
325 hydration (CV1 in Table 1). The results are displayed in Figure 10. Two rates of increase of  
326 axial swelling pressure could be distinguished from the beginning of the hydration process: the  
327 first one corresponded to a period from the beginning of the hydration up to 20 days, when  
328 swelling pressure reached 0.43 MPa. Then, the pressure continued increasing at a different rate,  
329 until 45 days of hydration. At this moment, the swelling pressure reached 0.8 MPa. Afterwards,  
330 a slight increase was observed, finally reaching 0.87 MPa after 440 days. Nevertheless, the  
331 swelling pressure continued increasing, which means that the sample was not completely  
332 stabilized, even though the rate of increase was quite low compared to those observed before.  
333 The value of pressure observed after 440 days (0.87 MPa) was significantly lower than the  
334 value obtained by applying the swell-consolidation method on the consolidation curve (7.1  
335 MPa).

## 336 **Discussion**

337 The swell-consolidation and constant-volume method revealed different values of swelling  
338 pressure: the swell-consolidation method provided 7.1 MPa, whereas a value of 0.87 MPa was  
339 found with the constant-volume method. This was common results and Wang et al. (2012)  
340 explained that by the coupling between the microstructure deformation and the macrostructure  
341 deformation, combined to the effect of friction.

342 Cui et al. (2013) carried out oedometer tests with loading/unloading/reloading on natural stiff  
343 Ypresian clays (YPClay). They concluded that each unloading/reloading curve could be  
344 satisfactorily considered as bi-linear with a small and a larger slope separated by a threshold  
345 vertical stress. This stress was found, through hydrations tests under constant volume  
346 conditions, to be the swelling pressure corresponding to the void ratio just before the unloading

347 or reloading. By applying this approach to the pellet/powder mixture at zero suction (SCO3),  
348 with the consideration of the unloading path as bilinear, a swelling pressure of 4.2 MPa  
349 (threshold vertical stress) would be obtained, corresponding to a void ratio  $e = 0.72$  (dry density  
350  $\rho_d = 1.61 \text{ Mg/m}^3$ ). This value was comparable with that estimated by the method of Wang et al.  
351 (2012): a value of 4.15 MPa of swelling pressure was found at a dry density of  $1.49 \text{ Mg/m}^3$ .  
352 This showed that the true swelling pressure of the mixture should be lower than that determined  
353 by the swell-consolidation method, because the value would correspond to the point separating  
354 the two parts of unloading curve, thus below the consolidation curve.

355 Further analysis of the swelling pressure evolution with time displayed in Figure 10 revealed  
356 the processes governing the structural changes of the pellet/powder mixture while wetting. At  
357 the very beginning of the test with wetting from the bottom, the observed swelling pressure and  
358 the corresponding rate of increase were due to the pellets located at the top of the sample, where  
359 the suction was still high. After a period of 20 days, a second rate of increase higher than the  
360 first one was observed. The reason was that water has arrived to pellets or powder grains located  
361 at the top of the sample at this state; thus, the involved pellets and powder grains started swelling  
362 and the pressure increased accordingly. It was suspected that all inter-pellet/grain voids were  
363 filled with swollen bentonite after 40 days of hydration, as the rate of increase of swelling  
364 pressure decreased. However, the rate was not equal to zero, meaning that the specimen was  
365 not homogeneous. Taking into account the relationship proposed by Wang et al. (2013a)  
366 between the swelling pressure and the dry density of bentonite, a final value of 4.15 MPa of  
367 swelling pressure was found. Obviously, the swelling pressure of 0.87 MPa obtained by the  
368 constant method was quite far from this value. Molinero Guerra et al. (2018) performed an  
369 infiltration test on the same mixture with the same global dry density ( $1.49 \text{ Mg/m}^3$ ) using a  
370 metallic column (60 mm in diameter and 120 mm in height) equipped with a force transducer  
371 for the axial swelling pressure measurement and several total pressure transducers for the radial



372 swelling pressure measurements at different positions (20 mm, 40 mm, 60 mm, 80 mm and  
373 100 mm from the bottom of sample). Water infiltration was allowed from both the top and the  
374 bottom of sample. They observed an axial swelling pressure of 2.5 MPa after 845-day  
375 infiltration, also lower than the expected value (4.15 MPa). The radial pressures at 20 mm, 40  
376 mm, 60 mm, 80 mm were almost the same, around 4 MPa. By contrast, the value at 100 mm  
377 was lower, around 3 MPa. In addition, the swelling pressures were developed mainly during  
378 the first 100 days, the variations after 100 days appearing negligible. They attributed the  
379 difference between the radial swelling pressures by the heterogeneity of the sample: a higher  
380 swelling pressure was expected at a position where the local dry density was greater. They  
381 explained the lower axial pressure by the effect of friction between the sample and the column  
382 wall, as also identified by Saba et al. (2014). Indeed, the expected sample homogenization  
383 process implied the local movements of bentonite particles. Thus, the wall friction could be  
384 mobilized even in constant-volume condition. The results obtained in this study showed that  
385 even for a sample of 35 mm height, the effect of cell wall friction was significant, hampering  
386 or slowing down the sample homogenization process.

