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(Article begins on next page)

Salinity and oven-drying effects on the plasticity of a marine soft clay

Laxmi Suwalⁱ⁾, Jubert Pineda^{ii)*}, Brett Turnerⁱⁱⁱ⁾ and Guido Musso^{iv)}

- i) ATC Williams, Newcastle, Broadmedow, NSW, 2292, Australia (formerly at The University of Newcastle Australia)
- ii) Priority Research Centre for Geotechnical Science and Engineering, School of Engineering, The University of Newcastle, Callaghan, NSW 2308, Australia
- iii) School of Civil and Environmental Engineering, University of Technology Sydney, Ultimo NSW 2007, Australia (formerly at The University of Newcastle Australia)
- iv) Politecnico di Torino, Corso Duca degli Abruzzi 24, 10129 Torino, Italy

Corresponding author(*): Jubert.Pineda@newcastle.edu.au

Abstract

Considering the extensive use of plasticity-based correlations in geotechnical practice to estimate soil parameters, this paper evaluates the influence of pore fluid salinity and soil drying on the plasticity of Ballina clay, a estuarine soft clay from northern New South Wales (Australia). A comprehensive experimental study which includes controlled leaching/salinisation paths applied to natural (remoulded) as well as oven-dried clay prior to the estimation of the Atterberg limits is presented. Plasticity tests are complemented with chemical analysis of the pore fluid carried out to evaluate the processes involved in the leaching/salinisation mechanisms for remoulded and oven-dried clay. Strong dependency of liquid limit on pore fluid salinity and oven-drying are observed in Ballina clay. Leaching modifies the soil fabric from an initially saline-sodic flocculated towards a normal flocculated arrangement. The experimental results show that changes in soil plasticity upon leaching are largely reversible upon salinisation paths. Oven-drying promotes the stacking of clay minerals (aggregation) which in turn reduces the water absorption capacity of the clay. **The consequences of neglecting both salinity and drying effects in practice when adopting well-established relationships between mechanical parameters and soil plasticity are also briefly discussed.**

39 **Notation**

40 *LL: liquid limit*

41 *PL: plastic limit*

42 *PI: plasticity index*

43 *PSD: particle size distribution*

44 *w: gravimetric water content*

45 *w_{fluid}: fluid content*

46 *r: salinity*

47 *EC_{fluid}: electrical conductivity of the pore fluid*

48 *EC_{bulk}: electrical conductivity of bulk's soil*

49 *SEM: Scanning Electron Microscopy*

50 *SAR: solution adsorption ratio*

51 *I: solution ionic strength*

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76 **1. Introduction**

77 Several engineering problems such as NAPL soil contamination, rainfall driven erosion, salt water
78 intrusion due to sea level rise and salt leaching are associated with changes in pore fluid
79 chemistry. A classical example related to marine soft soils is the formation of sensitive and ‘quick’
80 clays in Scandinavia due to salt leaching by fresh water (e.g. Rosenqvist, 1953; Bjerrum, 1954,
81 1967; Torrance, 1974). The large increase in sensitivity in those soils is mainly caused by the
82 reduction in the remoulded shear strength, a phenomenon associated with the development of a
83 meta-stable microstructure (e.g. Rosentqvist, 1953). A direct consequence of salt leaching is the
84 change in soil plasticity. Apart from those early studies on sensitive clays from Scandinavia, most
85 of the available literature has been devoted to the behaviour of pure clays or artificial mixtures
86 (e.g. Ohtsubo et al., 1985; Sridharan et al., 1989; Di Maio, 1996; Di Maio et al, 2004; Palomino
87 and Santamarina, 2005; Calvello et al., 2005; Jang and Santamarina, 2016; Musso et al., 2023).
88 Those studies have shown that liquid limit reduces with the increase in salt concentration, and
89 this effect is small for illite, moderate for kaolinite and significant in bentonites. The opposite trend
90 is reported for natural non-expansive soft clays, i.e. the liquid limit increases with pore fluid salinity
91 (e.g. Musso et al., 2023). The magnitude of this change seems to be controlled by the
92 mineralogical composition of the natural soil. Nevertheless, a strong decrease in liquid limit has
93 been reported in soils exposed to drying prior to testing, leading to misleading soil’s clasification.
94 This phenomenon, which appears to be strongly soil-dependent, is attributed to several factors
95 like the presence of organic matter (e.g., Hight et al., 1992), halloysite minerals (e.g. Herrera et
96 al., 2007; Wesley, 2010; Jang and Santamarina, 2016) as well as to irreversible changes in soil
97 microstructure due to soil aggregation (e.g., Rao et al., 1989; Pandian et al., 1991).

