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1 **Hydro-mechanical path dependency of claystone/ bentonite-mixture**
2 **samples characterized by different initial dry densities**

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8 **Abstract:** In the context of the French Cigéo-project, a mixture composed of 70% processed Callovo-
9 Oxfordian claystone spoil and 30% MX80-bentonite could be a potential backfill material, whose in-
10 stallation aims to stabilize the surrounding rock formation and to limit the propagation of the excavation
11 damaged zone. The backfill material must sustain the overburden pressure, despite it might be exposed
12 to different hydraulic and mechanical paths. The reference concept considers employing conventional
13 compaction techniques, although their employment involves spatial variations in the dry density after
14 compaction. In general, as the initial dry density has a significant impact on the hydro-mechanical be-
15 havior of backfill materials, it is of major importance to relate the variations in the initial dry density to
16 differences in the behavior. This experimental laboratory study aimed to analyze how variations in the
17 initial dry density affects the swelling and compression behavior of the claystone/ bentonite-mixture, in
18 particular in unsaturated state. Further, it evaluated whether those variations affected possible hydro-
19 mechanical path dependences. The experimental program comprised suction-controlled oedometer and
20 constant-volume swelling pressure experiments, in which samples characterized by different initial dry
21 densities were exposed to different hydro-mechanical paths. The analysis of microstructural and water
22 retention characteristics complemented the program. Major results indicated that the magnitude of swell-
23 ing pressure at a given suction depends considerably on the initial dry density, but it is independent of
24 the imposed hydro-mechanical path. Interestingly, the dependency of the yield behavior on the hydro-
25 mechanical path appears to be more pronounced as the initial dry density increases.

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28

29 Keywords: Callovo-Oxfordian claystone, MX80-bentonite, Microstructure, Water retention curve, Hy-
30 dro-mechanical behavior, Hydro-mechanical load path dependency

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36

2 1 Introduction

3 In the context of the French Cigéo-project, different materials are studied as to whether
4 they could be employed to backfill drifts and shafts of the future repository for radioactive
5 waste located at a depth of approximately 500 m in the clay-rich Callovo-Oxfordian sedimentary
6 rock formation, henceforth referred to as Callovo-Oxfordian (COX) claystone formation
7 [5]. The installation of backfill aims to stabilize the surrounding rock formation and to limit the
8 propagation of the excavation damaged zone (EDZ) that arises from the excavation of drifts
9 and shafts and lowers the integrity of the repository system. The backfill must thus exhibit such
10 a swelling pressure and compressibility that it sustains the overburden pressure upon terminating
11 the closure phase. As depicted in Fig. 1, the backfill might undergo different combinations
12 of hydraulic and mechanical paths. The path A – C (– D) describes a possible case of (stepwise)
13 backfill material saturation under constant-volume conditions. It might be followed by loading
14 through the overburden pressure, whose onset occurs once convergence processes cause the
15 partial degradation of the concrete lining. Under laboratory conditions, the path A – C corre-
16 sponds to the path imposed in constant-volume swelling pressure experiments, whereas path A
17 – B – C – D is imposed in oedometer experiments.

18 A potential backfill material is a mixture composed of processed Callovo-Oxfordian clay-
19 stone spoil and MX80-bentonite, whose dominant mineralogical constituents are phyllosilicates
20 (e.g. smectites, illite, illite/ smectite mixed layer minerals) [7, 21]. Since conventional compac-
21 tion techniques are presumably employed to compact it to the maximum dry density at the
22 optimum water content in situ, the material is initially characterized by a degree of saturation
23 greater than 80% and low suctions. The initial dry density determines not only the hydro-me-
24 chanical behavior of smectite-containing materials, but also their microstructural and water re-
25 tention characteristics [e.g. 1]. It is thus of major interest to analyze how variations in the initial

26 dry density affect the behavior of such materials upon imposing different hydraulic and me-
27 chanical paths and their combinations.

28 In general, there is a wealth of studies aiming to analyze the microstructural and water
29 retention characteristics and volume-change behavior of smectite-containing materials, in par-
30 ticular in unsaturated conditions [15, 19, 23, 27, 34, 36].

31 Unsaturated smectite-containing materials are characterized by micro- and macropores,
32 referring to pores between clay aggregates and soil aggregates, respectively. Mechanical and
33 hydraulic loads affect differently those pore populations. Densification reduces only the amount
34 of macropores, whereas the amount of micropores remains stable [e.g. 37]. Hydration affects
35 both pore populations as the amount of micropores increases and the amount of macropores
36 decreases due to the swelling of smectite particles [e.g. 27].

37 The structure determines the water retention characteristics of smectite-containing mate-
38 rials. Densification provokes a shift of the air entry value (AEV) to higher suction values as it
39 reduces the size and shape of macropores. The initial dry density affects those characteristics,
40 only if suction values are lower than few hundred MPa. Below those suction values, dominant
41 capillary phenomena are controlled by macropores [e.g. 15].

42 Among others, Lloret et al. [23] studied the volume-change behavior of FEBEX-bentonite
43 in unsaturated conditions. Their experimental program comprised suction-controlled oedometer
44 and constant-volume swelling pressure experiments under different hydro-mechanical paths.
45 Experimental protocols considered the preparation of samples at the hygroscopic water content.
46 Oedometer experiments were conducted on samples, which were characterized by one initial
47 dry density, whereas samples studied in constant-volume swelling pressure experiments were
48 compacted to different initial dry densities. Oedometer experiments indicated that the material
49 rigidified and the yield stress shifted to higher vertical stresses as the suction was increased.
50 The material response considerably depended on the hydro-mechanical path attributable to a

51 differently evolving microstructure. The imposition of a wetting-loading sequence involves an
52 initial increase in total porosity due to material swelling. Pore-merging accounts for the fact
53 that the mechanical loading affects all pores. A loading-wetting sequence reduces the macro-
54 porosity during mechanical loading and the later collapse of the majority of macropores limits
55 material swelling upon wetting. The former sequence thus yields a greater final total porosity
56 than the latter. There are only a few studies aiming to determine the impact of variations in the
57 initial dry density on the yield behavior of smectite-containing materials upon hydro-mechani-
58 cal loading. Nowamooz and Masrouri [30] studied the saturated and unsaturated compression
59 behavior of silt/ bentonite-mixtures by means of suction-controlled oedometer experiments.
60 Sample preparation comprised their compaction to different dry densities at the optimum water
61 content. At a given suction, the yield stress increases as the initial dry density increases, and, at
62 a given initial dry density, the material rigidifies as the suction increases. There is still little
63 understanding about whether variations in the initial dry density alter the yield behavior of
64 smectite-containing materials upon hydro-mechanical loading, and how they affect possible
65 hydro-mechanical path dependencies.

66 Suction-controlled constant-volume swelling pressure experiments of Lloret et al. [23]
67 showed that the evolution of swelling pressure was characterized by a double peak pattern upon
68 stepwise decreasing suctions. The characteristic pattern evolved, regardless of the initial dry
69 density. The first maximum value was attributed to the initial swelling of smectite particles,
70 which was followed by the partial collapse of soil aggregates indicating the yield stress. The
71 second maximum value ensued as the swelling of particles prevails over the collapse of soil
72 aggregates. Romero [33] conducted similar experiments on Boom-clay samples and hypothe-
73 sized that the yield loci determined in suction-controlled oedometer and constant-volume swell-

74 ing pressure experiments must coincide. However, only a few studies corroborated the hypothesis [e.g. 4]. Further, there are no information about whether variations in the dry density affect
75 the coincidence.

77 Experiment results are commonly interpreted and reproduced by adopting the constitutive
78 models proposed in Alonso et al. [2] (*Barcelona Basic Model (BBM)*) and Alonso et al. [3]
79 (*Barcelona Expansive Model (BExM)*). The latter acknowledges the existence of micro- and
80 macropores and is thus capable to reproduce the characteristic features of smectite-containing
81 materials. As respective experimental studies are scarcely available, only a few constitutive
82 models allow to consider the impact of variations in the initial dry density on the hydro-me-
83 chanical behavior [38, 43].

84 In the context of in situ compacted backfill materials, this study aimed to evaluate the
85 impact of variations in the initial dry density on the response of the claystone/ bentonite-mixture
86 to different hydro-mechanical paths. Moreover, it analyzed whether those variations affect the
87 postulated coincidence of yield loci. The evaluation was accomplished by conducting suction-
88 controlled swelling pressure and oedometer experiments on samples which were compacted to
89 different initial dry densities at the optimum water content. Experiments were complemented
90 by analyzing the microstructural and water retention characteristics of the potential backfill
91 material. Obtained results can be useful to improve existing constitutive models regarding the
92 integration of the impact of the initial dry density.