387 The results from test SCO4 allowed the reversibility of swelling of the pellet/powder mixture  
388 to be investigated. After wetting under zero suction, the sample was dried by imposing  $s = 262$   
389 MPa. The results showed a drastic decrease of volumetric strain, reaching a final value of 3%.  
390 This suggested that plastic swelling strain occurred upon wetting under zero suction, as shown  
391 in Figure 11. This was probably due to pellets fissuring upon wetting. Wang et al. (2013b) also  
392 reported the fissuring phenomenon for compacted bentonite/sand mixture upon wetting.  
393 Compared to test SCO3, a slightly higher volumetric strain was reached in test SCO4,  
394 suggesting a certain effect of the pellet/powder distribution that is specific for each sample. The  
395 final points of tests SCO1, SCO2 and SCO3 appeared well aligned, defining a wetting line. This

396 line was below the drying line estimated from test SCO4. The difference between the two lines  
397 was larger at higher suction.

398 The evolution of the yield stress with suction is presented in Figure 12, together with the values  
399 obtained by Marcial et al. (2003) and Wang et al. (2013b) for compacted bentonite with initial  
400 void ratio of 0.559 and compacted bentonite/sand mixture with initial void ratio of 0.635,  
401 respectively. A good coincidence was observed for compacted bentonite and the pellet/powder  
402 mixture at zero suction. For other values of suction, in a standard fashion, the yield stress  
403 increased as suction increased. However, this phenomenon was not clear for the pellet/powder  
404 mixture: a much lower yield stress value was obtained. A possible explanation is as follows:  
405 for non-zero suctions, the pellet/powder mixture did not completely lose its initial granular  
406 mixture; thus, the deformations were essentially governed by the rearrangement and possible  
407 crushing of pellets while loading. These deformations were of plastic nature. However, in the  
408 case of zero suction, the mixture entirely lost its granular structure; thus, the volume change  
409 behavior was comparable to that of a common compacted bentonite. This observation was  
410 supported by analyzing the evolution of volumetric strain with time: at 138 MPa and 9 MPa of  
411 suctions, the volumetric strains decreased almost instantaneously while loading; however, at  
412 zero suction, the decrease rate of volumetric strain was clearly lower than those observed at  
413 other suctions, because the pellets lost their granular structure in that case.

#### 414 **4. Conclusion**

415 The water retention, compressibility and swelling properties of a 80-pellet/20-powder bentonite  
416 mixture were investigated through the water retention tests under free (pellet) and constant-  
417 volume (mixture) conditions, the suction controlled oedometer tests and the hydraulic tests on  
418 samples at a dry density of 1.49 Mg/m<sup>3</sup>.

419 The new cell designed for the WRC determination under constant-volume condition allowed  
420 the vapor exchange in all directions, which significantly accelerated the hydration process.

421 Similar water retention properties were observed for pellet/powder mixture under constant-  
422 volume condition and single pellet under free swelling condition at suction higher than 4 MPa,  
423 indicating that the suction was mainly controlled by the micro-pores inside the pellets or powder  
424 grains, and the swell of clay particles was accommodated by existing macro-pores in the  
425 mixture. On the contrary, at lower suctions, constant-volume condition defined a lower water  
426 retention capacity, showing the restriction effect on clay particles swelling and thus their  
427 hydration. Note that more tests are needed to complete the data in the range of suctions lower  
428 than 4 MPa.

429 The investigation of the compressibility properties revealed lower values of yield stress than  
430 the common bentonite mixtures, except for the specimen hydrated at zero suction. This is due  
431 to the granular structure in the case of non-zero suction where the volume change behavior was  
432 suspected to be governed by the rearrangement and crushing of pellets, and the loss of the  
433 granular structure in the case of zero suction.

434 The irreversibility of the volumetric strain was evidenced by imposing a wetting-drying cycle  
435 to the pellet/powder mixture under constant loading. This was probably due to the pellets/grains  
436 fissuring upon wetting.

437 Different swelling pressure values were obtained by applying different methods. A higher  
438 swelling pressure was obtained with the swell-consolidation method. Further analysis based on  
439 the method proposed by Cui et al. (2013) showed that the swell-consolidation systematically  
440 overestimates the swelling pressure because for a given density the swelling pressure must be  
441 lower than the corresponding pressure on the consolidation curve: the swelling pressure  
442 corresponded to the point separating the two parts of unloading curve, thus below the

443 consolidation curve. Nevertheless, the swelling pressure determined by the constant-volume  
444 method seemed quite low (0.87 MPa) with respect to the expected one (4.15 MPa). This could  
445 be attributed to the effect of cell wall friction because the expected sample homogenization  
446 process implied the local movements of bentonite particles, thereby mobilizing the cell wall  
447 friction even in constant-volume condition and with a sample of limited height (35 mm). The  
448 cell wall friction would hamper or slow down the sample homogenization process.

