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# Effect of water chemistry on the hydro-mechanical behaviour of compacted mixtures of claystone and Na + /Ca 2+ bentonites for deep geological repositories for deep geological repositories

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1      **Effect of water chemistry on the hydro-mechanical behaviour of compacted**  
2      **mixtures of claystone and Na<sup>+</sup>/Ca<sup>2+</sup> bentonites for deep geological repositories**  
3                                 for deep geological repositories

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23 **Abstract:** In the French deep geological disposal for radioactive wastes, compacted  
24 bentonite/claystone mixtures have been considered as possible sealing materials. After  
25 emplacement in place, such mixtures are hydrated by the site solution as well as the cement  
26 solution produced by the degradation of concrete. In this study, the effects of synthetic site  
27 solution and cement solution on the hydro-mechanical behaviour of compacted mixtures of  
28 claystone and two types of bentonites ( $\text{Na}^+$  MX80 and  $\text{Ca}^{2+}$  Sardinia bentonites) were investigated  
29 by carrying out a series of swelling pressure, hydraulic conductivity and Mercury Intrusion  
30 Porosimetry (MIP) tests. It was found that for the MX80 bentonite/claystone mixture hydrated  
31 with synthetic site solution, the swelling capacity was reduced compared to the case with  
32 deionised water owing to the transformation of Na-montmorillonite to multi-cation dominant  
33 montmorillonite by cation exchanges. For the Sardinia bentonite/claystone mixture, the similar  
34 increasing rate of swelling pressure was observed during the crystalline swelling process for  
35 different solutions, suggesting insignificant cation exchanges. Additionally, the cations in the  
36 synthetic site solution could reduce the thickness of diffuse double layer and the osmotic swelling  
37 for both MX80 bentonite/claystone and Sardinia bentonite/claystone mixtures. The large-pore  
38 volume increased consequently and enhanced water flow. In the cement solution, the hydroxide  
39 could also dissolve the montmorillonite, reducing the swelling pressure, and increased the large-  
40 pore volume, facilitating the water flow. Furthermore, the decrease of swelling pressure and the  
41 increase of hydraulic conductivity were more significant in the case of low dry density because  
42 of more intensive interaction between montmorillonite and hydroxide thanks to the high  
43 permeability.

44 **Keywords:** bentonite/claystone mixture; synthetic site solution; cement solution; bentonite type;  
45 swelling pressure; hydraulic conductivity

## 46 **1. Introduction**

47 Deep geological repository has been accepted as a solution for the long-term storage of high-level  
48 radioactive wastes in many countries ([Sellin and Leupin, 2013](#)). To ensure the long-term safe isolation  
49 of radioactive wastes in deep geological formations, bentonite-based materials are commonly used as  
50 sealing materials ([Pusch, 1982](#); [Dixon et al., 1985](#); [Villar and Lloret, 2008](#); [Ye et al., 2010](#)), while  
51 cement (in mortar or concrete) is used to construct the tunnel retaining structures and tunnel  
52 plugs/seals ([Sánchez et al., 2006](#)). After the repositories are closed, the bentonite-based materials will  
53 be hydrated by the pore water of the host rock and swell, filling the technological voids and restricting  
54 the release of radionuclides from the radioactive wastes to the surrounding environment. It has been  
55 reported that the pore water of the host rock considered in several countries is of certain salinity ([Deng  
56 et al., 2011](#); [Nguyen et al., 2013](#); [Wang et al., 2014](#); [Sun et al., 2018](#)). In addition, the cement in  
57 contact with groundwater for a long period can degrade and release a large number of  $\text{Ca}^{2+}$  and  $\text{OH}^-$ ,  
58 resulting in a high-pH alkaline solution ([Savage et al., 2002](#); [Karnland et al., 2007](#); [Anh et al., 2017](#);  
59 [Sun et al., 2018](#)). In the performance assessment of the storage repositories, it appears essential to  
60 account for the effect of such water chemistry on the hydro-mechanical behaviour of bentonite-based  
61 materials.

62 In the past decades, the influence of saline solution on the hydro-mechanical behaviour of  
63 bentonite-based materials was studied by many researchers ([Karnland et al., 2006](#); [Rao et al., 2006](#);  
64 [Castellanos et al., 2008](#); [Komine et al., 2009](#); [Zhu et al., 2013](#)). It was found that the pore water  
65 salinity could significantly decrease the swelling pressure ([Komine et al., 2009](#); [Zhu et al., 2013](#)) and  
66 increase the hydraulic conductivity ([Castellanos et al., 2008](#); [Zhu et al., 2013](#)). [Yukselen-Aksoy et al.  
67 \(2008\)](#) and [Zhu et al. \(2013\)](#) reported that the cations in the pore water might alter the mineralogical

68 compositions through cation exchanges and influence the crystalline swelling process, resulting in a  
69 lower swelling pressure. Additionally, the cations in the pore water could decrease the thickness of  
70 diffuse double layer, affecting the osmotic swelling behaviour ([Siddiqua et al., 2011](#); [Castellanos et  
71 al., 2008](#); [Schanz and Tripathy, 2009](#); [Zhu et al., 2013](#); [Du et al., 2021](#)). At a given void ratio, a larger  
72 large-pore volume could be expected for the specimen hydrated with a saline solution, leading to a  
73 higher hydraulic conductivity.

74 In addition to the saline solution, alkaline solution can also significantly influence the hydro-  
75 mechanical behaviour of bentonite-based materials ([Karnland et al., 2007](#); [Herbert et al., 2008](#); [Lee  
76 et al., 2012](#); [Chen et al., 2016](#); [Sun et al., 2018, 2019, 2020](#); [Liu et al., 2020](#)). [Chen et al. \(2016\)](#)  
77 experimentally determined the swelling pressures and hydraulic conductivities of GMZ bentonite  
78 hydrated with various NaOH solutions and found that the final swelling pressure decreased and the  
79 hydraulic conductivity increased significantly with the increase of NaOH solution concentration.  
80 [Karnland et al. \(2007\)](#) investigated the constant-volume swelling pressures of compacted Wyoming  
81 MX80 bentonite hydrated with NaOH and Ca(OH)<sub>2</sub> solutions and found that 0.1 M NaOH (pH=12.9)  
82 or saturated Ca(OH)<sub>2</sub> solutions (pH=12.4) did not significantly change the swelling pressure of  
83 compacted MX80 bentonite. In contrast, [Sun et al. \(2018; 2019\)](#) compared the swelling pressures and  
84 mineralogical compositions of GMZ bentonite hydrated with Beishan site solution (pH=8.5), young  
85 concrete water (pH=13) and evolved concrete water (pH=12) and observed a remarkably lower  
86 swelling pressure in the case of young concrete water. They explained this phenomenon by the  
87 dissolution of montmorillonite upon saturation with alkaline solutions and the formation of some less  
88 active zeolites and calcium hydrated silicates. Additionally, the montmorillonite content decreased  
89 linearly as the pH increased ([Sun et al., 2019](#)).