93 2 Material

94 Following, the section recapitulated the general characteristics of the mixture studied (e.g.
95 physical characteristics, physico-chemical characteristics, compaction characteristics), and de-
96 scribed its microstructural and water retention characteristics afterwards.

97 2.1 General characteristics

98 The mixture studied was composed of 70% processed Callovo-Oxfordian (COX) clay-
99 stone spoil and 30% MX80-bentonite in weight. The latter was limited to 30% in order to reduce
100 mineralogical and physico-chemical incompatibilities between the potential backfill material
101 and the surrounding rock formation. Callovo-Oxfordian claystone spoil arose during the exca-
102 vation of drifts of Andra URL at a depth of – 490 m (Bure, Meuse/ Haute-Marne region,
103 France). The subsequent processing comprised its crushing and sieving to a maximum grain
104 diameter of 2 mm. The processed material was stored in airtight containers afterwards. Hence-
105 forth, processed COX-claystone spoil is referred to as COX_c. At that depth, intact COX-clay-
106 stone contains 27% carbonates (e.g. calcite, dolomite), 46% phyllosilicates (e.g. smectites, il-
107 lites), 24% tectosilicates (e.g. quartz, feldspars), and minor fraction of other mineral phases [7].
108 Likewise, MX80-bentonite (Wyoming, USA) was crushed and sieved to maximum grain diam-
109 eters of 2 mm and filled into air-tight containers. The dominant mineral phases are phyllosili-
110 cates (e.g. smectites), whose fraction is about 84%. The mineralogical composition is completed
111 by tectosilicates (e.g. quartz) [21].

112 The mixture was characterized by an initial water content (w_{ini}), liquid limit (LL), plastic
113 limit (PL) and specific gravity (G_s) of 6.4%, 112.5%, 34.7%, and 2.64, respectively [25]. Its
114 cation exchange capacity (CEC) of 39 meq/100g and specific surface area (SSA) of 32 m²/g
115 were determined by means of the cobalt hexamine method [32] and the BET-method [17], re-
116 spectively. The cobalt hexamine method also revealed that the exchangeable cation species
117 were dominated by Na⁺- and Ca²⁺-ions. An elevated compaction energy increased the maximum
118 dry density ($\rho_{d, max}$) and decreased the optimum water content (w_{opt}) of the mixture [25]. Ac-
119 cordingly, the optimum water content and maximum dry density were 29.0% and 1.45 Mg/m³,
120 respectively, in the case of standard compaction energy employment, and 18.2% and
121 1.72 Mg/m³, respectively, in the case of modified compaction energy employment. The grain

122 size distribution was determined by means of laser diffractometry and dry sieving and was
123 characterized by 10% - and 60% - passing (D_{10}/D_{60}) values of 0.008 mm and 0.8 mm, respec-
124 tively.

125 **2.2 Microstructural characteristics**

126 Mercury intrusion porosimetry (MIP) experiments were conducted on samples initially
127 compacted to dry densities of 1.72 Mg/m³ and 1.44 Mg/m³ at a water content of 18.2% in order
128 to highlight how possible variations in the initial dry density affect the microstructure. The latter
129 value corresponded to a reduction of the initial dry density by about 17.5% with respect to the
130 maximum dry density. Its consideration facilitated to interpret the results of swelling pressure
131 and oedometer experiments presented later. MIP-experiments were conducted by means of an
132 AutoPore IV 9500 (Micromeritics, U.S.A). The MIP-technique bases on the principle of for-
133 cing a non-wetting fluid, e.g. mercury, to enter a porous medium by incrementally increasing
134 the injection pressure. A review of the technique is given in Romero and Simms [35]. Prior to
135 the analyzes, samples were dried by heating them up to 80°C under vacuum conditions. Apart
136 from that, the experiment protocol followed that established by Cuisinier and Laloui [8]. Ap-
137 proaches for data processing and presentation were adopted from Juang and Holtz [20].

138 As depicted in Fig. 2a and Fig. 2c, pore size distribution curves of samples were charac-
139 terized by two distinct peaks, regardless of the initial dry density. They corresponded to micro-
140 and macropore populations. According to Romero et al. [36], the inflection point of pore size
141 distribution curves indicated the transition from micro- to macropores. Pores, whose diameters
142 were smaller and greater than the diameter at the inflection point, are referred to as micro- and
143 macropores, respectively. Their quantities are expressed by the micro-void ratio (e_m) and
144 macro-void ratio (e_M), respectively. The unaccountable void ratio (e_{un}) is defined as the differ-
145 ence between the calculatable total void ratio (e_{tot}) and the macro-void ratio and accounts for
146 the pores, whose diameter was too small or too large to be detected.

147 The transition of macro- to micropores occurred at pore diameters ranging between 4 and
148 5×10^2 nm, regardless of the initial dry density. In all cases, most micropores occurred at a pore
149 diameter of about 2×10^1 nm. In the case of denser samples, the majority of macropores occurred
150 at a pore diameter of about 8×10^4 nm. Interestingly, in the case of looser samples, the increasing
151 pore size distribution curve indicated that the diameter, at which the majority of macropores
152 occurred, appeared to exceed the limit of the device. Densification reduced the macro- and
153 unaccountable void ratio by about 50%, whereas the micro-void ratio remained stable. The ob-
154 servation were in accordance with the literature as micropores are expected to react sensitively
155 to the hydraulic but not to the mechanical path, such as those imposed by means of the static
156 compaction method [16, 35, e.g. 36]. The differences in the unaccountable void ratio might be
157 attributed to pores, whose diameters were in the millimeter range involving the low initial dry
158 density.

159 In general, the microstructure of mixture samples comprised micro- and macropores, like
160 other smectite-containing materials. As expected, the latter pore population was significantly
161 reduced by densification. It must be thus assumed that the water retention characteristics of
162 denser and looser material differ considerably.

163 2.3 Water retention characteristics

164 Samples were characterized by initial dry densities of 1.72 Mg/m^3 and 1.44 Mg/m^3 and a
165 water content of 18.2%. A range between few tens of kPa to some hundreds of MPa were im-
166 posed by combining the osmotic and vapor equilibrium technique. Their individual ranges over-
167 lapped at suctions of about 7 MPa [e.g. 9, 31]. The former technique bases upon the principle
168 that water molecules can go through a semipermeable membrane and move from the pore so-
169 lution to a macro-molecular (polyethylene glycol (PEG)) solution and vice versa. The migration
170 and its direction is driven by a concentration gradient. Suction is functionally related to the
171 concentration of PEG-molecules in solution as their concentration determines the magnitude of

172 the gradient. A review of the technique is given in Delage and Cui [10]. The vapor equilibrium
173 technique adopts Kelvins equation relating suction to the relative humidity in a closed system.
174 The closed system comprises a gas phase and a liquid phase, whose specific solutes control the
175 relative humidity in the adjacent gas phase. In general, saturated salt solutions are employed as
176 a liquid phase. A review of the technique is given in Delage et al. [12] and Delage et al. [13].

177 As depicted in Fig. 3, denser samples were initially characterized by lower void ratios
178 and higher degrees of saturation due to the higher initial dry density. Upon hydration, the void
179 ratio and degree of saturation of looser and denser samples increased comparably. Drying had
180 no considerable consequences on both sample types with regard to their void ratios. It affected
181 the degree of saturation of denser samples more significantly than that of looser samples.

182 The impact of variations in the initial dry density appeared to vanish when imposed suc-
183 tion exceeded values of 200 MPa. This observation was in accordance with the results of mi-
184 crostructural analysis as the vanishing impact of initial dry density indicated the existence of
185 micro- and macropores (Fig. 2). The imposition of a suction higher than 200 MPa involved the
186 fact that residual water was available only in micropores, whose size and shape were insensitive
187 to compaction and varying initial dry densities. According to the different diffuse double layer
188 theories, electrostatic interactions between the water molecules and the clay mineral surfaces
189 account for a complete immobilization of the residual water in micropores [e.g 34].

190 In general, the water retention characteristics of differently dense mixture samples con-
191 firmed the results of microstructural analysis as the vanishing impact of initial dry density at
192 high suctions indicated the existence of micro-and macropores.