98
99 Natural soft soils deposits of Quaternary age are commonly found along Australian coastlines
100 (e.g., Low et al., 2011; Pineda et al., 2016). These deposits, sedimented under marine conditions,
101 serve as foundation soil for most of the onshore country’s transport and energy infrastructure.
102 Their mineralogical composition is complex, including non-active minerals (kaolinite and illite),
103 interstratified minerals (illite/smective or illite-bearing smectite) (Pineda et al., 2016) and, in some
104 cases, active minerals (smectite) (Low et al., 2011). An important feature of these soils is the
105 influence of oven-drying on soil plasticity. Results obtained by Kelly et al. (2013) and Pineda et
106 al. (2013, 2016) on Ballina clay, a soft clay from northern New South Wales, demonstrate a
107 reduction in liquid limit of 30% – 40% when the soil is dried prior to testing. Although this behaviour
108 was assumed to be the result of the organics present in the natural soil (1-3%), such a mechanism
109 has not yet been verified.

110
111 Considering the extensive use of plasticity-based correlations in geotechnical practice to obtain
112 soil parameters (e.g. Wroth and Muir Wood, 1978; Carrier and Beckham, 1984; Carrier, 1985;

113 Chandler, 1988; Muir Wood, 1990; Terzaghi et al., 1996; ASTM, 2005), this paper evaluates the
114 influence of pore fluid salinity and oven-drying on soil plasticity for an Australian estuarine soft
115 clay. A comprehensive experimental study is presented, which includes controlled
116 leaching/salinisation paths applied to natural (remoulded) and oven-dried clay prior assessing
117 soil plasticity. Chemical analysis of the pore water is used to study the pore fluid chemical
118 changes due to leaching/salinisation for remoulded and oven-dried clay. **The consequences of
119 neglecting the effects of pore fluid composition and oven-drying in practice are also discussed.**

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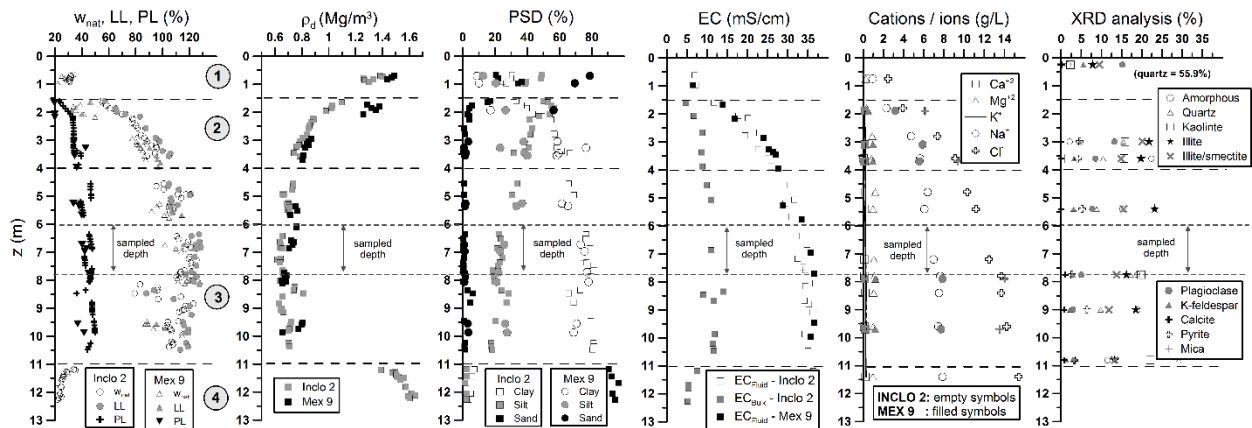
121 **2. Materials**

122 The soil tested is Ballina clay, an estuarine soft clay encountered at the National Soft Soil Testing
123 Facility NFTF (Kelly et al., 2017; Pineda et al., 2019) located in the town of Ballina, northern New
124 South Wales, Australia. Detailed *in situ* and laboratory characterisation studies were carried out
125 after the establishment of the NFTF in 2013 (Pineda et al., 2016; Kelly et al., 2017). As observed
126 in Fig. 1, the natural water content increases with depth from 20% ($z \approx 1$ m) up to 120% ($z > 7$
127 m). The liquid limit LL is typically 10-15% higher than the natural water content whereas the plastic
128 limit PL ranges between 20-53%. Natural dry density reduces from 1.50 Mg/m³ at ground surface
129 to 0.70 Mg/m³ at 4.5 m depth. However, minor changes are noticed between 3 m and 11 m.
130 Despite the differences in sand content (column PSD, Particle Size Distribution) observed above
131 **2 m**, the clay fraction is predominant up to 11 m depth, with maximum values around 82%. Soils
132 at Ballina site are composed of amorphous minerals, kaolinite, illite, quartz, interstratified
133 illite/smectite, plagioclase, pyrite, K-feldspar, mica and calcite. Sodium (0.93–8.23 g/L) and
134 chloride (2.5–15.4 g/L) are the most abundant cations and anions in the pore fluid whereas the
135 alkalinity of the pore water lies between 0.6-2.2 g/L of CaCO₃. The bulk electrical conductivity
136 EC_{bulk} increases with depth from 4 to 15 mS/cm whereas the pore fluid conductivity EC_{fluid}
137 increases from 7 up to 36 mS/cm (average value below 5 m). This corresponds to salt
138 concentrations between 4–23.5 g/L. The presence of salts during soil deposition promoted a
139 porous fabric composed by aggregates separated by large voids. Four soil layers can be identified
140 in the upper 13 m (see Fig. 1) (Pineda et al., 2019). Layer 1 is a silty-sand alluvial crust. Layers
141 2 and 3 comprises the upper and lower estuarine silty clay (hereafter Ballina clay). Layer 4 is the
142 silty-sand transition layer which overlies a sand layer and a deeper stiff clay layer.