90 In France, Callovo-Oxfordian (COx) claystone has been considered as a potential geological  
91 host rock for high-level radioactive waste disposal. To reduce the excavation waste and ensure the  
92 mineralogical compatibility, a mixture of crushed COx claystone and bentonite in the form of pre-  
93 compacted blocks has been proposed as a possible sealing material by the French National  
94 Radioactive Waste Management Agency (Andra) (Wang et al., 2014; Zeng et al., 2019; Middelhoff,  
95 2020). Wang et al. (2014) carried out a series of constant-volume swelling pressure tests on  
96 compacted MX80 bentonite/COx claystone mixture with a bentonite fraction of 70% (dry density of  
97 1.7 Mg/m<sup>3</sup>) and observed a slight decrease of swelling pressure with the synthetic site solution after  
98 700 days of hydration. Cuisinier et al. (2009) studied the microstructure of compacted COx claystone  
99 at a dry density of 1.61 Mg/m<sup>3</sup> hydrated with portlandite-saturated solution for 1 year using mercury  
100 intrusion porosimetry (MIP) and scanning electron microscopy (SEM) techniques and found an  
101 increase of macro-pore void ratio after the fluid circulation. By contrast, Middelhoff (2020) indicated  
102 that the swelling pressure after about 7 days and the hydraulic conductivity after 1 year were hardly  
103 affected by the site solution and cement solution for the compacted MX80 bentonite/COx claystone  
104 mixture (a proportion of 30/70 in dry mass) at a dry density of 1.72 Mg/m<sup>3</sup>. Up to now, there was no  
105 consensus regarding the effects of site solution and cement solution on the hydro-mechanical  
106 behaviour of bentonite/claystone mixture. Additionally, to the authors' knowledge, the responses of  
107 the mixtures of COx claystone and different bentonites upon hydration with site solution and cement  
108 solution have not been systematically studied.

109 In this study, a series of infiltration tests using deionised water, synthetic site solution and cement  
110 solution were performed on compacted mixtures of COx claystone and Na<sup>+</sup> MX80 and Ca<sup>2+</sup> Sardinia  
111 bentonites at different dry densities. The effects of synthetic site solution and cement solution on the

112 swelling pressure and hydraulic conductivity were analysed. Moreover, the microstructure features  
113 before and after hydration were also determined using MIP technique, enabling the interpretation of  
114 the physico-chemical interaction between different bentonite/claystone mixtures and different fluids  
115 involved.

## 116 **2. Materials and methods**

### 117 *2.1. Materials*

118 According to the preliminary results of [Zeng et al. \(2020a\)](#), the bentonite/claystone mixtures with a  
119 proportion of 30/70 in dry mass were used in this study to fulfill the requirements of Andra in terms  
120 of swelling pressure and hydraulic conductivity with a dry density of 1.72-1.82 Mg/m<sup>3</sup>. To investigate  
121 the effect of bentonite type, two bentonites, MX80 and Sardinia bentonites, were considered. The  
122 MX80 bentonite is a Na-bentonite, extracted from Wyoming in the United States while the Sardinia  
123 bentonite is a Ca-bentonite, taken from Monte Furros, Italy. The main physical and chemical  
124 characteristics of the two bentonites are presented in [Table 1](#). The COx claystone was sampled from  
125 the Andra Underground Research Laboratory (URL) in Bure. It is composed of 40–45% interstratified  
126 illite/smectite, 30% carbonates, and 25%–30% quartz and feldspar. The COx claystone is  
127 characterized by a liquid limit of 41%, a plastic limit of 24% and a specific gravity of 2.70. Prior to  
128 testing, the bentonites and claystone were crushed to have a maximum grain size of 2.0 mm. The  
129 grain size distributions of the bentonites and claystone determined by dry sieving are presented in [Fig.](#)  
130 [1](#). The mean grain diameters ( $D_{50}$ ) of the MX80 bentonite, Sardinia bentonite, COx claystone are 0.55,  
131 1.06 and 0.58 mm, respectively.

132 Synthetic site solution and cement solution were used for the infiltration tests. These solutions  
133 respectively represent the site water at a depth of 490 m and the alkaline solution after the degradation

134 of concrete, and were prepared according to the recipe provided by Andra (Table 2). The chemical  
135 compositions of the solutions are summarized in Table 3. The pH values of the two solutions were  
136 measured, equal to 8.6 and 12.5, respectively. In addition, deionised water was also employed as a  
137 reference case.

## 138 2.2. Experimental methods

139 The bentonite and claystone powders with a proportion of 30/70 in dry mass were first mixed for  
140 more than 10 min. The gravimetric water contents of the MX80 bentonite, Sardinia bentonite and  
141 claystone under laboratory conditions (a relative humidity of about 60%) were determined by oven-  
142 drying at 105-°C for 24 h, equal to 11.4, 16.0 and 6.1%, respectively. Accordingly, the respective  
143 water contents of the MX80 bentonite/claystone and Sardinia bentonite/claystone mixtures were 7.7  
144 and 9.1%, respectively. Based on the target dry density, a pre-determined amount of mixtures were  
145 poured into a rigid steel ring with an inner diameter of 50 mm and statically compacted at a constant  
146 displacement rate of 0.05 mm/min. The final height of compacted specimens was 10 mm. For the  
147 MX80 bentonite/claystone mixture, nine specimens with dry densities of 1.6, 1.8 and 2.0 Mg/m<sup>3</sup> were  
148 compacted, three for each dry density (Table 4); for the Sardinia bentonite/claystone mixture, six  
149 specimens with dry densities of 1.6 and 1.8 Mg/m<sup>3</sup> were prepared. Afterwards, the compacted  
150 specimens were transferred into a testing cell with an inner diameter of 50 mm (Fig. 2) and placed  
151 between two porous stones and filter papers. A circular cap with a diameter of 50 mm was placed at  
152 the top of the specimens and then blocked using a screw to prevent axial swelling. After that,  
153 deionised water, synthetic site solution or cement solution was injected into the specimens from the  
154 bottom of the cell under a water head of about 1 m and the axial swelling force was monitored using  
155 a force transducer installed under the testing cell. For all the specimens, the hydration lasted for 90



156 days. All the tests were performed at constant ambient temperature ( $20 \pm 1$  °C).

157 At the end of the swelling pressure tests, the solution injection was continued for more than 24  
158 h using a pressure/volume controller under constant pressures. To minimise the disturbance of the  
159 microstructure and avoid hydraulic fracturing, the water injection pressure was lower than 1/10 of the  
160 final swelling pressure. For the MX80 bentonite/claystone mixtures at 1.6, 1.8 and 2.0 Mg/m<sup>3</sup> dry  
161 densities, the injection pressures were respectively 0.04, 0.10 and 0.20 MPa, while those for the  
162 Sardinia bentonite/claystone mixtures at 1.6 and 1.8 Mg/m<sup>3</sup> dry densities were 0.03 and 0.10 MPa,  
163 respectively. During the solution injection, the solution volume injected into the specimens was  
164 recorded. After stabilization of the flow rate, the specimens were regarded as saturated and the  
165 saturated hydraulic conductivity was calculated based on the Darcy's law:

$$166 \quad k = \frac{q}{iA} \quad (1)$$

167 where  $q$  is the flow rate (m<sup>3</sup>/s);  $i$  is the hydraulic gradient;  $A$  is the cross-section (m<sup>2</sup>).

168 After the infiltration tests, the specimens were taken out of the cell for microstructure  
169 observation. The specimens were first cut into small pieces (about 1 cm<sup>3</sup> in volume). Afterwards, the  
170 pieces were immersed in slush nitrogen (-210°C) obtained by previously submitting it to vacuum for  
171 instantaneous freezing, and then lyophilized in a vacuumed chamber (Delage et al., 1996). For the  
172 MIP test, the freeze-dried pieces were put in a low pressure chamber with a working pressure from  
173 3.6 to 2000 kPa and then a high pressure chamber with a working pressure from 0.2 to 228 MPa,  
174 enabling identification of pore entrance diameter 350 to 0.006 μm (the diameter at the narrowest  
175 position).