193 3 Oedometer and constant-volume swelling pressure experiments

194 The unsaturated compression and swelling behavior were studied by conducting oedom-
195 eter and constant-volume swelling pressure experiments, in which the suction was controlled
196 by means of the osmotic technique. Further, different hydro-mechanical paths were imposed to

197 analyze the impact of variations in the dry density on possible path dependencies. The experi-
198 ment approach considering variations in the initial dry density, the sample preparation, the ex-
199 periment protocols, and the experiment program are described below.

200 **3.1 Approach**

201 Full-scale experiments conducted in situ (e.g. the “Backfill and Plug Test” [18], the “Pro-
202 totype repository” [6]) highlighted the issues involving the employment of conventional com-
203 paction techniques to install smectite-containing backfill materials in situ. Their experiment
204 protocols envisaged compacting backfill materials to the maximum dry density at the optimum
205 water content in a layer-wise manner. Highest swelling pressure and lowest compressibility
206 were expected to be reached by this means [e.g. 26]. Unlike the initial water content, whose
207 value remained stable in the cross section of the drift, the initial dry density decreased by up to
208 20% with respect to the maximum dry density. They attributed this finding to a loss of compac-
209 tion energy ensuing as the handling of the compactor became difficult, in particular close to the
210 drift top and drift walls.

211 In order to portray spatial variations in the initial dry density, samples were compacted to
212 the maximum dry density of 1.72 Mg/m^3 at the optimum water content of 18.2%, which was
213 obtained by means of modified Proctor experiments. They were complemented by samples
214 characterized by dry densities of 1.66 Mg/m^3 , 1.59 Mg/m^3 , and 1.44 Mg/m^3 and a water con-
215 tent of 18.2%. The values corresponded to a reduction of the dry density by 2.5%, 7.5% and
216 17.5%, respectively, with respect to the maximum dry density.

217 **3.2 Sample preparation**

218 The material was prepared at the optimum water content and then filled in an airtight
219 container. Deaired/ demineralized water was added in order to reach the optimum water content
220 [25]. Samples were compacted to the different dry densities at the optimum water content by
221 means of the static compaction method at a controlled deformation rate of 0.1 mm/s. Samples,

222 whose microstructural and water retention characteristics were previously presented, were sim-
223 ilarly prepared. Henceforth, the type indicates the initial dry density of samples. Their initial
224 characteristics are compiled in Table 1.

225 3.3 Suction-controlled oedometer experiments

226 The protocol of suction-controlled oedometer experiments considered samples of all
227 types. The cell employed in suction-controlled oedometer experiments was composed of a pis-
228 ton, mold, and base (e.g. Delage et al. [11] or Nowamooz and Masrouri [29] for further infor-
229 mation about its design). The base was characterized by one inlet, one outlet and a mesh, so
230 solutions could be circulated beneath the mesh. It covered a groove spiraling to the center axis
231 of the cylindrical cell and connecting the inlet and outlet. In combination with a semi-permeable
232 membrane separating the sample from the mesh, the design allowed to adopt the osmotic tech-
233 nique in order to impose suctions. The oedometer allowed to apply vertical stresses of up to
234 3000 kPa. PEG-reservoirs were placed in temperature-controlled baths in order to minimize
235 temperature effects.

236 3.4 Suction-controlled constant-volume swelling pressure experiments

237 Samples of all types were considered in order to study the evolution of swelling pressure
238 upon stepwise hydration. The constant-volume swelling pressure cell proposed by Yigsaw et
239 al. [40] was similarly designed allowing to adopt the osmotic technique. However, a porous
240 disc was employed instead of the mesh, and the piston was substituted for a lid, which contained
241 the load sensor. By eventually screwing the components together, a pre-stress of 50 kPa was
242 imposed.

243 3.5 Hydro-mechanical paths and experimental program

244 The protocol of suction-controlled swelling pressure and oedometer experiments com-
245 prised different hydro-mechanical paths depicted in Fig. 4 and Fig. 5, respectively. Their impo-
246 sition aimed to determine how different paths affect the swelling and compression behavior of

247 the potential backfill material, in particular in unsaturated conditions. Since backfill materials
248 are not expected to undergo drying, hydration referred exclusively to the (stepwise) saturation
249 of the mixture, whose initial suction was about 2700 kPa (Table 1).

250 Hydro-mechanical paths A, B, C, and D differed in the magnitude of the initial hydration
251 step followed by stepwise mechanical loading and unloading. Hydro-mechanical loads were
252 changed when strains stabilized at rates lower than 0.5 % (of the initial sample height) per day.

253 Hydro-mechanical paths E, F, and G differed in the number of stages, in which different
254 suctions were imposed. Imposed suctions were decreased when the swelling pressure stabilized
255 at rates lower than 5 kPa per day.

256 Henceforth, samples are identifiable by means of their sample type and imposed hydro-
257 mechanical paths. The prefix refers to the sample type, whereas the imposed hydro-mechanical
258 path is indicated by the suffix. For instance, sample I - D was characterized by an initial dry
259 density of 1.72 Mg m^{-3} , whose compression behavior was analyzed by adopting hydro-mechan-
260 ical path D (Fig. 4). The experiment program developed is described in Table 2.

261 It was believed that this comprehensive experimental program allowed to evaluate how
262 variations in the initial dry density affect the behavior of a potential backfill material under
263 different hydro-mechanical paths.

264 4 Results

265 Following, the results of suction-controlled oedometer and constant-volume swelling
266 pressure experiments are presented. Since the free-swell potential experiments represented the
267 first stage of oedometer experiments, their results are described in the corresponding section.

268 4.1 Suction-controlled oedometer experiments

269 Oedometer experiments were conducted on type I- to type IV-samples by imposing hy-
270 dro-mechanical paths A to D. They were aimed at studying how the one-dimensional compres-
271 sion behavior of the mixture varies as response to the different initial dry densities of samples

272 and imposed hydro-mechanical paths. Additionally, information about the free-swell potential
273 were provided. Their results are depicted in Fig. 6a to Fig. 6h. For clarification purpose, the
274 presentation of the results obtained while unloading is omitted.

275 Compression curves were characterized by two or three zones, in which the slope of the
276 compression line changed. Hydro-mechanical paths C and D involved two zones, whereas the
277 imposition of hydro-mechanical paths A and B implied the development of three zones consti-
278 tuting s-shaped compression curves. Compression lines converged as net vertical stresses were
279 increased beyond about 1000 kPa. Those characteristics were observed, regardless of the initial
280 dry density of samples. The intersection of compression curves of less dense samples cannot be
281 reasonably explained, yet (compare Fig. 6h). In elastoplastic frameworks [e.g. 2], the first and
282 second zones are referred to as the elastic and plastic domains, which are separated by the yield
283 stress. Deformations in the elastic domain appeared to be little affected by varying initial dry
284 densities, whereas decreasing suctions involved greater elastic deformations. Deformations in
285 the plastic domain became less pronounced as initial dry densities and imposed suctions in-
286 creased. In addition, similarly dense samples exhibited smaller plastic deformations in response
287 to increasing suctions. Among the different sample types, yield stresses appeared to converge
288 as initially imposed suctions were reduced.

289 Oedometer experiments also indicated that the free-swell potential increased as the initial
290 dry density was increased, in particular upon imposing lower initial suctions.

291 In general, the initial dry density affected the one-dimensional compression behavior of
292 mixture samples more considerably when higher suctions were imposed. In saturated state,
293 compression curves appeared to be comparable with regard to their characteristics and shape.
294 The free-swell potential was considerably affected by the initial dry density, in particular in
295 ranges of lower suctions.

296 4.2 Suction-controlled constant-volume swelling pressure experiments

297 Constant-volume swelling pressure cells controlling suction by means of the osmotic
298 technique were employed in order to study the swelling pressure evolution of the mixture under
299 different hydro-mechanical paths. Evolution of swelling pressure recorded are depicted in
300 Fig. 7a and Fig. 7b.

301 In the case of sample II-E, the evolution was not measured due to a malfunction of the
302 load sensor. It still became evident in Fig. 7a that the stabilized swelling pressure at each suction
303 stage increased as the initial dry density increased. The pattern of the curves was generally
304 characterized by a post-peak stabilization at lower or comparable stresses. In the cases of type
305 III- and IV-samples, the decrease of swelling pressure occurred upon imposing a suction smaller
306 than 1000 kPa, whereas, in the case of the type I-sample, there was only a slight reduction and
307 ensued when suctions were smaller than 100 kPa. As highlighted in Fig. 7b, the mixture reacted
308 to the imposition of different hydration paths identically, since the stabilized swelling pressures
309 at the corresponding suction stages differed negligibly.