143

144 The soil used in the experiments described below was obtained from high-quality block samples
145 retrieved with the Sherbrooke sampler between 6.0-7.8 m depth. At this level, natural water
146 content ranges between 110–122% whereas clay, silt and sand contents vary between 79-81%,
147 18-20% and 0-2%, respectively. The soil classifies as clay of extremely high plasticity with soil
148 activity around 1. The organic content at this depth ranges around 1.8%. Tables 1 and 2

149 summarise the mineralogical composition and the geochemical results reported by Pineda et al.
 150 (2016).



151
 152 **Fig. 1.** Index properties and geochemical composition of soil deposits at Ballina site (modified
 153 from Pineda et al., 2016)
 154

155 **Table 1.** XRD analysis for natural specimens (Pineda et al., 2016)

Depth (m)	Amorphous (%)	Quartz (%)	Plagioclase (%)	K-feldspar (%)	Calcite (%)	Pyrite (%)	Kaolinite (%)	Mica (%)	Illite (%)	Illite/smectite (%)
5.4	15.2	8.8	7.7	3.1	0	5.2	19.3	2.1	23.16	15.44
7.5	18.9	17.3	5	2.8	1	2.7	19.8	2.1	16.2	13.8

156

157 **Table 2.** Ions in pore fluid for natural specimens (Pineda et al., 2016)

Depth (m)	Ca ⁺⁺ (g/l)	Mg ⁺⁺ (g/l)	K ⁺ (g/l)	Na ⁺ (g/l)	Cl ⁻ (g/l)	Br ⁻ (g/l)	S ₂ ⁻ (g/l)
7.2	0.09	1.06	0.22	6.97	12.46	0.05	0.17
7.8	0.09	1.09	0.26	7.63	13.68	0.05	0.15

158

159

160 3. Experimental procedures

161 3.1 Fluid content

162 In soils that contains salts dissolved in the pore fluid, a correction to the gravimetric water content
 163 must be applied (e.g. Noorany, 1984; Frydman et al., 2008). The corrected water content (or fluid
 164 content) is then expressed as (Noorany, 1984):

165
$$W_{fluid} = \frac{w}{(1-r)-r*w} \quad (1)$$

166 where w_{fluid} is the fluid content, w is the water gravimetric water content and r is the salinity defined
 167 as the ratio between the mass of dissolved salts and the total mass of fluid $r = M_{salt} / M_{fluid}$. In this
 168 study, synthetic NaCl solutions were used to achieve predetermined salinities. Fig. 2a shows the
 169 relationship between NaCl solubility and the electrical conductivity of the pore fluid EC_{fluid}
 170 (supernatant) measured in the laboratory. The salinity r required in the estimation of the fluid
 171 content (Eq. 1) was obtained from Fig. 2b.