### 176 **3. Experimental results**

#### 177 *3.1. Swelling pressure*

178 The evolutions of swelling pressure for different dry densities are depicted in Fig. 3. On the whole,  
179 the curves can be divided into two stages: a relatively large primary swell (stage I) and a small  
180 secondary swell (stage II). In stage I, the swelling pressure increased quickly. The overall increasing  
181 rate for the MX80 bentonite/claystone mixture hydrated with synthetic site solution and cement  
182 solution was slightly lower than that of the specimens hydrated with deionised water. Moreover, the  
183 synthetic site and cement solutions reduced the elapsed time required to complete stage I. By contrast,  
184 the increasing rate for the Sardinia bentonite/claystone mixture was almost the same regardless of the  
185 permeating solutions. In stage II, for the MX80 bentonite/claystone mixture with large dry densities  
186 (1.8 and 2.0 Mg/m<sup>3</sup>), the swelling pressure tended to stabilization. By contrast, for the MX80  
187 bentonite/claystone mixture at a low dry density (1.6 Mg/m<sup>3</sup>) and the Sardinia bentonite/claystone  
188 mixtures at dry densities of 1.6 and 1.8 Mg/m<sup>3</sup>, the swelling pressure reached a peak, decreased  
189 sharply and then tended to stabilization. The specimens at a low dry density and hydrated with cement  
190 solution exhibited a more remarkable decrease of swelling pressure after reaching a peak. Moreover,  
191 at a given dry density, the decrease of swelling pressure after reaching a peak was more significant  
192 for the Sardinia bentonite/claystone mixture compared to the MX80 bentonite/claystone mixture. The  
193 final swelling pressures of the specimens at various dry densities and hydrated with different solutions  
194 are summarized in Fig. 4. There were reasonable linear relationships between the final swelling  
195 pressure and the dry density for the specimens hydrated with different solutions. In case of high dry  
196 densities (1.8 and 2.0 Mg/m<sup>3</sup>), the final swelling pressure was almost independent of the permeating  
197 solution. In case of low dry density (1.6 Mg/m<sup>3</sup>), the synthetic site solution and cement solution more  
198 or less reduced the swelling pressure. Additionally, from Fig. 4, a slightly lower final swelling  
199 pressure could be observed on the Sardinia bentonite/claystone mixture compared to the MX80

200 bentonite/claystone mixture, especially in the case of low dry density of  $1.6 \text{ Mg/m}^3$ .

### 201 *3.2. Saturated hydraulic conductivity*

202 At the end of swelling pressures tests, the saturated hydraulic conductivities were determined for  
203 various dry densities and the results are presented in Fig. 5. For the specimens hydrated with a given  
204 solution, the hydraulic conductivity decreased with the increase of dry density. The synthetic site  
205 solution and cement solution increased the hydraulic conductivity for both the MX80  
206 bentonite/claystone and the Sardinia bentonite/claystone mixtures. This is consistent with the results  
207 of Villar (2006) and Chen et al. (2016) while studying the effects of saline and alkaline solutions on  
208 the hydraulic conductivity of MX80 bentonite/granite mixture (a proportion of 30/70 in dry mass)  
209 and GMZ bentonite, respectively. Additionally, larger increases of hydraulic conductivity due to the  
210 water chemistry were observed on the specimens hydrated with cement solution. The lower the dry  
211 density of specimens, the more significant the influences of synthetic site solution and cement  
212 solution on the saturated hydraulic conductivity. In addition, compared to the MX80  
213 bentonite/claystone mixture, the compacted Sardinia bentonite/claystone mixture exhibited a larger  
214 hydraulic conductivity at the same dry density.

### 215 *3.3. Microstructure observation*

216 Figs. 6 and 7 illustrate the pore size distributions of the specimens before and after hydration. From  
217 the cumulative curves (Figs. 6a and 7a), it could be observed that the final value of intruded mercury  
218 void ratio was lower than the global void ratio. This difference indicated the existence of a large  
219 amount of porosity which was inaccessible with the maximum pressure applied in the MIP tests. From  
220 the density curves (Figs. 6b and 7b), the as-compacted specimens exhibited a typical bimodal porosity,  
221 allowing two main pore populations to be defined: a population of intra-aggregate pores (small pores)

222 with a mean pore diameter of 0.02  $\mu\text{m}$  and a population of inter-aggregate pores (large pores) with  
223 a mean pore diameter of 5  $\mu\text{m}$ . This is consistent with the observation of Delage et al. (1996; 2006)  
224 who found that the soils compacted dry of optimum could be described by a typical aggregate  
225 microstructure. After hydration, the mean pore diameter of large pores increased to about 25  $\mu\text{m}$ . By  
226 contrast, the mean pore diameter of small pores remained unchanged but the peak value decreased  
227 remarkably. For the specimens at low dry densities (1.6 and 1.8  $\text{Mg/m}^3$ ), a new pore population with  
228 a mean diameter of 0.2-0.5  $\mu\text{m}$  (defined as medium pores) appeared between the large and small  
229 pores. Following the suggestion of [Bian et al. \(2019\)](#) and [Zeng et al. \(2020b\)](#), the respective delimiting  
230 diameters between the large and medium pores and between the medium and small pore were taken  
231 as 2 and 0.04  $\mu\text{m}$ , respectively. To further investigate the effects of synthetic site solution and cement  
232 solution on the pore structure of bentonite/claystone mixtures, the void ratios of four populations were  
233 determined based on the delimiting values and the cumulative curves. The results are summarized in  
234 [Figs. 8 and 9](#). It can be clearly observed that the synthetic site solution and cement solution increased  
235 the larger-pore and small-pore void ratios and decreased the medium-pore void ratio. Similar  
236 phenomenon was observed by [Wang et al. \(2014\)](#) and [Liu et al. \(2020\)](#) while investigating the effect  
237 of saline solution on the pores structure of compacted MX80 bentonite/claystone (70/30 in dry mass)  
238 and the effect of NaOH solution on the pore structure of compacted GMZ bentonite, respectively.  
239 Moreover, the cement solution had a more remarkable influence on the pore structure changes than  
240 the synthetic site solution. Comparison of the pore structures of two bentonite/claystone mixtures  
241 shows that at a given dry density, the Sardinia bentonite/claystone mixture exhibited a lower large-  
242 pore void ratio and larger medium-pore and small-pore void ratios than the MX80 bentonite/claystone  
243 mixture.

244 **4. Interpretation and discussion**

245 *4.1. Effects of synthetic site solution and cement solution on hydro-mechanical behaviour*

246 When the clay minerals in the bentonite/claystone mixtures were wetted with water, their hydration  
247 could be described by crystalline and osmotic swelling processes (Mitchell, 1993). At a low relative  
248 humidity, crystalline hydration is the predominant mechanism with water molecules being  
249 progressively adsorbed on the clay surfaces layer by layer, leading to an increase of the intra-  
250 aggregate pore volume. Meanwhile, the swollen aggregates invaded the inter-aggregate pores and  
251 reduced the inter-aggregate-pore volume. Obviously, this process depended on the global dry density.  
252 The lower the dry density, the more significant the swelling of aggregates. With the transformation  
253 of intra-aggregate and inter-aggregate pores to the medium-pore population, a remarkable increase of  
254 medium-pore void ratio occurred (Figs. 8 and 9). During the crystalline swelling process, the clay  
255 particles would be divided into smaller ones and fissure-like 2-dimensional (2-D) pores with a mean  
256 pore size of 20  $\mu\text{m}$  appeared. The variation of large-pore volume after hydration was due to the  
257 combined effects of the invasion of swollen clay particles and the appearance of 2-D pores. As the  
258 suction decreased down to 4 MPa, the crystalline swelling reached a nearly steady state and a  
259 significant amount of water would be stored in inter-particle pores (Saiyouri et al., 2000). Due to the  
260 difference of cation concentration between the pore water and the clay particle surface, diffuse double  
261 layer would develop on a parallel assembly of clay particles (Liu, 2013) and osmotic swelling took  
262 place.