## 449 **References**

- 450 Abdullah, I., Mhaidib, A., (1998). Prediction of swelling potential of an expansive shale.  
451 Proceedings Of The Second International Conference On unsaturated soils. 27–30  
452 August. Beijing, China, vol. 1.
- 453 Abdullah, I., Mhaidib, A., (1999). Swelling behaviour of expansive shales from the middle  
454 region of Saudi Arabia. *Geotechnical and Geological Engineering* 16 (4), 291–307.
- 455 AFNOR, (1995). Sols : reconnaissance et essais: essai de gonflement à l'oedomètre,  
456 détermination des déformations par chargement de plusieurs éprouvettes. XP P 94-091
- 457 Agus, S., (2005). An Experimental study on hydro-mechanical characteristics of com-  
458 pacted bentonite-sand mixtures. PhD thesis. Weimar.
- 459 Basma, A.A., Al-Homoud, A.S., Husein, A., (1995). Laboratory assessment of swelling pres-  
460 sure of expansive soils. *Applied Clay Science* 9 (5), 355–368.
- 461 Börgesson, L., Karnland, O. and Johannesson, L. E., (1996). Modelling of the physical  
462 behaviour of clay barriers close to water saturation. *Engineering Geology* 41, No. 1–4,  
463 127–144.
- 464 Cui, Y. J., Nguyen, X. P., Tang, A. M., and Li., X. L., (2013). An Insight into the  
465 Unloading/reloading Loops on the Compression Curve of Natural Stiff Clays. *Applied*  
466 *Clay Science* 83-84: 343–48.
- 467 Dixon, D. A., Gray, M. N. and Graham, J., (1996). Swelling and hydraulic properties of  
468 bentonites from Japan, Canada and the USA. *Environmental Geotechnics* 1, 43–48.
- 469 Duong, T. V., Tang, A. M., Cui, Y. J., Trinh, V. N., Dupla, J. C., Calon, N., Canou, J., and  
470 Robinet, A. (2013). Effects of fines and water contents on the mechanical behavior of  
471 interlayer soil in ancient railway sub-structure, *Soils Found.*, vol. 53, no. 6, pp. 868–878.
- 472 Gens, A., Alonso, E.E., Suriol, J. & Lloret, A. (1995). Effect of structure on the volumetric  
473 behaviour of a compacted soil. Proc. of 1<sup>st</sup> conference on unsaturated soils. vol. 2, pp.  
474 83-88.

- 475 Gens, A., Vallejan, B., Sánchez, M., Imbert, C., Villar, M.V. and Van Geet, M., (2011).  
 476 Hydro mechanical Behavior of a Heterogenous Compacted Soil: Experimental  
 477 Observations and Modelling.
- 478 Hoffmann, C., Alonso, E.E. & Romero, E., (2007). Hydro-mechanical behaviour of bentonite  
 479 pellet mixtures. *Physics and Chemistry of the Earth*, 32(8-14), pp.832–849.
- 480 Imbert, C. & Villar, M.V., (2006). Hydro-mechanical response of a bentonite pellets/powder  
 481 mixture upon infiltration. *Applied Clay Science*, 32(3-4), pp.197–209.
- 482 Karnland, O., Nilsson, U., Weber, H., Wersin, P., (2008). Sealing ability of Wyoming  
 483 bentonite pellets foreseen as buffer material – laboratory results. *Physics and Chemistry  
 484 of the Earth, Parts A/B/C* 33, S472–S475.
- 485 Lloret, A., Villar, M. V., Sánchez, M., Gens, A., Pintado, X. and Alonso, E. E., (2003).  
 486 Mechanical behaviour of heavily compacted bentonite under high suction changes.  
 487 *Géotechnique* 53, No. 1, 27–40.
- 488 Marcial, D., Delage, P., Cui, Y.J., (2002). On the high stress compression of bentonites.  
 489 *Canadian Geotechnical Journal* 39, 812–820.
- 490 Marcial, D., (2003). Comportement hydromécanique et microstructural des matériaux de  
 491 barrière ouvragée, thèse. ENPC Paris, France.
- 492 Mokni, N. & Barnichon, J.D., (2016). Hydro-mechanical analysis of SEALEX in situ tests-  
 493 Impact of technological gaps on long term performance of repository seals. *Engineering  
 494 Geology*, 205, pp. 81-92.
- 495 Mokni, N., (2016). Analysis of hydro-mechanical behaviourbehavior of compacted  
 496 bentonite/sand mixture using a double structure formulation. *Environ Earth Sci* (2016)  
 497 75: 1087.
- 498 Molinero-Guerra, A., Mokni, N., Delage, P., Cui, Y. J., Tang, A. M., Aïmedieu, P., Bernier,  
 499 F. and Bornert, M., (2017). In-depth characterisation of a mixture composed of  
 500 powder/pellets MX80 bentonite. *Applied Clay Science*, 135, pp. 538-546.
- 501 Molinero-Guerra, A., Cui, Y. J., Mokni, N., Delage, P., Bornert, M., Aïmedieu, P., Tang, A.  
 502 M., Bernier, F. and (2018). Investigation of the hydro-mechanical behaviour of a  
 503 pellet/powder MX80 betonite mixture using an infiltration column. *Engineering  
 504 Geology*, 243, pp. 18-25.
- 505 Pintado, X., Lloret, A., Romero, E., (2009). Assessment of the use of the vapour equilibrium  
 506 technique in controlled-suction tests. *Canadian Geotechnical Journal* 46: 411-123
- 507 Saba, S., Cui, Y.J., Tang, A.M., and Barnichon, J.D., (2014). Investigation of the swelling  
 508 behaviour of compacted bentonite–sand mixture by mock-up tests. *Canadian  
 509 Geotechnical Journal*, 51(12), pp.1399–1412.
- 510 Sellin, P., and Leupin, O. X., (2013). The use of clay as an engineered barrier in radioactive-  
 511 waste management – a review. *Clays and Clay Minerals* 61, No. 6, 477–498.