310 In general, suction-controlled constant-volume swelling pressure experiments revealed
311 that the unsaturated swelling pressure of the mixture depended on the initial dry density rather
312 than on the hydraulic path. Further, the results depicted in Fig. 7b indicated a good repeatability
313 of swelling pressure experiments.

314 5 Discussion

315 The discussion adopted the elastoplastic frameworks proposed in Alonso et al. [2] and
316 Alonso et al. [3]. It analyzed how variations in the initial dry density affect the compression and
317 swelling behavior of mixture samples. The first and second sections are followed by a compar-
318 ison which evaluated how those variations alter the response of materials to the different hydro-
319 mechanical paths, in particular the yield behavior. Although representing the first stage of the
320 suction-controlled oedometer experiments, it appears to be more suitable to discuss the results

321 of free-swell potential experiments in conjunction with those of the swelling pressure experi-
322 ments.

323 **5.1 Compression behavior**

324 Suction-controlled oedometer experiments indicated that the shape of compression curves
325 depended on the hydro-mechanical path, in particular on the initially imposed wetting. The
326 dependency was observed, regardless of the initial dry density. The imposition of hydro-me-
327 chanical paths C and D led to compression curves characterized by the elastic and plastic do-
328 main. In contrast, s-shaped compression curves ensued through the imposition of hydro-me-
329 chanical paths A and B. Their nonlinear compression behavior in the plastic domain disagreed
330 with the formulation of the virgin compression line, whose slope is described by the parameter
331 $\lambda(s)$ [2]. The applicability of the framework thus appeared to be limited.

332 In general, at a given suction, loading induces the collapse of macropores, and in turn,
333 increases the degree of saturation. In other words, unsaturated samples could saturate through
334 compaction [e.g. 28]. The nonlinear behavior might be related to the fact that the compressibil-
335 ity varied as the degree of saturation increased [e.g. 44]. Models proposed in Gallipoli et al.
336 [14] and Zhou et al. [44] relate the compressibility to the degree of saturation, instead of suc-
337 tion. By this means, they allow to describe the transition of the compressibility in unsaturated
338 state to that in saturated state through compaction. This prediction was consistent as compres-
339 sion curves converged at high vertical stresses ($\sigma_v \geq 1000$ kPa), regardless of the imposed suc-
340 tion. In the context of in situ compacted backfill materials, the prediction implied a negligible
341 impact of the saturation state on the compression behavior when the overburden pressure fully
342 affects the backfill material.

343 In the frameworks proposed in Alonso et al. [2] and Alonso et al. [3], the loading-collapse
344 (LC) curve gives the functional relation between the yield stress and imposed suction. Corre-
345 sponding yield loci were derived from suction-controlled oedometer experiments. As depicted

346 in Fig. 8a, the variation of yield stress with imposed suction was almost identical, in the cases
347 of type I-, II-, and III-samples. LC-curves thus coincided. Yield stresses of type IV-samples
348 were lower, in particular when hydro-mechanical paths C ($s = 200$ kPa) and D ($s = 2000$ kPa)
349 were imposed. In ranges of lower suctions, the two LC-curves converged. Two conclusions can
350 be drawn from Fig. 8. At higher suctions, slight variations in the initial dry density had no
351 impact on the yield behavior. There was an apparent threshold, below which the initial dry
352 density came into effect. At lower suctions, the impact of variations in the initial dry density
353 vanished. It was manifested by the convergence of LC-curves.

354 At lower suctions, crystalline swelling and partial particle breakup ensued as the hydra-
355 tion front propagated entirely through the sample under free conditions. Collapse of macropores
356 was less probable due to the small vertical stress ($\sigma_v \approx 10$ kPa). The processes involved an
357 increase in micropores and a decrease in macropores as swelling particles penetrated the macro-
358 pores. Not only did pore merging turn the bimodal pore size distribution function into a uni-
359 modal one, it also increased the total porosity, regardless of the initial dry density [e.g. 41, 42].
360 In general, a unimodal pore size distribution function implies that mechanical loading affects
361 the overall structure. At lower suctions, loading in the elastic domain adapted the amount of
362 pores in all samples, as indicated by the coinciding void ratios in Fig. 8b. The pores collapsed
363 under comparable stresses, thus the stiffness of the overall structure and in turn, the yield
364 stresses appeared to converge.

365 In contrast, at higher suctions, the structure of mixture samples was characterized by mi-
366 cro- and macropores, and mechanical loading affected only the aggregates surrounding the lat-
367 ter pore population. LC-curves of type I-, II-, and III-samples might coincide due to a still com-
368 parable amount of macropores and, in turn, a similar stiffness of soil aggregates. Accordingly,
369 the elevated macroporosity in type IV-samples accounted for the lower yield stresses. It might

370 be of interest to corroborate the hypothesis of comparable structures after free swelling by
371 means of microstructural investigations (e.g. MIP-experiments).

372 In the context of in situ compacted backfill materials, the results of suction-controlled
373 oedometer experiments highlighted that the initial dry density has an impact of the compression
374 behavior of the mixture, in particular on the yield behavior, only if lower vertical stresses and
375 higher suctions are imposed. If samples saturate under free-swell conditions, an adaption of the
376 total porosity appears to involve a comparable stiffness of the overall structure.

377 5.2 Swelling behavior

378 Suction-controlled constant-volume swelling pressure experiments indicated that the evo-
379 lution of swelling pressure was characterized by a post-peak stabilization at lower or compara-
380 ble stresses. Although the increase in initial dry density accompanied an increase in swelling
381 pressure at a given suction (Fig. 7a), there was no functional relation between initial dry density
382 and swelling pressure.

383 As implied in the introduction, Lloret et al. [23] adopted parts of the elastoplastic frame-
384 work proposed in Alonso et al. [3] in order to interpret the double peak pattern. The initial
385 reduction of suction caused homogeneous swelling of particles. Increasing vertical stresses
386 compensated for the swelling of particles as the stiffness of soil aggregates was high. Decreas-
387 ing vertical stresses followed the first maximum value and indicated that the yield stress was
388 reached. The partial collapse of macropores lowered the stiffness of soil aggregates and pro-
389 gressing swelling of particles cannot compensate for the collapse of macropores. As the reduc-
390 tion of suction reduction, swelling of particles prevailed over collapse of macropores, and in-
391 creasing vertical stresses compensated for the swelling of particles again. The phenomenologi-
392 cal interpretations proposed in Massat et al. [24] and Yigsaw et al. [40] were similar to that of
393 Lloret et al. [23]. Yet, they specified that macrostructural rearrangements accounted for an in-
394 crease in material stiffness and a consequent dominance of swelling of particles. In this study,

395 the evolution of swelling pressure was characterized by a post-peak stabilization at lower or
396 comparable stresses. In the cases of type I-samples, vertical stresses stabilized at comparable
397 values after reaching the peak. Their lower initial macroporosity apparently limited the struc-
398 tural rearrangement, in particular the collapse of macropores. Conversely, in the cases of type
399 III- and IV-samples, the swelling of particles appeared to be less pronounced than the collapse
400 of macropores, thus the vertical stresses stabilized at lower stresses.

401 Wang et al. [39] described a potential functional relation between the imposed suction
402 and swelling pressure which implied a monotonous increase in swelling pressure. Since they
403 attributed this finding to the homogeneous swelling of particles and the high stability of soil
404 aggregates, the question was raised why aggregates might be differently stable.

405 Middelhoff et al. [25] conducted constant-volume swelling pressure experiments on mix-
406 ture samples prepared to the same initial conditions. Their experiment protocol comprised the
407 direct saturation of samples with deaired/ deionized water. The results complemented those
408 recently obtained and highlighted the hydraulic path independency of the material, as depicted
409 in Fig. 9a and Fig. 9b. The independence appeared to be valid, regardless of the initial dry
410 density of the material. Wang et al. [39] conducted similar swelling pressure experiments on
411 compacted bentonite/ claystone-mixture samples, whose mineralogical composition was dom-
412 inated by smectites. They also reported on a hydraulic path independency. According to their
413 interpretation, the direct and stepwise saturation triggered sequentially comparable structural
414 rearrangements and led eventually to a similar microstructure.

415 The protocol of suction-controlled oedometer experiments comprised the determination
416 of the free-swell potential of mixture samples at their initial stage. The relationship between the
417 initial dry density and free-swell potential of samples can be described by a linear function,
418 whose slope increased logarithmically as the suction decreases. In the case of suctions being
419 equal to 2000 kPa, no swelling was observed. Presumably, the increasing smectite mass in

420 denser samples caused the linear relation since each particle can undergo crystalline swelling
421 and particle breakup under the availability of water molecules [e.g. 22]. Major macrostructural
422 rearrangements were less probable due to the low initial vertical load.