263 Generally, the maximum number of water molecule layers intercalated between the unit layers  
264 depends on the clay type and the available cations in the permeating water (Marcial et al., 2002; Liu,  
265 2013). If the cation in the pore water is characterized by a high replacing power, cation exchanges  
266 can take place and the clay type will change. A typically competitive order that has been commonly

267 admitted is:  $\text{Na}^+ < \text{K}^+ < \text{Mg}^{2+} < \text{Ca}^{2+}$  (Mitchell, 1993; Mata, 2003; Sun et al., 2018). As shown in Table  
268 3, the synthetic site solution and cement solution used in this study contain a certain amount of  $\text{K}^+$ ,  
269  $\text{Mg}^{2+}$  or  $\text{Ca}^{2+}$  cations. According to the X-Ray diffractometry (XRD) results on GMZ bentonite by  
270 Sun et al. (2019), Na-montmorillonite would be transformed to Mg/Ca- dominant montmorillonite  
271 and Ca- dominant montmorillonite after the interaction with synthetic site solution and cement  
272 solution, respectively. During the crystalline swelling process, the water adsorption capacity of Na-  
273 montmorillonite was larger than that for Ca-montmorillonite (Liu, 2013; Du et al., 2020; Yotsuji et  
274 al., 2021). Therefore, this transformation of Na-montmorillonite would decrease the swelling capacity  
275 of the montmorillonite. Additionally, as the cation concentration of pore water increased, the repulsive  
276 force between the diffuse double layer and the thickness of the diffuse double layer decreased (Yong  
277 and Warkentin, 1975; Mitchell, 1976). Consequently, a lower swelling pressure was observed for the  
278 specimens hydrated with synthetic site solution (Fig. 4). At a given global void ratio, this decreased  
279 distance between clay particles leads to an increase of the large-pore void ratio. As a result, a larger  
280 hydraulic conductivity was observed for the specimens hydrated with synthetic site solution (Fig. 5)  
281 (Cuisinier et al., 2011).

282 Apart from the cations, the hydroxide in the cement solution can also greatly influence the hydro-  
283 mechanical behaviour of the bentonite/claystone mixtures. Upon contact with hydroxide, the  
284 montmorillonite might be dissolved, forming secondary minerals with much lower swelling capacity,  
285 such as analcime and calcium hydrated silicates gels (Sun et al., 2018; 2019). As result, the swelling  
286 capacity was reduced (Fig. 4). The produced calcium hydrated silicates gels were characterized by a  
287 large amount of small pores (Wang et al., 2017), leading to an increase of small-pore void ratio of the  
288 specimens hydrated with cement solution (Figs. 8 and 9) (Sun et al., 2019; Liu et al., 2020). By

289 contrast, the dissolution of montmorillonite would lead to an increase of the large-pore volume (Chen  
290 et al., 2016). Compared to the specimens hydrated with synthetic site solution, the specimens could  
291 be affected by the hydroxide in addition to the cations. Thus, the specimens hydrated with cement  
292 solution exhibited a larger large-pore volume and a higher hydraulic conductivity compared with  
293 those hydrated with synthetic site solution (Fig. 5).

#### 294 *4.2. Effects of bentonite type and dry density on hydro-mechanical behaviour*

295 Basically, the synthetic site solution and cement solution can influence the hydro-mechanical  
296 behaviour of bentonite-based materials mainly by means of cation exchanges, osmotic effect and  
297 montmorillonite dissolution. After the MX80 bentonite/claystone and Sardinia bentonite/claystone  
298 mixtures were in contact with synthetic site solution, cation exchanges could occur in the MX80  
299 bentonite/claystone mixture, with the transformation of some Na-montmorillonite to Mg-, and Ca-  
300 montmorillonite, while the Sardinia bentonite/claystone mixture would be immune to the cation  
301 exchange. This would reduce the swelling capacity of MX80 bentonite/claystone mixture. Thus, a  
302 slightly smaller increasing rate of swelling pressure for this mixture in stage I for MX80  
303 bentonite/claystone mixture, as compared to that hydrated with deionised water (Fig. 3a and b).  
304 Thanks to the higher permeability, the interaction between clay minerals and solution became more  
305 intensive for the MX80 bentonite/claystone mixture at a lower dry density upon the hydration with  
306 the synthetic site solution. By contrast, the cations in the synthetic site solution would exert an  
307 influence on the osmotic swelling of both the MX80 bentonite/claystone and the Sardinia  
308 bentonite/claystone mixtures at low dry densities. In general, the clay particles of Na-montmorillonite  
309 after crystalline swelling are thinner than those of Ca-montmorillonite (Saiyouri et al., 2004). It was  
310 reported that the clay particles are composed of 2–20 (and even more) aligned unit layers in Ca-

311 montmorillonite, but only of 1–5 unit layers in Na-montmorillonite (Liu, 2013). Therefore, during the  
312 osmotic swelling process, few double layer development could be expected in the Ca-montmorillonite,  
313 as compared to the Na-montmorillonite. The weaker osmotic swelling of Sardinia bentonite/claystone  
314 mixture could not compensate the reduction of swelling pressure induced by the collapse of aggregate  
315 structure. Thus, a more significant decrease was observed in stage II at low dry densities (Fig. 3a and  
316 b). Additionally, the cations in the synthetic site solution could reduce the repulsive force and the  
317 thickness of diffuse double layer, resulting in a lower swelling pressure and a higher hydraulic  
318 conductivity for the specimens hydrated with synthetic site solution (Figs. 4 and 5). For the specimens  
319 at high dry densities, the low amount of inter-particle water was insufficient to form the diffuse double  
320 layer (Pusch and Yong, 2006) and the effect of cations on the swelling pressure and hydraulic  
321 conductivity of the highly compacted MX80 bentonite/claystone and Sardinia bentonite/claystone  
322 mixtures was less significant.

## 323 **5. Conclusions**

324 The effects of synthetic site solution and cement solution on the hydro-mechanical behaviour of  
325 compacted Na<sup>+</sup> MX80 bentonite/claystone and Ca<sup>2+</sup> Sardinia bentonite/claystone mixtures were  
326 investigated by carrying out a series of swelling pressure and hydraulic conductivity tests together  
327 with microstructure observation. The results obtained allow the following conclusions to be drawn:

328 For the MX80 bentonite/claystone mixture, the cations in synthetic site solution could transform  
329 the Na-montmorillonite to multi-cation dominant montmorillonite, reducing the swelling capacity  
330 of the mixture. By contrast, for the Sardinia bentonite/claystone mixture, the cations in synthetic site  
331 solution did not significantly change the Ca-montmorillonite during the crystalline swelling. Thus,  
332 an insignificant effect was identified on the swelling pressure.



333 During the osmotic swelling, the presence of cations reduced the repulsive force by diffuse  
334 double layer and thus swelling pressure for both the MX80 bentonite/claystone and Sardinia  
335 bentonite/claystone mixtures. Additionally, a smaller thickness of diffuse double layer and a greater  
336 large-pore volume could be expected for the specimens hydrated with synthetic site solution. Thus, a  
337 slightly higher saturated hydraulic conductivity was obtained in that case, as compared to the case of  
338 deionised water. Furthermore, a more significant influence of synthetic site solution was observed on  
339 the specimens at a lower dry density thanks to the well-developed diffuse double layer.

340 In addition to the cations, the hydroxide also reduced the swelling pressure of both the MX80  
341 bentonite/claystone and Sardinia bentonite/claystone mixtures. The hydroxide led to a slight increase  
342 of large-pore volume and thus an increase of saturated hydraulic conductivity. Moreover, the lower  
343 the dry density, the more intensive the interaction between the montmorillonite and hydroxide and  
344 the more significant the changes in swelling pressure and hydraulic conductivity, due to the higher  
345 permeability.

346 Over the experiment time scale of 90 days, only a slight deterioration of sealing performance of  
347 the bentonite/claystone mixture was identified due to the low cation concentration and the low ionic  
348 strength of permeating solutions. The effect of water chemistry in the very long-term lifespan of the  
349 underground radioactive waste repository needs to be investigated in further work.