- 512 Schanz, T. and Al-Badran, Y., (2014). Swelling pressure characteristics of compacted Chinese  
513 Gaomiaozi bentonite GMZ01. *Soils and Foundations* 54, No. 4, 748–759.
- 514 Villar, M.V., Rivas P. (1994). Hydraulic properties of montmorillonite-quartz and saponite-  
515 quartz mixtures. *Applied Clay Science* 9, 1-9
- 516 Villar, M.V. (2005). MX-80 Bentonite. Thermo-Hydro-Mechanical Characterization  
517 Performed at CIEMAT in the Context of Prototype Project. Ciemat technical report  
518 1053. Madrid, Spain.
- 519 Villar, M. V., Gómez-Espina, R. and Guitiérrez-Nebot, L., (2012). Basal spacings of smectite  
520 in compacted bentonite. *Applied Clay Science* 65–66, 95–105.
- 521 Wang, Q., Tang, A. M., Cui, Y. J., Delage, P., and Gatmiri, B., (2012). Experimental study on  
522 the swelling behaviour of bentonite/claystone mixture. *Eng. Geol.*, vol. 124, no. 1, pp.  
523 59–66.
- 524 Wang, Q., Tang, A.M., Cui, Y.J., Barnichon, J.D. and Ye, W.M., (2013a). A comparative stud  
525 on the hydro-mechanical behaviour of compacted bentonite/sand plug based on  
526 laboratory and field infiltration tests. *Eng. Geol.* 162, 79–87.
- 527 Wang, Q., Tang, A.M., Cui, Y.J., Barnichon, J.D. Ye, W.M., (2013b). Investigation of the  
528 hydro-mechanical behaviour of compacted bentonite/sand mixture based on the BExM  
529 model. *Computer and Geotechnics* 54, 46-52.
- 530 Yahia-Aissa, M., Delage, P., Cui, Y.J., (2001). Suction–water relationship in swelling clays.  
531 *Clay Science for Engineering*. In: Adachi, K., Fukue, M. (Eds.), *Proceedings of the IS-*  
532 *Shizuoka International Symposium on Suction, Swelling, Permeability and Structure of*  
533 *Clays*. Shizuoka, Japan Balkema, pp. 65–68.
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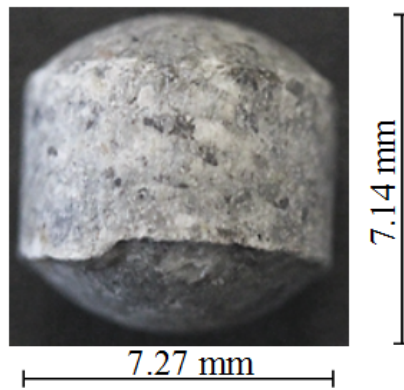
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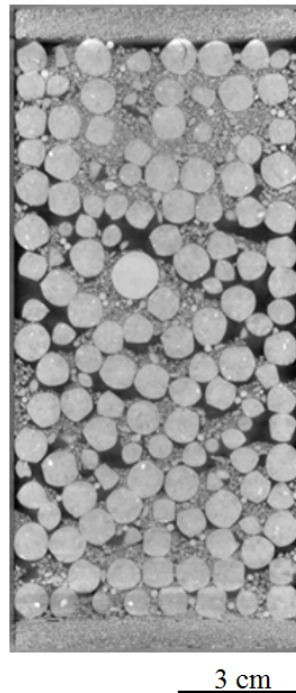