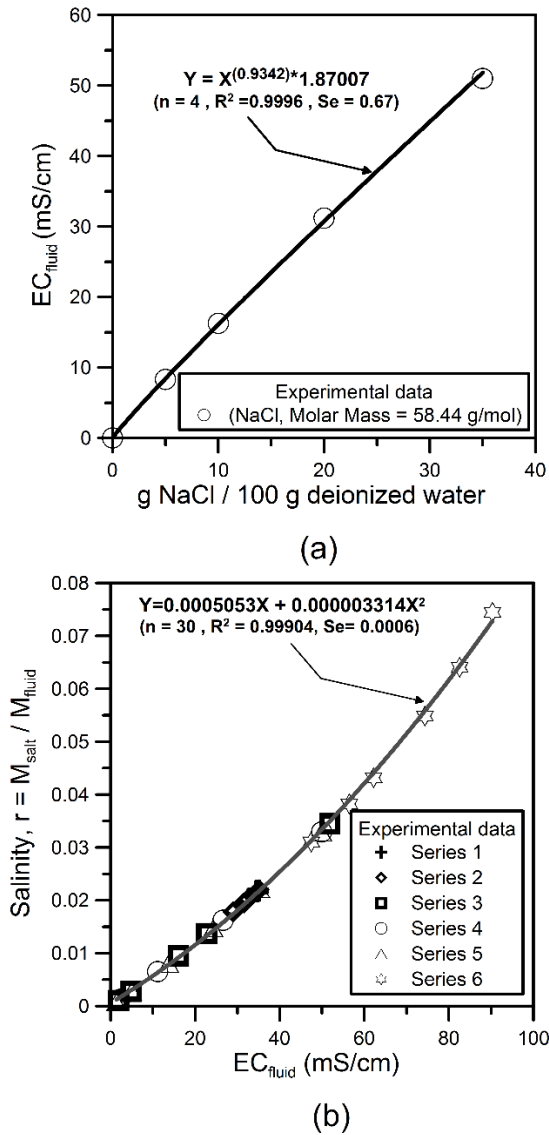


Fig. 2 Salinity and pore fluid conductivity

172
 173
 174 **3.2 Leaching / salinisation tests**
 175 Leaching and salinisation paths were applied to remoulded clay (at natural water content) and
 176 oven-dried material (initially mixed with deionized water or saline solutions up to 1.5 times its
 177 natural water content) before the estimation of Atterberg limits. Saline solutions were percolated
 178 through soil samples by applying a vacuum pressure of 30 kPa, using the experimental setup
 179 shown in Fig. 3a. Salt solutions, prepared using the relationships of Fig. 2b, were periodically
 180 renewed to avoid changes in the target EC_{fluid} . Percolation continued until equilibrium between

181 the electrical conductivity of the reference solution and the percolated fluid was achieved
182 ($EC_{\text{solution}} \approx EC_{\text{fluid}}$): up to 15 weeks were required to achieve fully leached conditions. The mass
183 of the percolated fluid was measured during this process and used in the geochemical analyses
184 presented below. Each sample was prepared as follows. After placing remoulded clay or clay
185 powder inside PVC containers, gently vibration in a shaking table for 5 minutes allowed to remove
186 the trapped air from the soil mass. Two layers of geotextile were used as porous filters at top and
187 bottom ends. The salt solution was then poured on top of the specimen, whereas the bottom
188 drainage was connected to the vacuum line. Periodic measurements of EC_{fluid} and pH of the
189 percolated fluid were made using an Orion EC (013005MB DuraProbe) electrode and an Orion
190 pH (9165BN) electrode, respectively.

191

192 **3.3 Atterberg limits tests**

193 The liquid limit LL was evaluated using the fall cone apparatus (AS 1289.3.9.1-2002). A standard
194 80 g cone with an apex angle of 30° with a smooth surface was employed. The penetration of the
195 cone measured after 5 seconds was used in the estimation of the LL. The plastic limit PL was
196 estimated using the thread rolling method (AS 1289.3.2.1-2002). In conjunction with LL and PL
197 tests, EC_{bulk} , EC_{fluid} and pH were measured as follows. EC_{bulk} was estimated by inserting a probe
198 with automatic temperature compensation (Field Scout Direct Soil EC Meter; Spectrum
199 Technologies, Inc) into the soil mass. The soil was then compressed, under a very slow strain-
200 controlled mode, to squeeze out the 15 cm^3 of pore fluid required to measure EC_{fluid} . Finally, the
201 pH of the pore fluid was measured.

202

203 **3.4 Ion chromatography analysis**

204 Pore water chemistry was evaluated by ion chromatography (IC) analysis using an ICS-5000 ion
205 chromatograph (Thermo Fisher Scientific). Anion analysis was performed via an AS18/AG18
206 analytical and guard columns using 30 mM KOH eluent. Cation analysis was done using the
207 CS12/CG12 analytical and guard columns and 23 mM MSA eluent. All ion analysis used
208 suppressed electrochemical detection (conductivity). Ion chromatography calibration standards
209 were prepared from certified anion and cation standard solutions (Thermo Fisher) by dilution with
210 $18.2 \text{ M}\Omega/\text{cm}$ DI water. To optimise the ion chromatograph peak separation and resolution, all
211 samples were filtered using $0.45 \mu\text{m}$ nylon membrane filter (Pall-Life Sciences) and diluted with
212 $18.2 \text{ M}\Omega/\text{cm}$ DI water.