350

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509 Fig. 9 Changes in inaccessible-pore, small-pore, medium-pore and large-pore void ratios for the  
510 compacted Sardinia bentonite/claystone mixtures. (a) 1.6 Mg/m<sup>3</sup> and (b) 1.8 Mg/m<sup>3</sup>



511 **Table 1** Physical and chemical properties of MX80 and Sardinia bentonites

Soil property	MX80	Sardinia
Specific gravity	2.76	2.54 <sup>b</sup>
Consistency limit		
Liquid limit (%)	494	143 <sup>b</sup>
Plastic limit (%)	46	69 <sup>b</sup>
Plasticity index (%)	448	74 <sup>b</sup>
Cation exchange capacity (CEC) (meq/100 g)	80 <sup>a</sup>	65 <sup>c</sup>
Main minerals	Montmorillonite (86%) Quartz (7%)	Montmorillonite (60-90%) <sup>d</sup> Illite (10-14%) <sup>d</sup>

512 a Karnland et al. (2006)

513 b Vitale et al. (2016)

514 c Cara et al. (2000)

515 d Pusch and Karnland (1988)

516

**Table 2** Recipe for the synthetic site solution and cement solution preparation

Content (g/L)	NaCl	NaHCO <sub>3</sub>	KCl	CaSO <sub>4</sub> •2H <sub>2</sub> O	MgSO <sub>4</sub> •7H <sub>2</sub> O	CaCl <sub>2</sub> •2H <sub>2</sub> O	Na <sub>2</sub> SO <sub>4</sub>	Ca(OH) <sub>2</sub>
Site solution	1.950	0.130	0.035	0.630	1.020	0.080	0.700	-
Cement solution	1.286	-	0.596	-	-	-	-	1.408

517

518 **Table 3** Composition of the synthetic site solution and cement solution

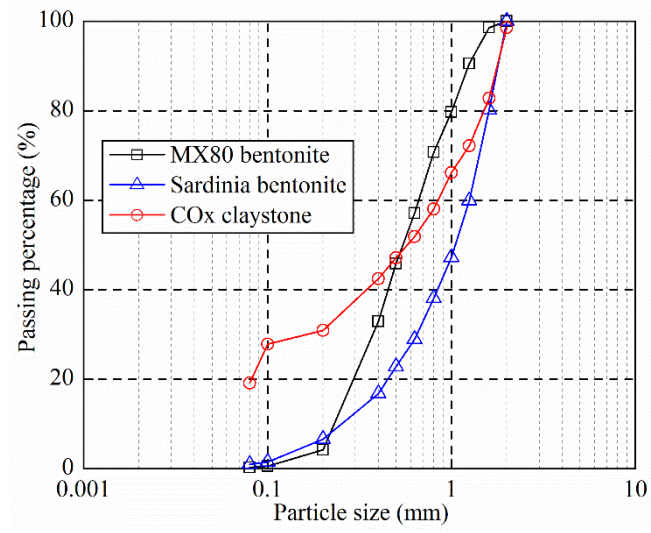
Compound (mmol/L)	Na <sup>+</sup>	K <sup>+</sup>	Ca <sup>2+</sup>	Mg <sup>2+</sup>	Cl <sup>-</sup>	SO <sub>4</sub> <sup>2-</sup>	CO <sub>3</sub> <sup>2-</sup>
Site solution	44.6	0.47	4.2	4.1	34.8	12.7	1.58
Cement solution	22.2	8.05	19	-	30.2	-	-

519

**Table 4** Experimental programme

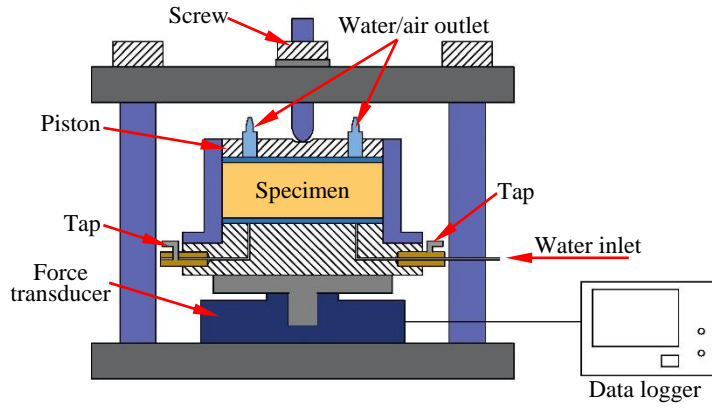
Test No.	Bentonite type	Dry density (Mg/m <sup>3</sup> )	Injected water	MIP
T01	MX80	1.6	Deionised water	✓
T02	MX80	1.6	Site solution	✓
T03	MX80	1.6	Cement solution	✓
T04	MX80	1.8	Deionised water	✓
T05	MX80	1.8	Site solution	✓
T06	MX80	1.8	Cement solution	✓
T04	MX80	2.0	Deionised water	✓
T05	MX80	2.0	Site solution	✓
T06	MX80	2.0	Cement solution	✓
T04	Sardinia	1.6	Deionised water	✓
T05	Sardinia	1.6	Site solution	✓
T06	Sardinia	1.6	Cement solution	✓
T04	Sardinia	1.8	Deionised water	✓
T05	Sardinia	1.8	Site solution	✓
T06	Sardinia	1.8	Cement solution	✓

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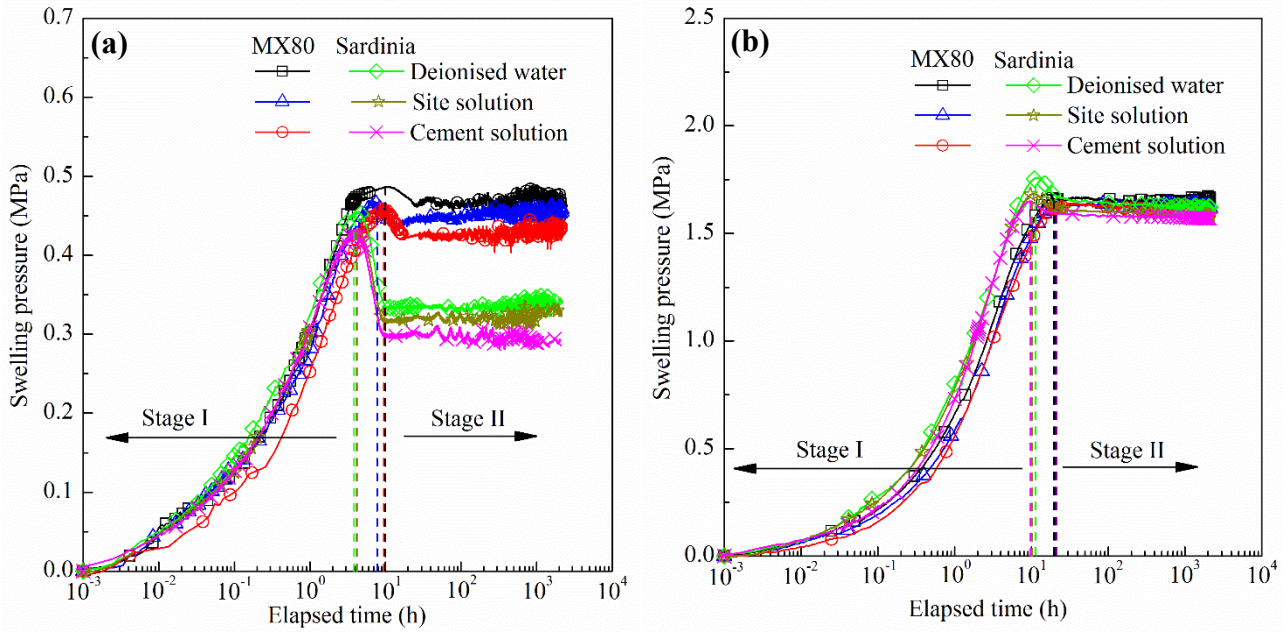
**Fig. 1** Grain size distribution of MX80 and Sardinia bentonites and crushed COx claystone