423 In general, suction-controlled constant-volume swelling pressure experiments indicated
424 that stepwise wetting involved a non-monotonic increase in swelling pressure, regardless of the
425 initial dry density. The post-peak stabilization at lower or comparable stresses was attributed to
426 structural rearrangements, in particular particle swelling and partial macropore collapse. In the
427 case of denser samples, the latter aspect might account for the stabilization at comparable
428 stresses. Accordingly, the partial collapse was less significant due to the smaller amount of
429 macropores therein. The evolution of swelling pressure was hydraulic path independent poten-
430 tially attributable to a similar final microstructure. The free-swell potential experiments indi-
431 cated a functional relation between the initial dry density and final free-swell potential at a
432 given suction. It was attributed to the availability of water molecules and the larger amount of
433 smectite particles in denser samples.

434 5.3 Comparison of yield behavior

435 As described above, the (suction-controlled) propagation of the hydration front through
436 the sample under constant-volume conditions involved the initial swelling of particles, followed
437 by the collapse of aggregates and the final dominance of particle swelling over aggregate col-
438 lapse. The swelling pressure evolved in conjunction with those structural rearrangements. In
439 contrast, under oedometric conditions, structural rearrangements were terminated before load-
440 ing. The differently rearranging structure might account for the fact that the yield loci of denser
441 samples differed significantly, whereas those of looser samples coincided, as depicted in
442 Fig. 10.

443 In the case of type I- and III-samples, the partial collapse of aggregates and the subsequent
444 swelling of particles appeared to increase the density of the material and, in turn, the stiffness

445 of the overall structure [24]. Their yield stress increased as a consequence. Type IV-samples
446 were characterized by an elevated fraction of macropores, and their partial collapse cannot be
447 compensated by the swelling of particles. The densification was less effective, and the stiffness
448 of the overall structure measured under constant-volume conditions became comparable to that
449 determined under oedometric conditions. Indeed, the findings partially corroborated the hy-
450 pothesis suggested by Romero [33] as the yield loci of looser samples coincided. However, the
451 yield behavior of denser samples appeared to considerably depend on the hydro-mechanical
452 path. Conclusion

453 The French reference considers a mixture composed of 70% processed Callovo-Oxford-
454 dian claystone spoil and 30% MX80-bentonite to be a material, which could be potentially em-
455 ployed to backfill drifts and shafts of the future repository for intermediate- and high-level ra-
456 dioactive waste in the clay-rich Callovo-Oxfordian sedimentary rock formation. Regardless of
457 the saturation state, the convergence of the surrounding rock formation likely induces mechan-
458 ical loading. Thus, the occurrence of different hydro-mechanical paths is possible. Installed by
459 means of conventional compaction methods, the backfill material is likely characterized by
460 spatial variations in its initial dry density. Since the initial dry density is one of the factors
461 controlling the water retention characteristics and hydro-mechanical behavior of smectite-con-
462 taining material, it is of major relevance to analyze the impact of its variations on the perfor-
463 mance of claystone/ bentonite-mixtures, in particular in unsaturated state.

464 At its initial stage, this laboratory experimental program analyzed the mixture with regard
465 to its microstructural and water retention characteristics. Subsequent suction-controlled oedom-
466 eter and constant-volume swelling pressure experiments aimed to evaluate how variations in
467 the initial dry density affect the compression and swelling behavior. The final comparison of
468 experiment results revealed whether the hydro-mechanical behavior of the mixture depends on

469 the hydro-mechanical path, and whether variations in the dry density have an impact on possible
470 dependencies. Following conclusions can be drawn from the experiments conducted:

- 471 1. The results of microstructural analysis agreed with the trends generally described in the
472 literature. In contrast to micropores, whose amount remained stable, macropores were
473 considerably affected by densification. A reduction of dry density by about 20% de-
474 creased their amount by about 50%.
- 475 2. As expected, water retention characteristics highlighted the existence of micro- and
476 macropores since the impact of the initial dry density vanished at higher suctions. The
477 observation was related to the fact that micropores control capillary phenomena at higher
478 suctions.
- 479 3. The impact of the initial dry density on the compression behavior of mixture samples
480 vanished when higher vertical stresses or lower suctions were imposed. Although suctions
481 were imposed, the compression curves of unsaturated samples converged into those of
482 saturated samples at higher stresses.
- 483 4. At a given suction, the free-swell potential of the mixture was linearly related to the initial
484 dry density. In turn, the slope of this linear function varied logarithmically with the im-
485 posed suction. The findings were attributed to the larger amount of smectite particles in
486 denser samples and to the greater availability of water molecules at lower suctions.
- 487 5. Suction-controlled hydration of mixture samples under constant-volume conditions in-
488 volved the evolution of swelling pressure, whose development was characterized by a
489 post-peak stabilization at lower or comparable stresses. The pattern evolved regardless of
490 the initial dry density and was attributed to rearrangements at the micro- and macrostruc-
491 tural level. The swelling pressure increased with increasing initial dry density at a given
492 suction. Apart from those findings, experiments revealed that the evolution of swelling

493 pressure was independent of the hydraulic path potentially attributable to a similar final
494 microstructure.

495 6. The comparison of results highlighted that the initial dry density of samples had an impact
496 on their hydro-mechanical path dependency. In the case of looser samples, the yield be-
497 havior under constant-volume and oedometric conditions was comparable, whereas, in
498 the case of denser samples, the yield stresses were shifted to lower values under oedomet-
499 ric conditions. The difference in yield locus position was attributed to an adaption of the
500 total porosity and a comparable stiffness of the overall structure.

501 This study confirmed that the microstructural and water retention characteristics of the clay-
502 stone/ bentonite-mixture considerably changed as a response to variations in their initial dry
503 density. Variations in the initial dry density had a significant impact on the swelling behavior
504 of the mixture, regardless of the saturation state. Regarding the compression behavior, the im-
505 pact vanished as lower suctions or higher vertical stresses were imposed. In the context of in
506 situ compacted backfill materials, neither the initial dry density nor the saturation state accord-
507 ingly have an impact on the compression behavior when the material is subjected to the over-
508 burden pressure once the concrete lining degrades. The evolution of swelling pressure under
509 constant-volume conditions was hydraulic path independent, whereas the yield behavior was
510 hydro-mechanical path independent only when the initial dry density of the material was lower.
511 Regarding future laboratory experimental studies, it might be of interest to corroborate the hy-
512 pothesis of the comparable stiffness of the overall structure by complementing the different
513 stages in oedometer experiments by means of X-ray tomography.

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522 **Availability of data and material**

523 The datasets generated during and/or analysed during the current study are available from the
524 corresponding author on reasonable request.

525 **Author's contributions**

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527 **Methodology:** Marvin Middelhoff, Olivier Cuisinier, Farimah Masrouri, Jean Talandier; **For-**
528 **mal analysis and investigations:** Marvin Middelhoff; **Writing – original draft preparation:**
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532 Jean Talandier