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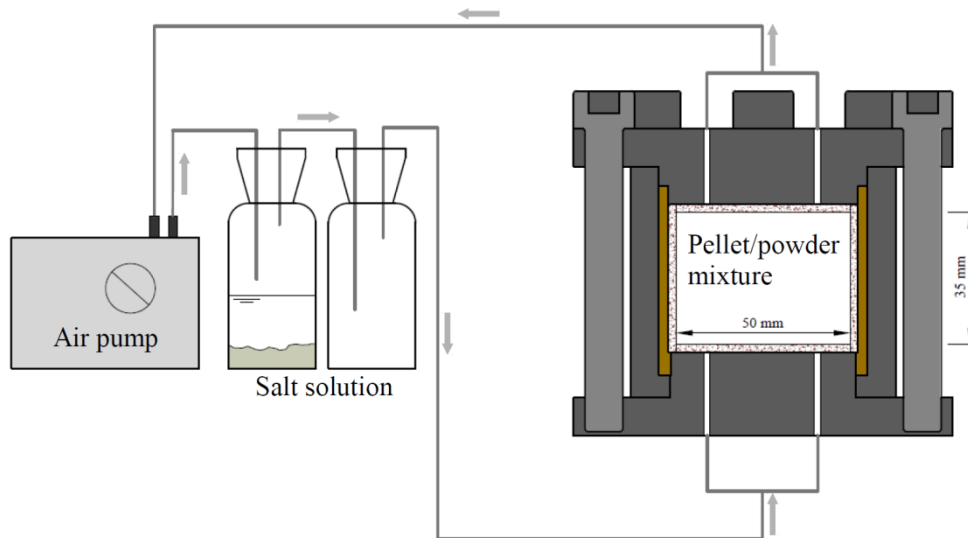
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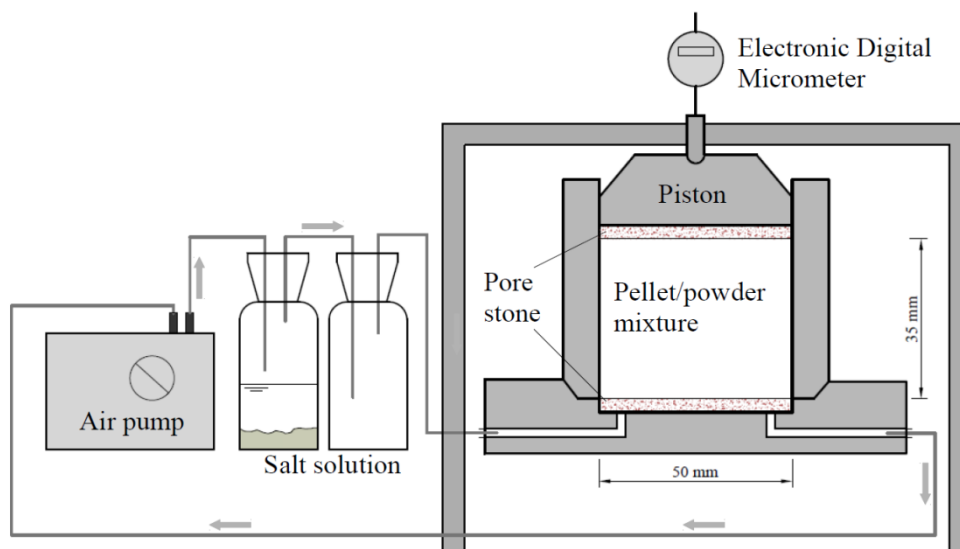
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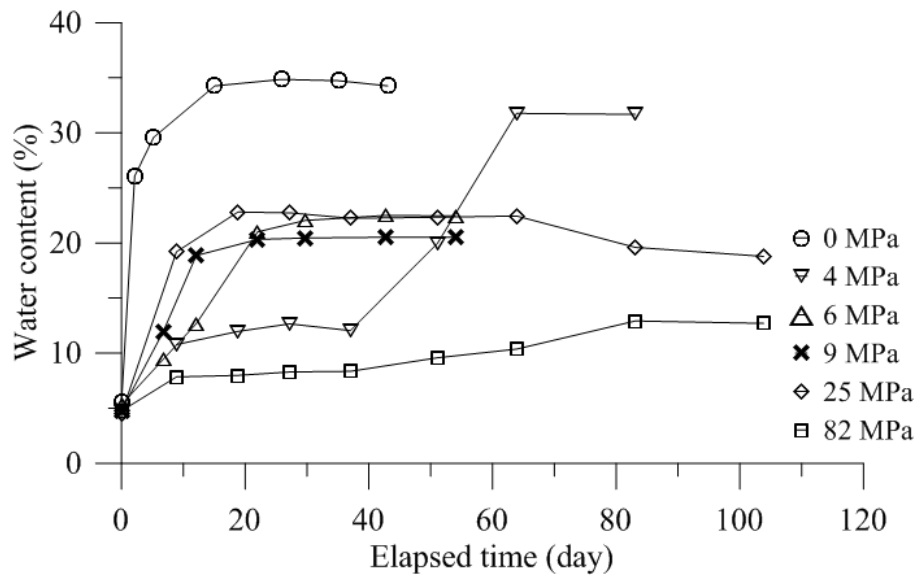