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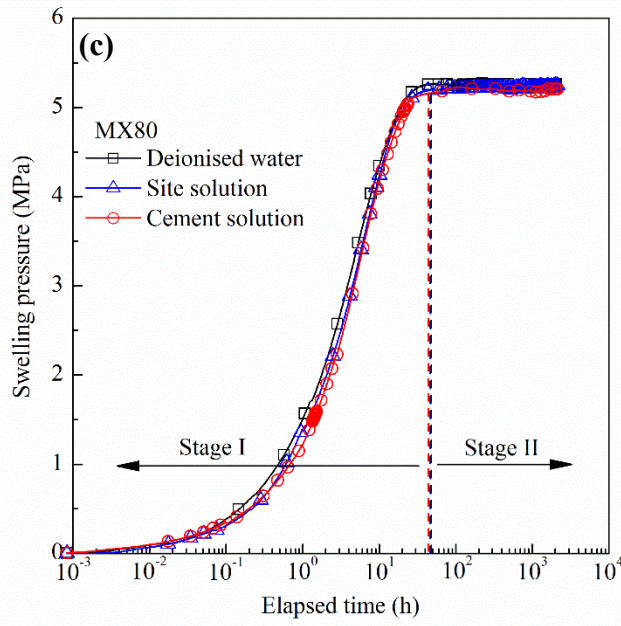
**Fig. 2** Schematic diagram of the constant-volume cell for swelling pressure and hydraulic conductivity tests

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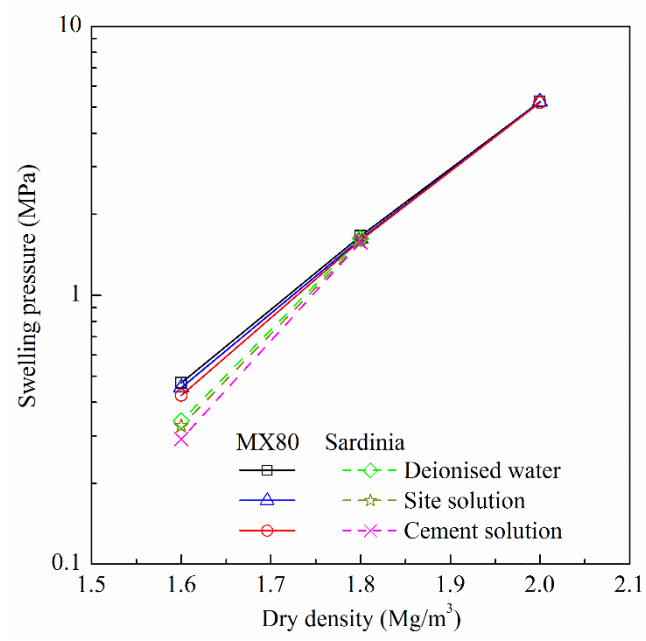
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**Fig. 3** Evolution of axial swelling pressure of specimens at different dry densities. (a) 1.6 Mg/m<sup>3</sup>, (b) 1.8 Mg/m<sup>3</sup> and (c) 2.0 Mg/m<sup>3</sup>

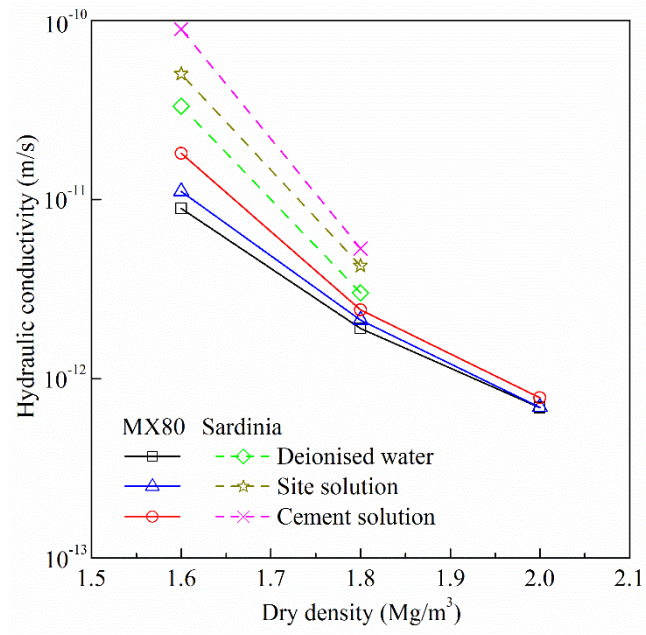
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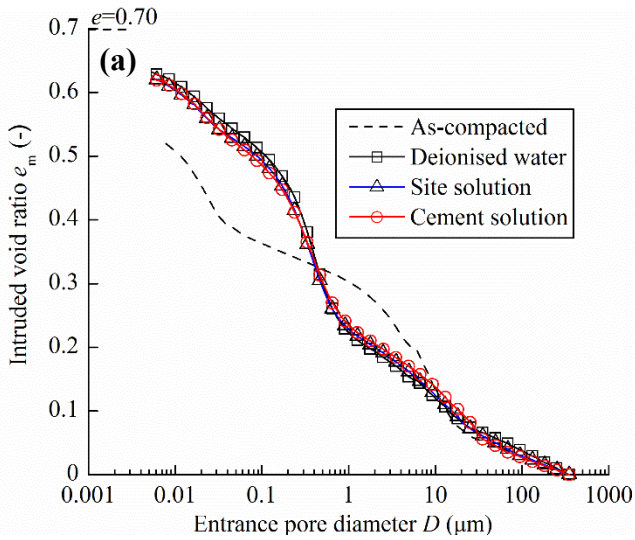
**Fig. 4** Final swelling pressure versus dry density