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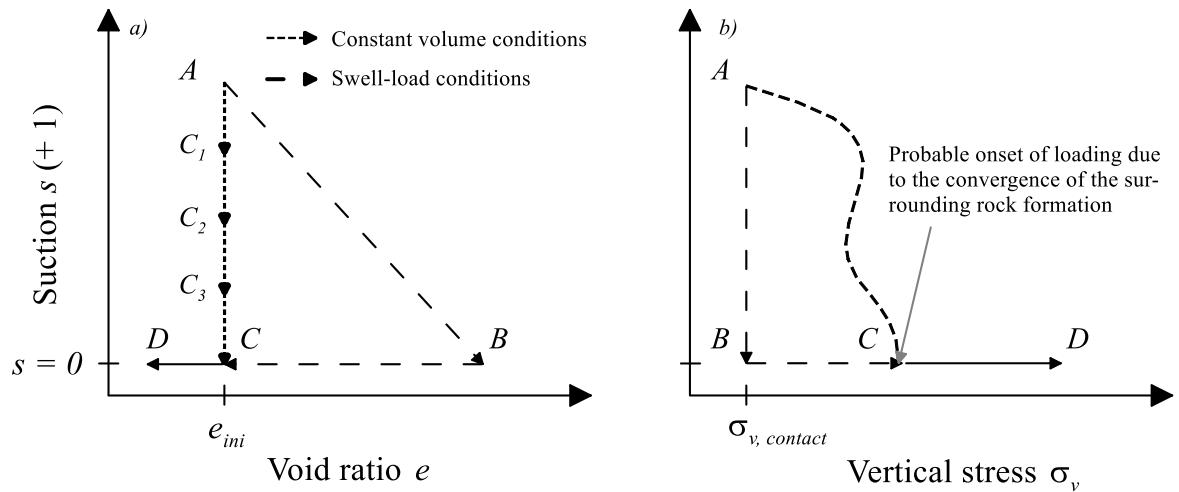
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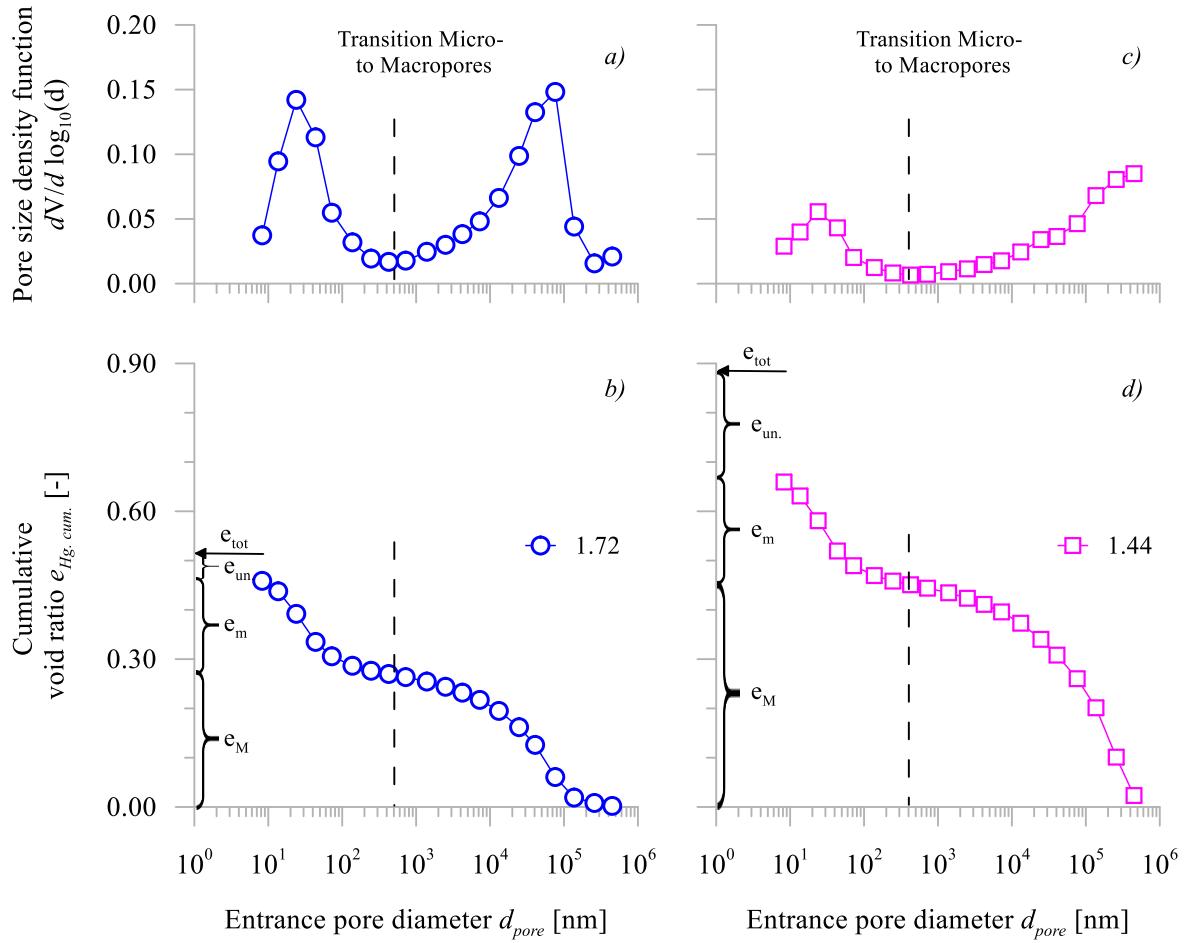
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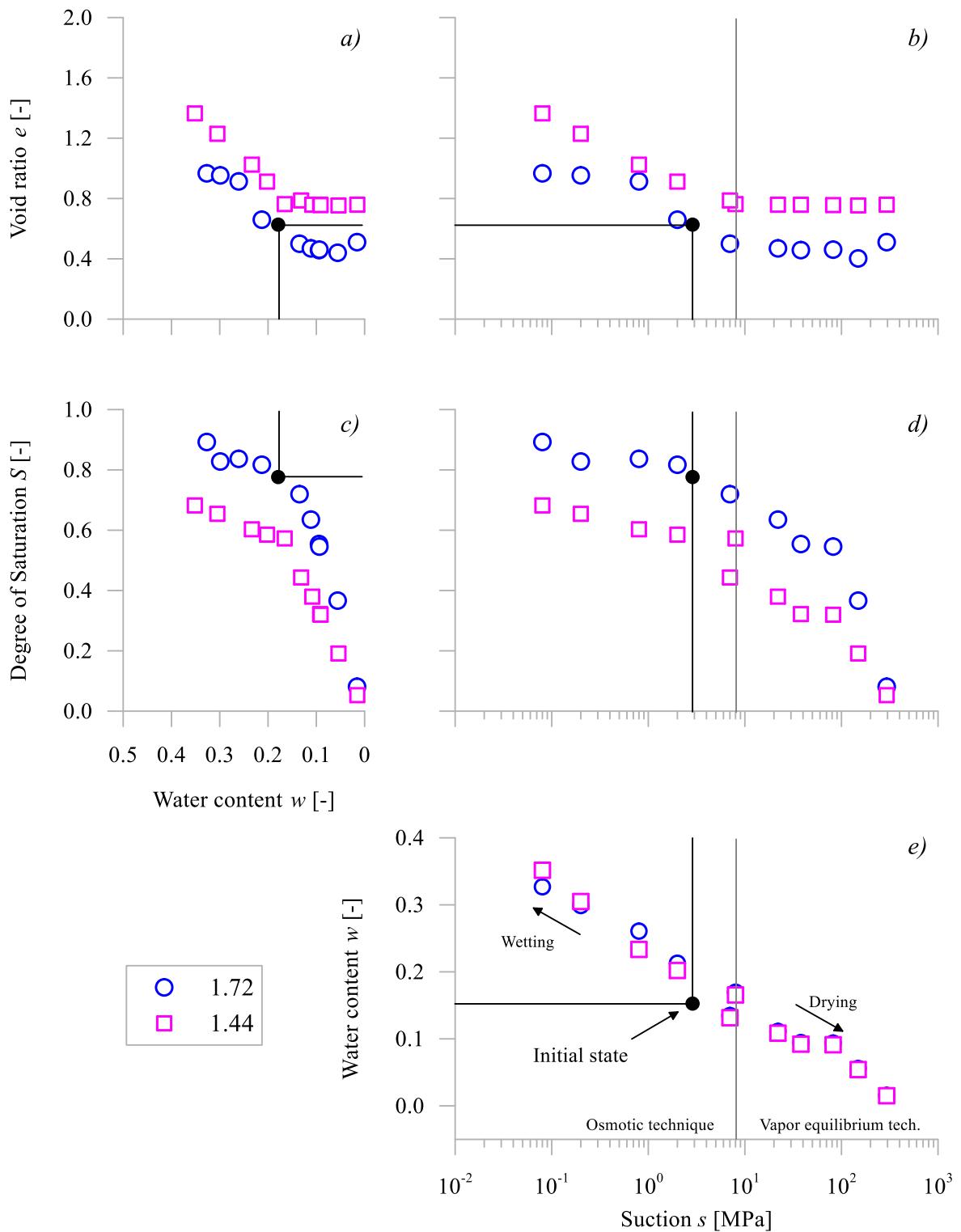
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659 **Fig. 1** Possible combinations of hydraulic and mechanical paths subjected to the backfill upon terminat-
660 ing the installation phase