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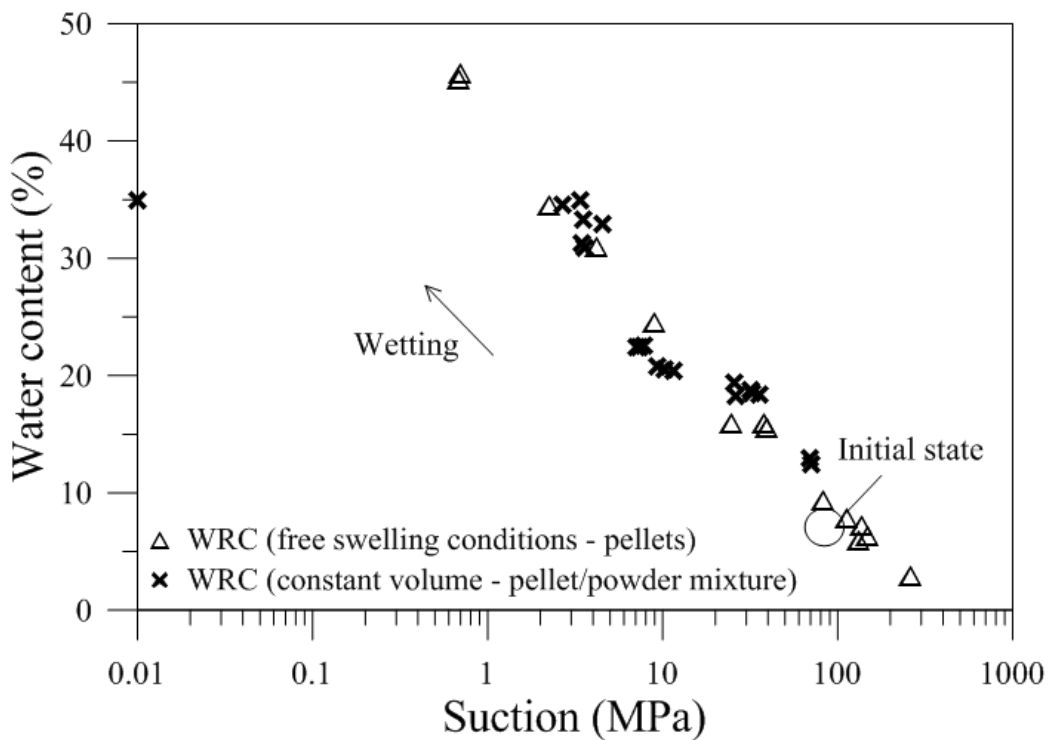
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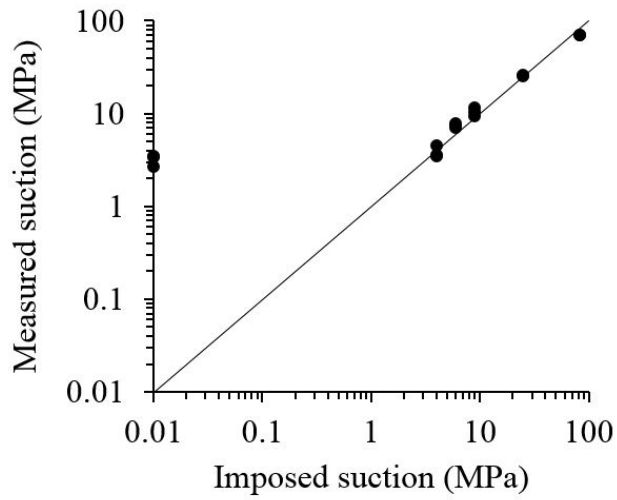
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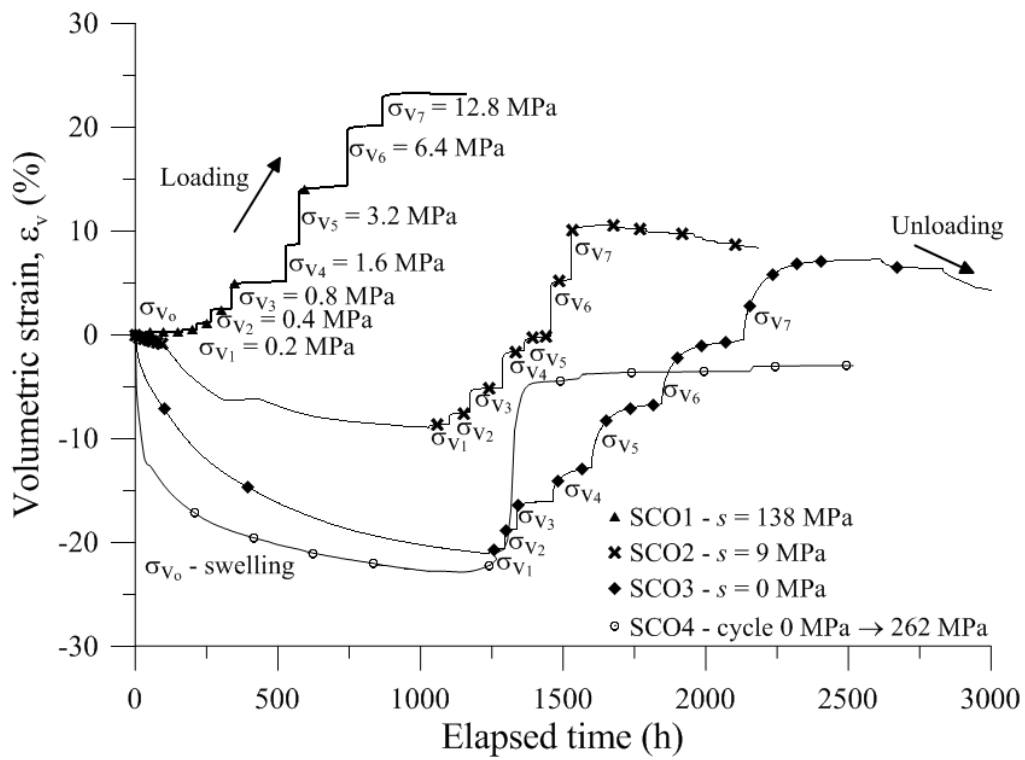