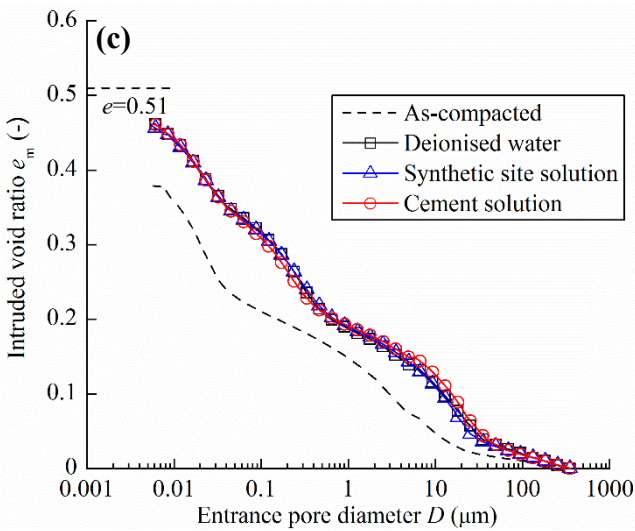
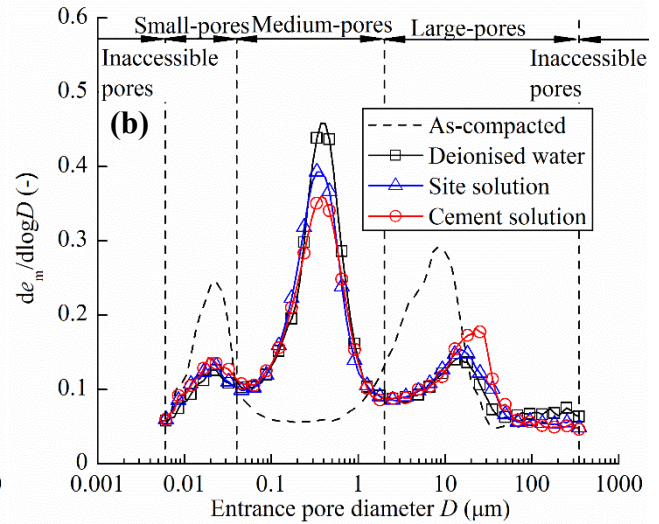
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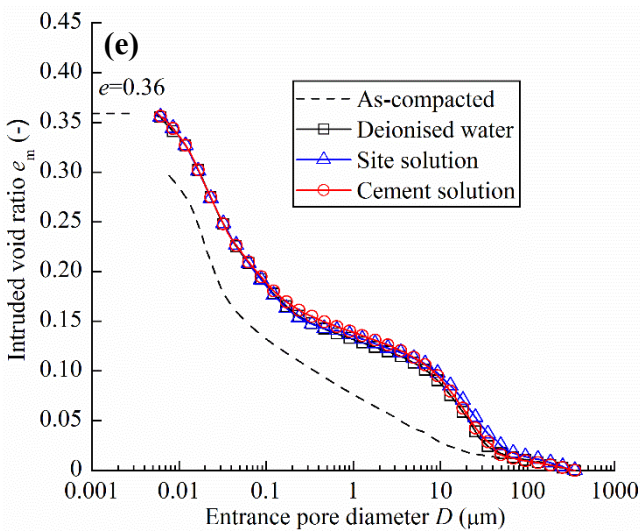
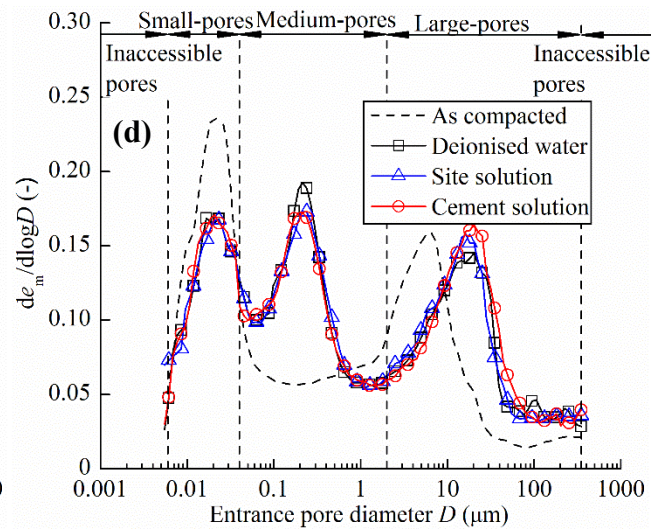
**Fig. 5** Hydraulic conductivity versus dry density



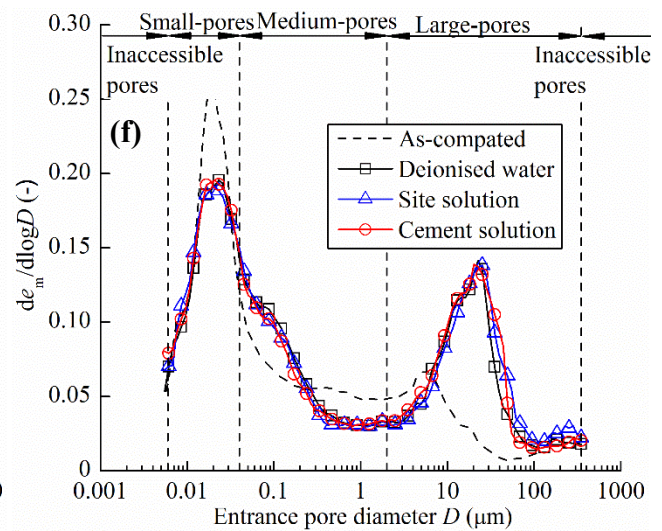
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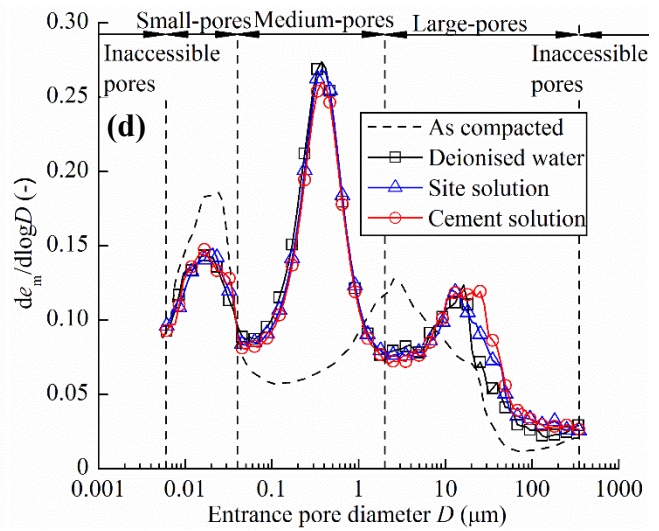
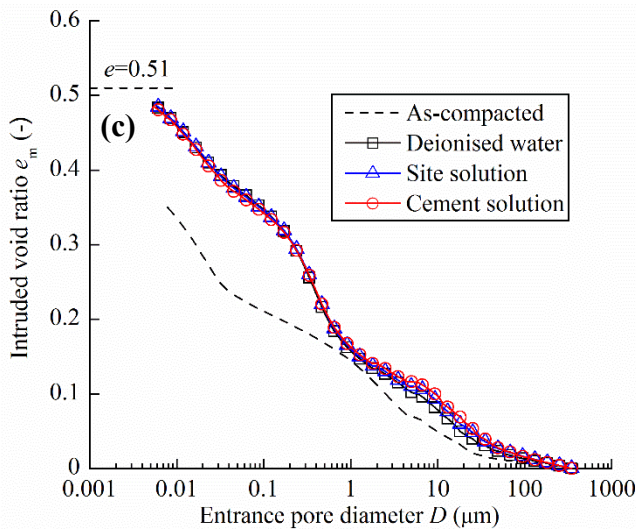
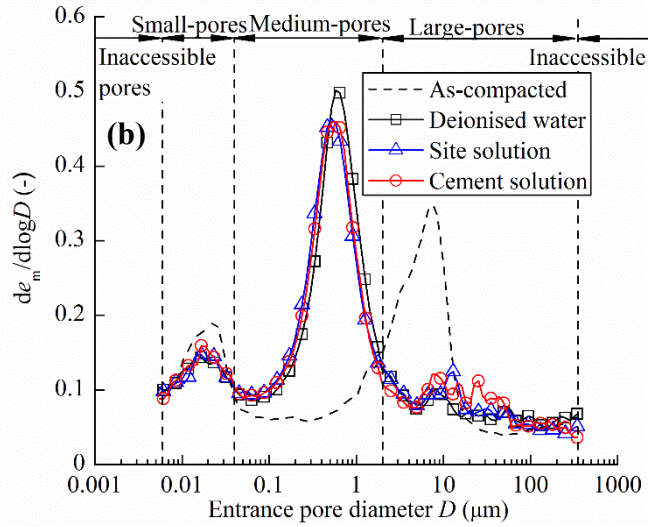
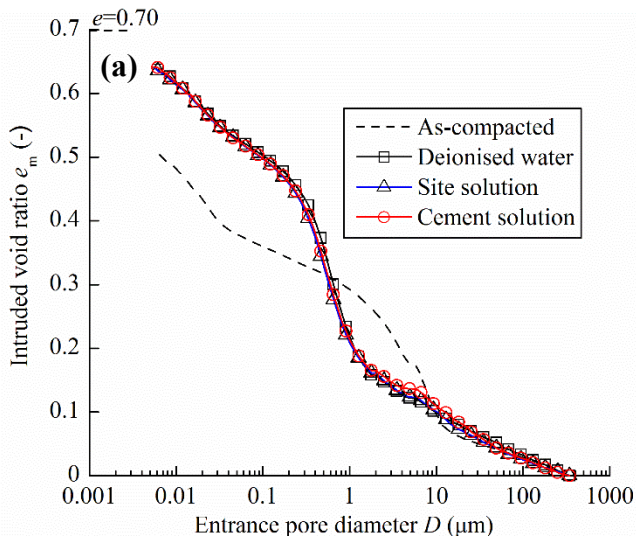
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**Fig. 6** Pore size distribution of MX80 bentonite/claystone mixtures: (a) cumulative curves of specimens at a dry density of 1.6 Mg/m<sup>3</sup>, (b) density function curves of specimens at a dry density of 1.6 Mg/m<sup>3</sup>, (c) cumulative curves of specimens at a dry density of 1.8 Mg/m<sup>3</sup>, (d) density function curves of specimens at a dry density of 1.8 Mg/m<sup>3</sup>, (e) cumulative curves of specimens at a dry density of 2.0 Mg/m<sup>3</sup> and (f) density function curves of specimens at a dry density of 2.0 Mg/m<sup>3</sup>