213

214 **4. Experimental program**

215 The influence of pore fluid salinity and oven-drying on soil plasticity was evaluated through six
216 test series summarized in Table 3 and Fig. 3b. Clay specimens were either mixed or percolated
217 with different solutions before evaluating soil's plasticity. In Series 1, which represents the

218 'natural' conditions, a synthetic solution prepared at *in situ* electrical conductivity ($EC_{fluid}=35$
219 mS/cm) was added to the remoulded clay at its natural water content. The mixture was then
220 mixed, without preliminarily drying the soil. In Series 2, natural clay was mixed with five solutions
221 with conductivities ranging between 0 (deionised water) to 50 mS/cm (sea water). Although
222 changes in pore fluid salinity occurred in Series 2, controlled leaching/salinisation paths were not
223 imposed. An equilibration period of 48 h was allowed to the clay-fluid mixture before estimating
224 LL and PL. Series 3 involved controlled leaching and desalinisation paths to achieve target
225 conductivities. Remoulded specimens were leached from 35 mS/cm to 25 mS/cm, 15 mS/cm, 5
226 mS and 0 mS/cm. Salinisation from 35 mS/cm to 50 mS/cm was also applied in Series 3. To
227 check the reversibility of the leaching-salinisation process on soil plasticity, in Series 4 a large
228 volume of remoulded clay was initially fully leached and then sub-samples were subjected to
229 controlled (re)salinisation to 5, 15, 25 and 50 mS/cm. Series 5 and Series 6 tested oven-dried
230 clay. To do so, remoulded clay was first oven-dried at 60°C for 7 days. A large amount of clay
231 powder was then percolated with deionised water in Series 5 until full leaching was achieved.
232 Afterwards, the leached clay was separated in small containers and exposed to controlled re-
233 salinisation to 5, 15, 25, 35 and 50 mS/cm. In Series 6, oven-dried clay was mixed (i.e. controlled
234 leaching-salinisation not intended) with different synthetic solutions ($EC_{fluid}: 0 \rightarrow 50$ mS/cm). In all
235 cases, an equalisation of 48h was allowed to the clay-fluid mixture before estimating the Atterberg
236 limits. Chemical tests performed on leachate fluid provided the basis for understanding the
237 chemical changes occurring during the leaching and salinisation processes described below.

238
239 **Table 3** Test series carried out to evaluate salinity and drying effects on soil plasticity

Series	Soil	Comments
1	Remoulded (natural water content)	Clay mixed with $EC_{fluid} = 35^*$ mS/cm
2		Clay mixed with $EC_{fluid}: 0, 5, 15, 25$ and 50 mS/cm
3		Clay leached to 0, 5, 15 and 25 mS/cm. Salinisation to 50 mS/cm
4		Clay fully leached followed by re-salinisation to 5, 15, 25, 35 and 50 mS/cm
5	Oven-dried (60°C)	Clay powder mixed with deionised water until achieving full leaching followed by re-salinisation to 5, 15, 25, 35 and 50 mS/cm
6		Clay powder mixed with $EC_{fluid}: 0, 5, 15, 25, 35$ and 50 mS/cm

* $EC_{in situ} = 35$ mS/cm.