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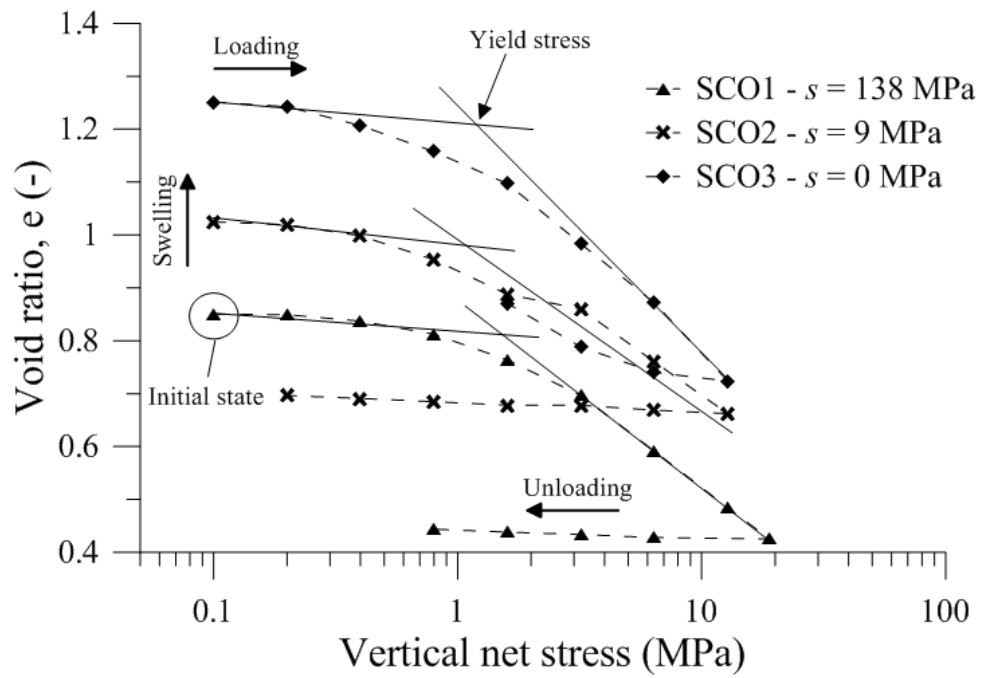
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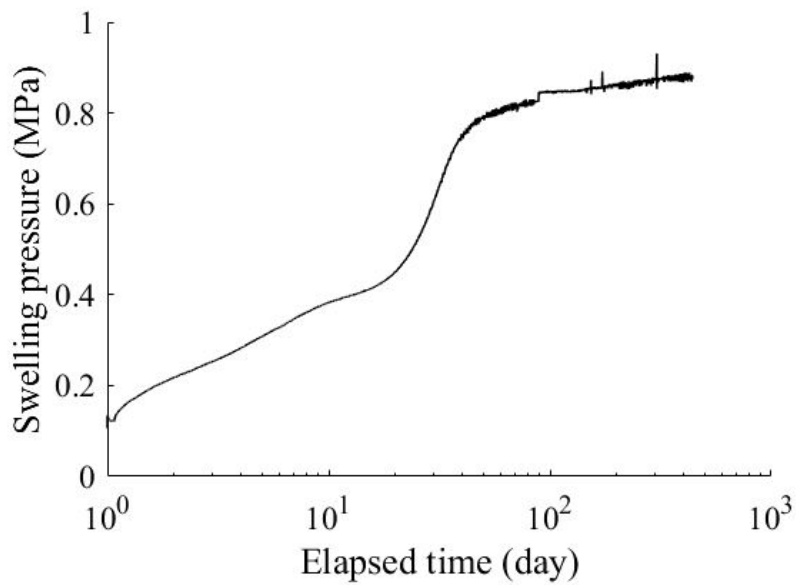