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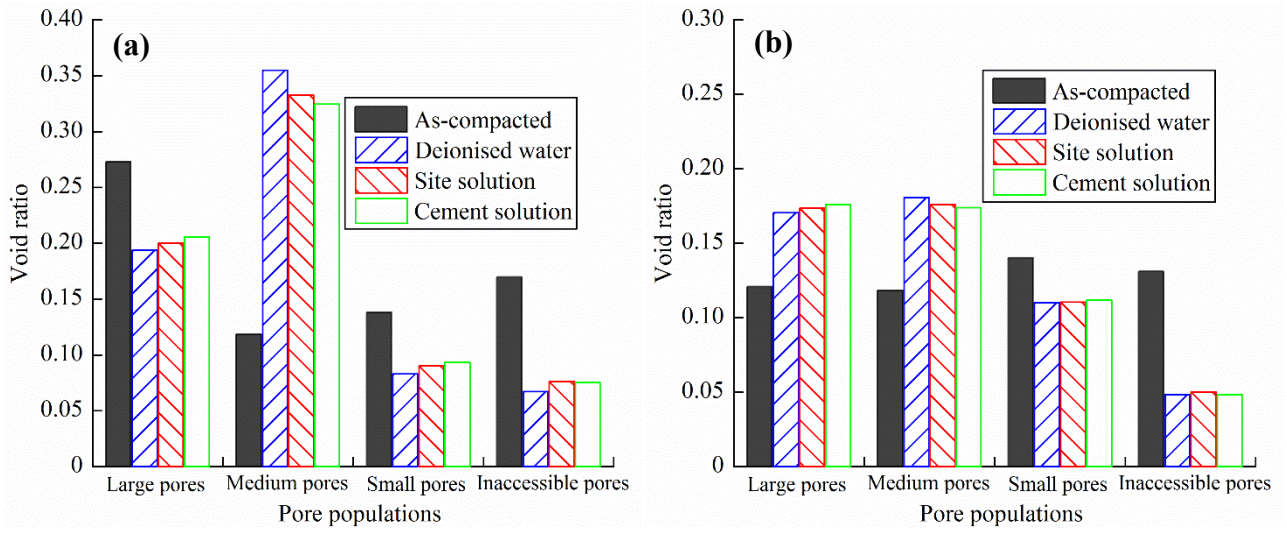
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**Fig. 7** Pore size distribution of Sardinia bentonite/claystone mixtures: (a) cumulative curves of specimens at a dry density of 1.6 Mg/m<sup>3</sup>, (b) density function curves of specimens at a dry density of 1.6 Mg/m<sup>3</sup>, (c) cumulative curves of specimens at a dry density of 1.8 Mg/m<sup>3</sup> and (d) density function curves of specimens at a dry density of 1.8 Mg/m<sup>3</sup>



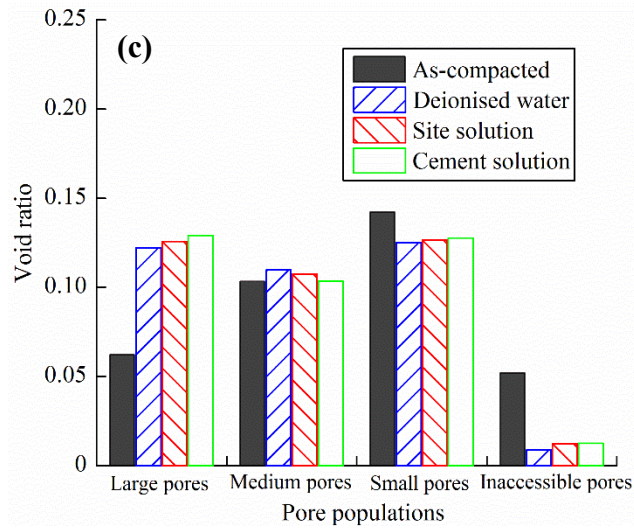
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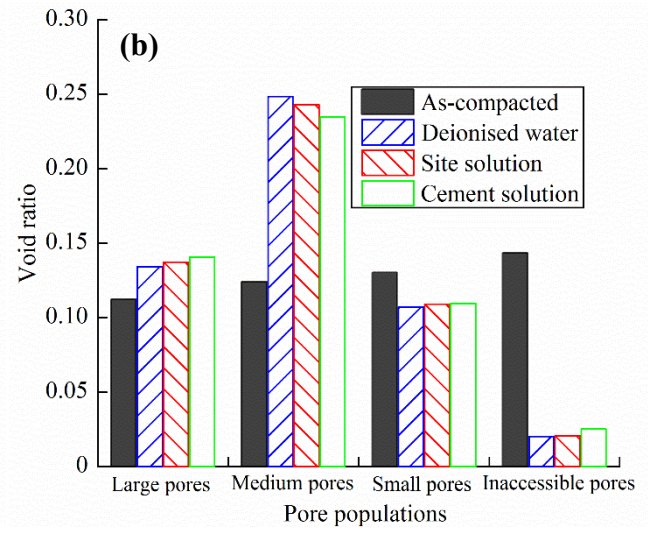
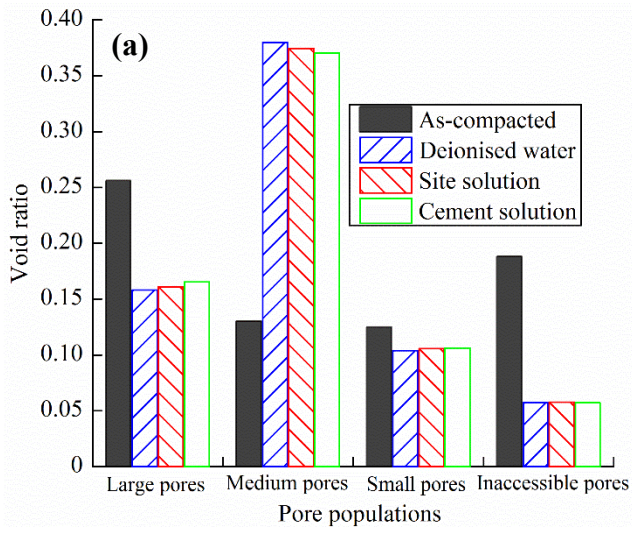
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**Fig. 8** Changes in inaccessible-pore, small-pore, medium-pore and large-pore void ratios for the compacted MX80 bentonite/claystone mixtures. (a) 1.6 Mg/m<sup>3</sup>, (b) 1.8 Mg/m<sup>3</sup> and (c) 2.0 Mg/m<sup>3</sup>



**Fig. 9** Changes in inaccessible-pore, small-pore, medium-pore and large-pore void ratios for the compacted Sardinia bentonite/claystone mixtures. (a) 1.6 Mg/m<sup>3</sup> and (b) 1.8 Mg/m<sup>3</sup>

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