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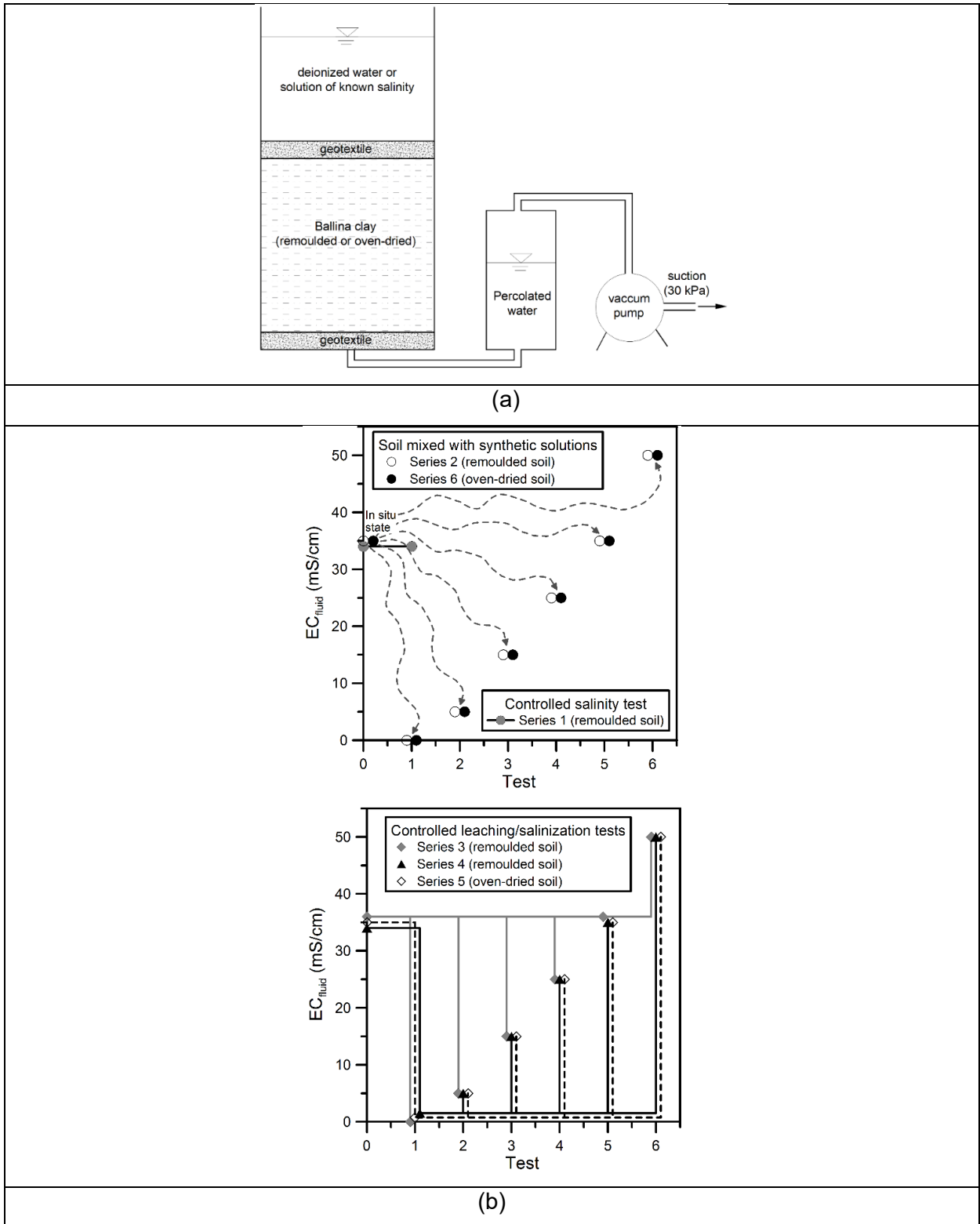


Fig. 3 Experimental set up (a) and testing program (b)

243

244 **5. Results**

245 **5.1 Liquid limit and plastic limit**

246 Fig. 4 shows the corrected water content (fluid content)-cone penetration plots obtained from the
 247 fall cone tests. Results from Series 2, where remoulded clay was mixed with solutions between
 248 0 → 50 mS/cm, plot closer to the results obtained for the natural soil (Series 1). Results for

249 remoulded clay exposed to leaching (Series 3 and 4) are located below the line for natural soil
250 (Series 1). There, the fully leached clay defines the lower boundary. Contrasting results are
251 observed in oven-dried specimens (Series 5 and 6). Interestingly, oven-dried clay exposed to
252 controlled leaching and re-salinisation (Series 5) shows the same behaviour as the remoulded
253 clay tested in Series 3 and Series 4. However, the opposite response is observed for oven-dried
254 clay mixed with NaCl solutions (Series 6). The specimen mixed with synthetic sea water (50
255 mS/cm) defines the lower bound for oven-dried clay. The flatter slope of the plots for oven-dried
256 clay in Series 6 indicates a decrease in its water retention capacity.