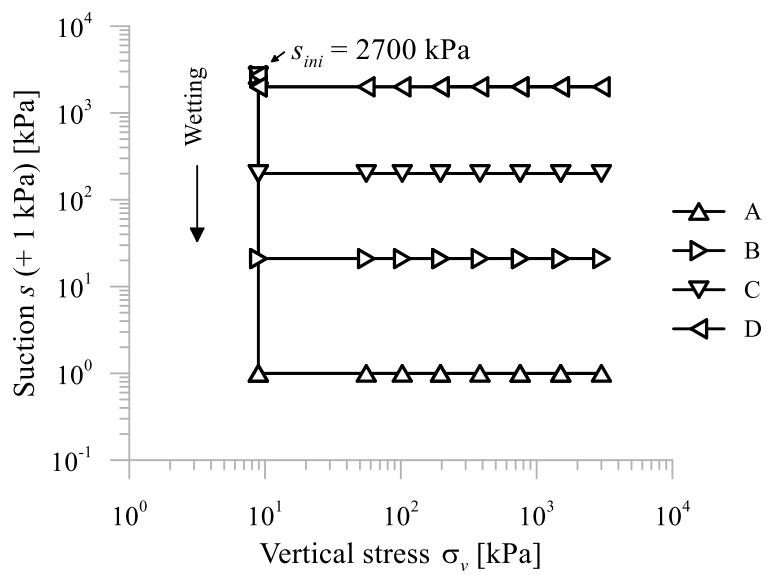
661

662 **Fig. 2** Results of mercury intrusion porosimetry (MIP) experiments conducted on samples characterized
 663 by initial dry densities of $\rho_{d,ini} = 1.72 \text{ Mg/m}^3$ and $\rho_{d,ini} = 1.44 \text{ Mg/m}^3$



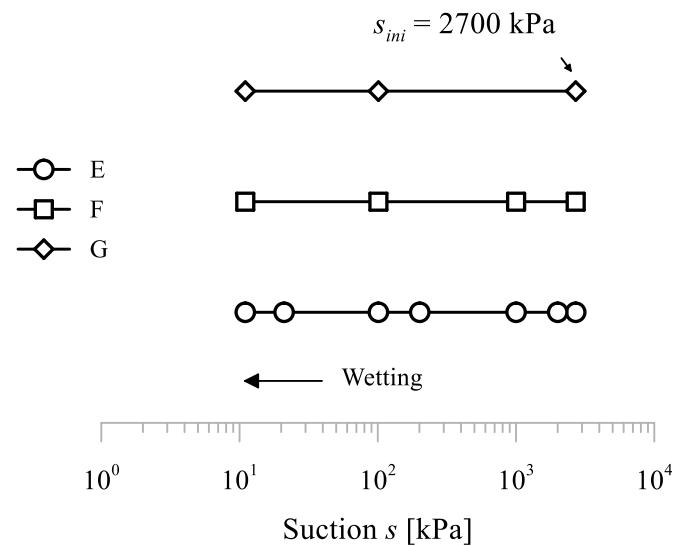
664

665 **Fig. 3** Water retention characteristics of mixture samples characterized by initial dry densities of $\rho_{d,ini} =$
 666 1.72 Mg/m^3 and $\rho_{d,ini} = 1.44 \text{ Mg/m}^3$



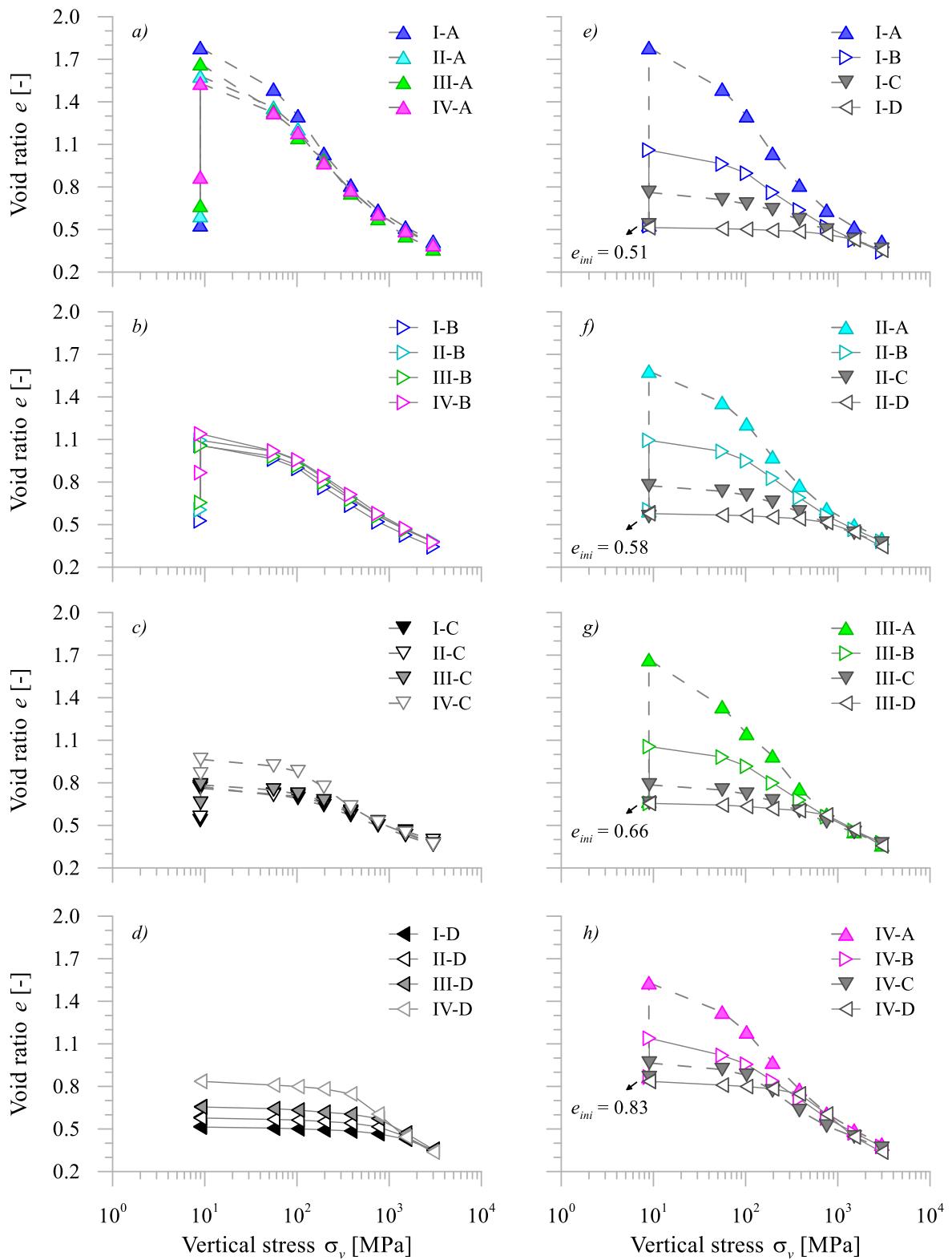
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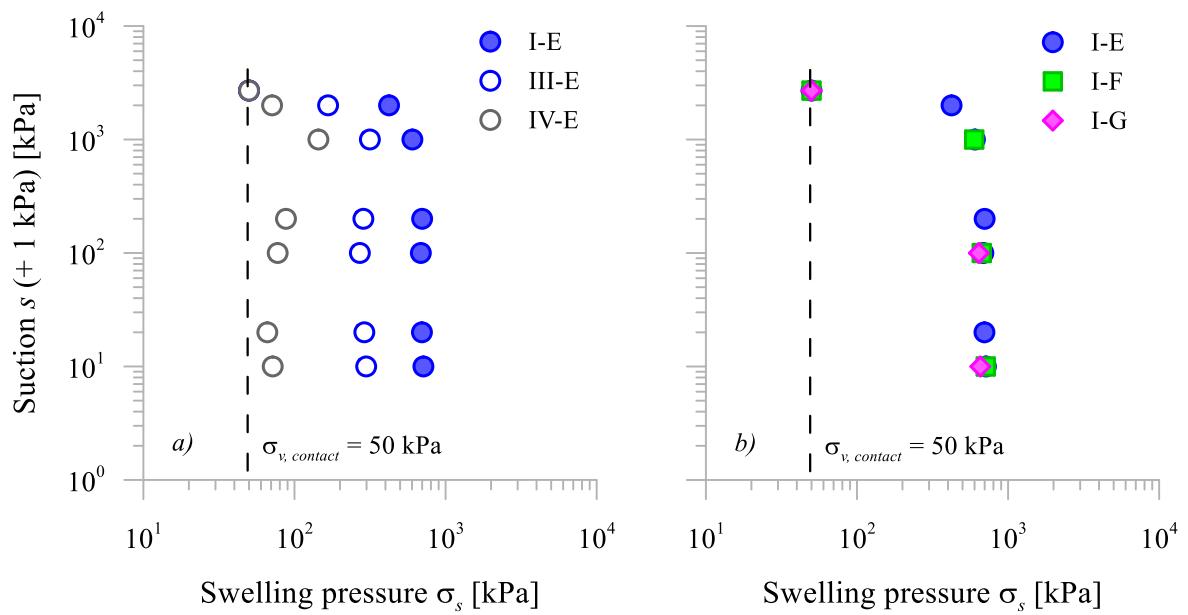
668 **Fig. 4** Hydro-mechanical paths followed in suction-controlled oedometer experiments