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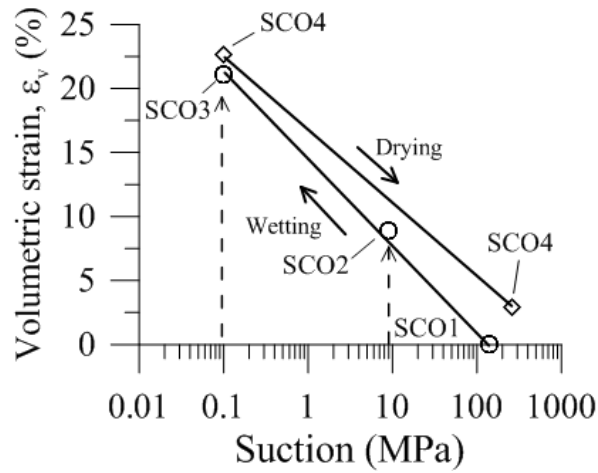


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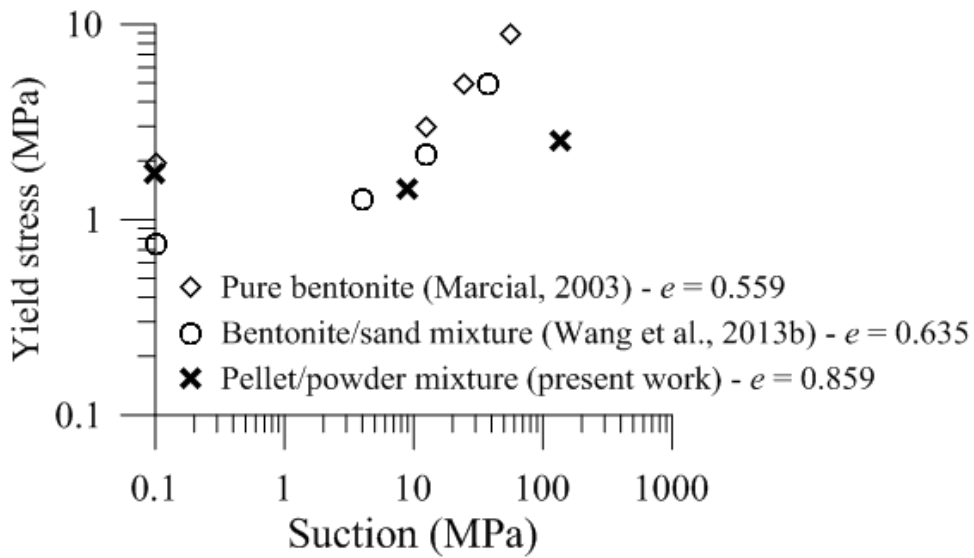
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Table 1. Test program for investigating the compressibility and swelling properties of pellet/powder bentonite mixture.

Test No.	Hydration condition	Suction (MPa)	Investigated property	Method
SCO1	Constant water content	138	Compressibility	-
SCO2	Vapor transfer	9	Compressibility	-
SCO3	Injected deionized water	0	Compressibility and swelling	Swell-consolidation
SCO4	Injected deionized water and vapor transfer	0 → 262	Swelling	-
CV1	Injected synthetic water	0	Swelling	Constant-volume

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Table 2. Receipt for preparing the synthetic water.

Components	NaHCO <sub>3</sub>	Na <sub>2</sub> SO <sub>4</sub>	NaCl	KCl	CaCl <sub>2</sub> 2H <sub>2</sub> O	MgCl <sub>2</sub> O6H <sub>2</sub> O	SrCl <sub>2</sub> 6H <sub>2</sub> O
Mass (g) per litre of solution	0.28	2.216	0.615	0.075	1.082	1.356	0.053

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