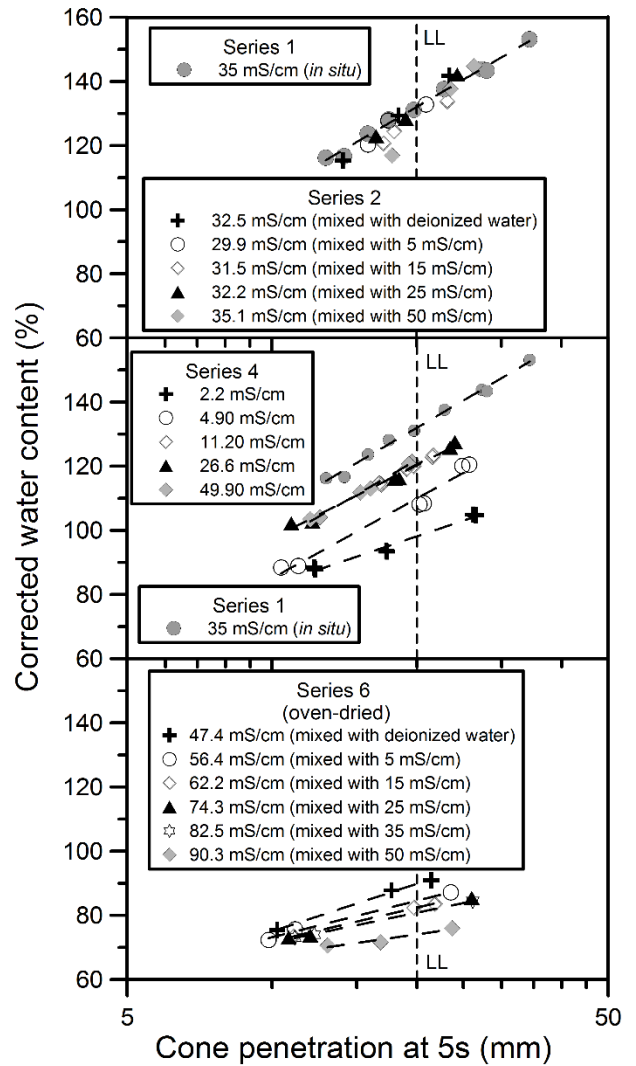
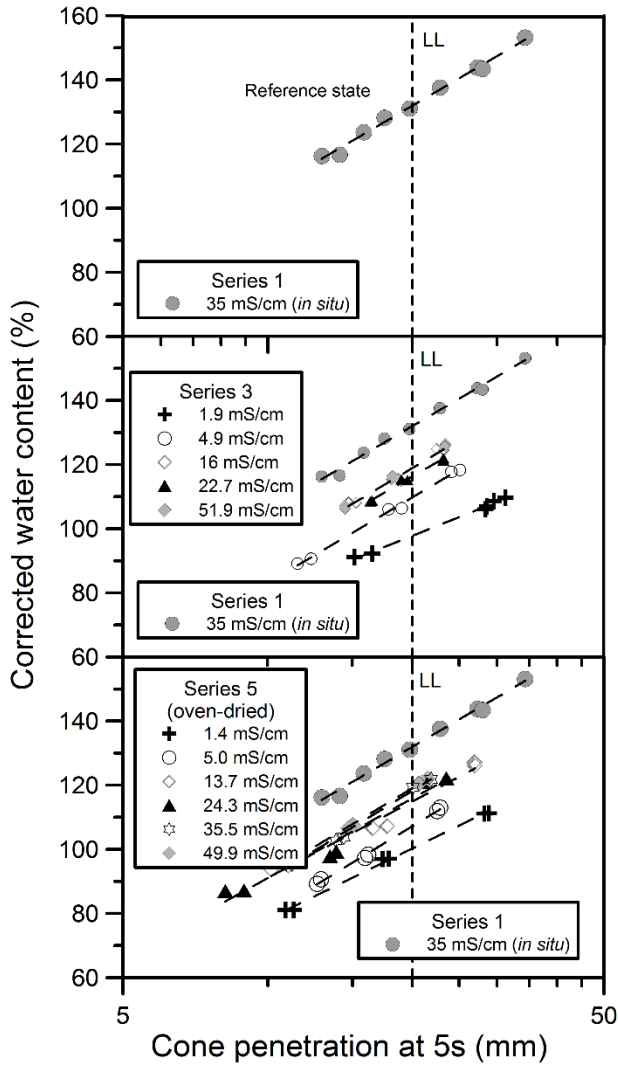
257
258 Fig. 5a shows the fluid content at LL estimated according to Eq. 1, as a function of the measured
259 EC_{fluid} . The LL for natural soil (Series 1) is 132 ± 0.6 % (average from 3 tests). The correction for
260 salt content causes an increase in LL up to values around 139%. Apart from one specimen, the
261 LL for remoulded clay mixed with solutions lower than 35 mS/cm (Series 2) shows negligible
262 changes with respect to the natural clay (133 ± 1 %). Nevertheless, LL reduces to 125% when
263 the clay is mixed with $EC_{fluid}=50$ mS/cm. Leaching applied to remoulded clay in Series 3 causes
264 the reduction of LL. However, this reduction occurs at two different rates. For remoulded clay
265 leached to $EC_{fluid} \geq 10$ mS/cm, LL decreases only by 15%. When the salinity reduces below 10
266 mS/cm, LL reduces rapidly towards values around 98% (34% less than the natural soil).
267 Salinisation of the clay to 51.7 mS/cm (sea water) also reduces LL to 120%. To evaluate the
268 reversibility of leaching on LL, results from Series 3 are compared with those from Series 4 where
269 remoulded clay was initially leached and then re-salinised to predetermined values (see Table 3).
270 The strong similarities between results from Series 3 and Series 4 indicate that leaching effects
271 on LL are largely reversible. However, it is important to note that the LL for natural soil (Series 1)
272 is not recovered. The 12% deficit observed in Fig. 5a demonstrates that full reversibility is not
273 achieved, likely due to permanent changes in soil fabric.