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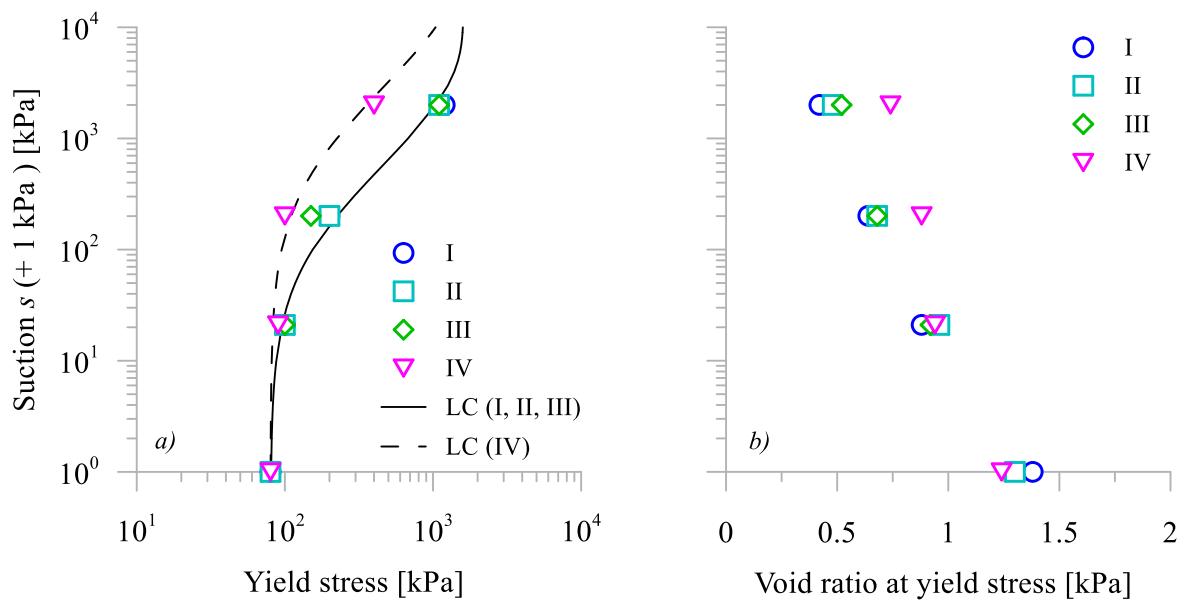
670 **Fig. 5** Hydration paths followed in suction-controlled constant-volume swelling pressure experiments





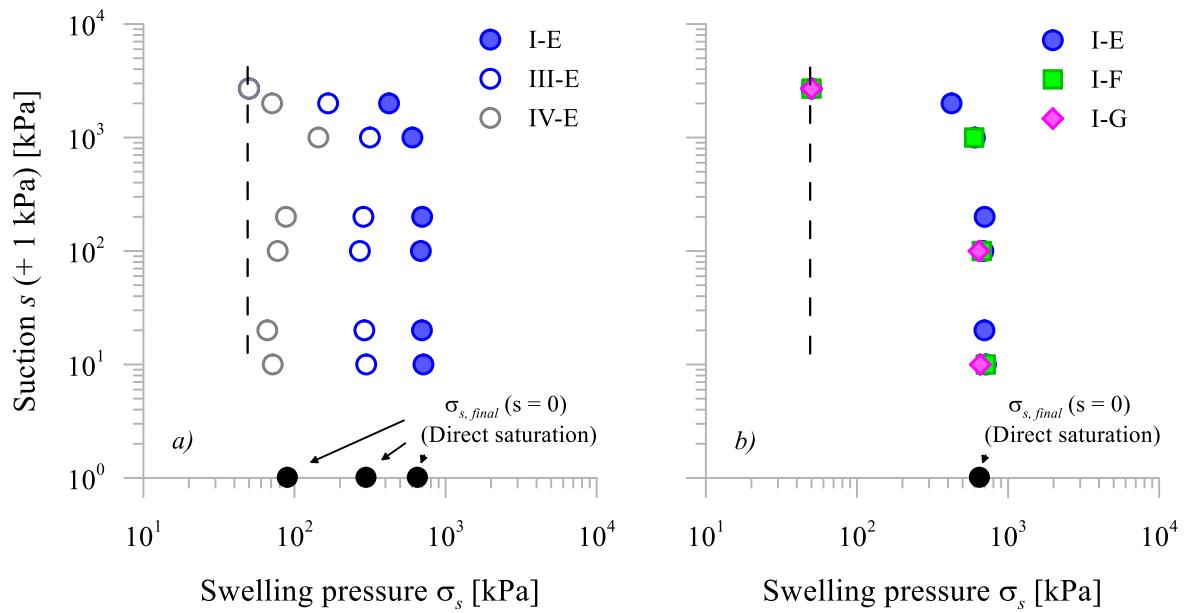
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675 **Fig. 7** Swelling pressure evolution of a) samples characterized by different initial dry densities upon
 676 imposing hydration path E and b) samples characterized by the same initial dry density upon
 677 imposing hydration paths E, F, and G



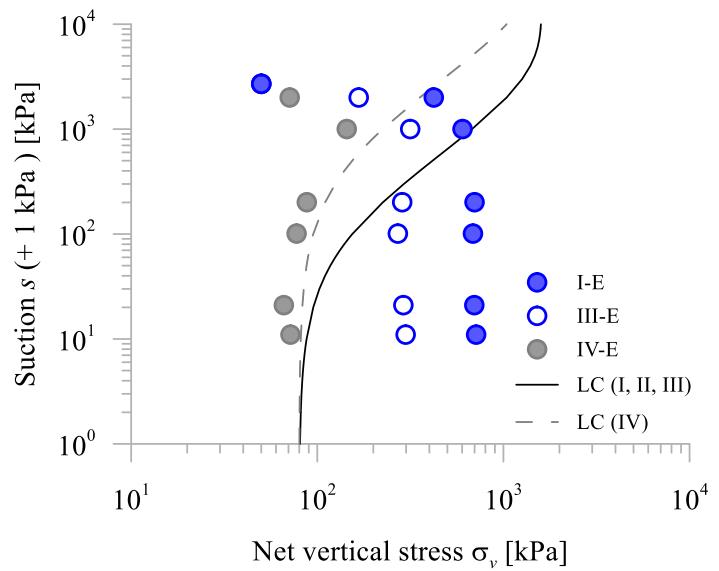
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679 **Fig. 8** Loading-collapse (LC) curves derived from suction-controlled oedometer experiments



680

681 **Fig. 9** Comparison between the swelling pressure evolution of a) samples characterized by the same
682 initial dry density upon saturating directly and imposing hydration paths E and b) samples upon saturat-
683 ing directly and imposing hydration paths E, F, and G (Values of direct saturation were taken from [19])



684

685 **Fig. 10** Comparison between LC-curves derived from suction-controlled oedometer experiments and
 686 swelling pressure evolution under constant-volume conditions

Table 1 Initial characteristics of samples

Type	Initial dry density $\rho_{d, ini}$ [Mg/m ³]	Initial water content $w_{ini.}$ [-]	Initial void ratio $e_{ini.}$ [-]	Initial porosity $n_{ini.}$ [-]	Initial degree of saturation $S_{ini.}$ [-]	Initial suction* $s_{ini.}$ [kPa]
I	1.72	0.182	0.53	0.35	0.90	2700
II	1.66	0.182	0.59	0.37	0.82	
III	1.59	0.182	0.66	0.40	0.73	
IV	1.44	0.182	0.86	0.46	0.56	

*: measured by means of chilled mirror method

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Table 2 Experiment program including microstructural and water retention analysis, suction-controlled constant-volume swelling pressure and oedometer experiments

Sample type ¹	Oedometer experiments ²				Swelling pressure experiments ³		
	A	B	C	D	E	F	G
I	✓	✓	✓	✓	✓	✓	✓
II	✓	✓	✓	✓			
III	✓	✓	✓	✓			
IV	✓	✓	✓	✓			

¹: compare Table 1

²: Hydro-mechanical paths (compare Fig. 4)

³: Hydro-mechanical paths (compare Fig. 5)

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