274
275 The effects of oven-drying on LL is evaluated by comparing results from Series 5, where oven-
276 dried clay was fully leached prior to re-salinisation, with those from Series 4. Interestingly, LL
277 measured in Series 5 is only 6% lower than that measured in Series 4. Minor modifications in soil
278 fabric seem caused by oven-drying at 60°C in cases where both remoulded clay and oven-dried
279 soil are equilibrated at the same salinity (conductivity) prior to fall cone testing. However, this is
280 not the case when salt solutions are mixed with oven-dried clay that has not been previously
281 leached (Series 6). There, the increase in salinity promotes the reduction in LL. LL drops to 90%
282 when the oven-dried clay is mixed with deionised water and it reduces further to 74% when the
283 clay is mixed with solution at 50 mS/cm. The comparison between Series 2 (natural soil) and
284 Series 6 (oven-dried soil) indicates that oven-drying strongly reduces the water retention capacity
285 of the soil (LL reduces by around 60%). Such a behaviour is explained by the densification of clay

286 aggregates, a mechanism that becomes more evident with the increase in soil salinity. Because
287 in practice soils are commonly oven-dried prior to testing and the chemistry of the pore fluid is
288 rarely evaluated, results from Series 6 are valuable to elucidate the consequences of neglecting
289 these two phenomena. The comparison of the LL obtained in Series 6 using deionised water
290 against the value for the natural soil (Series 1) represents the maximum error expected in
291 practice. An error of 42% in LL is observed. If the chemistry of the pore fluid is controlled but the
292 soil is previously oven-dried, larger reduction in LL may occur. Note that the value of LL for natural
293 soil is not recovered when the soil is previously subjected to leaching, re-salinisation or oven-
294 drying. Fig. 5a also presents the variation of fluid content at plastic limit (hereafter PL) with EC_{fluid}
295 for the six series tested in this study. Interestingly, PL is almost unaffected by changes in pore
296 fluid concentration, leaching/salinisation paths and oven-drying. An average value of 41%
297 represents all the scenarios covered in this study. The negligible sensitivity of PL to changes in
298 salinity and oven-drying is consistent with previous studies on active and non-active clays (e.g.,
299 Muir Wood, 1990).

300
301 Fig. 5b shows the path followed by all specimens within the Casagrande's chart. Remoulded clay
302 not exposed to oven-drying (Series 1 - 4) as well as oven-dried soil exposed to leaching and re-
303 salinisation (Series 5) classifies as clay of extremely high plasticity (CE). Oven-drying clay no
304 leached prior to testing (Series 6) promotes severe reduction in PI which changes the soil's
305 classification to silt of very-high plasticity (MV).

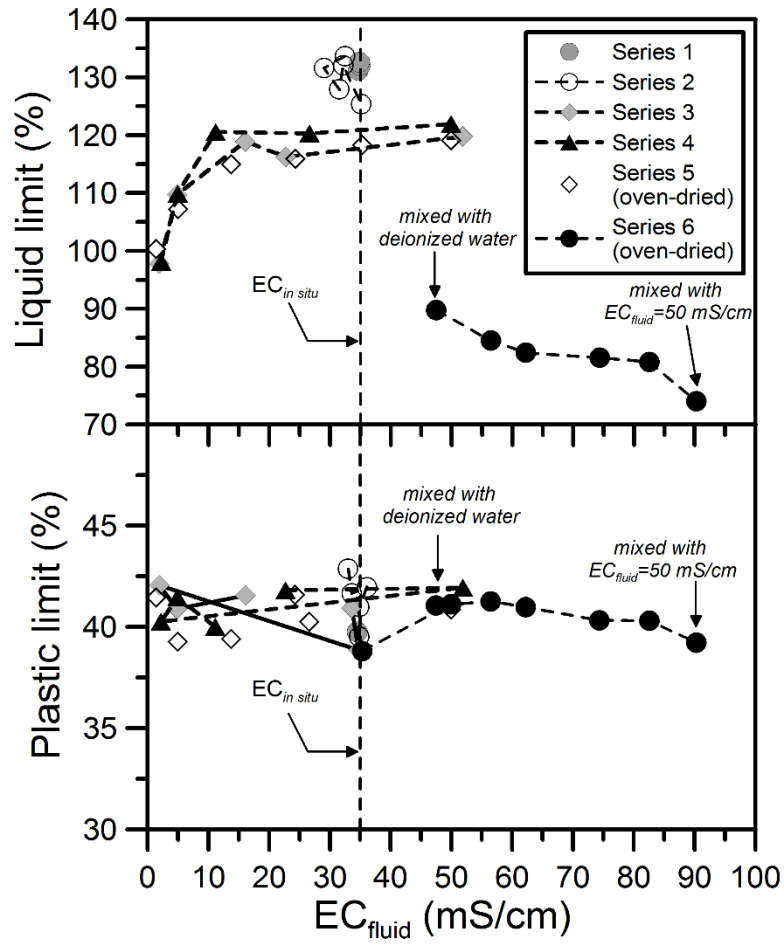
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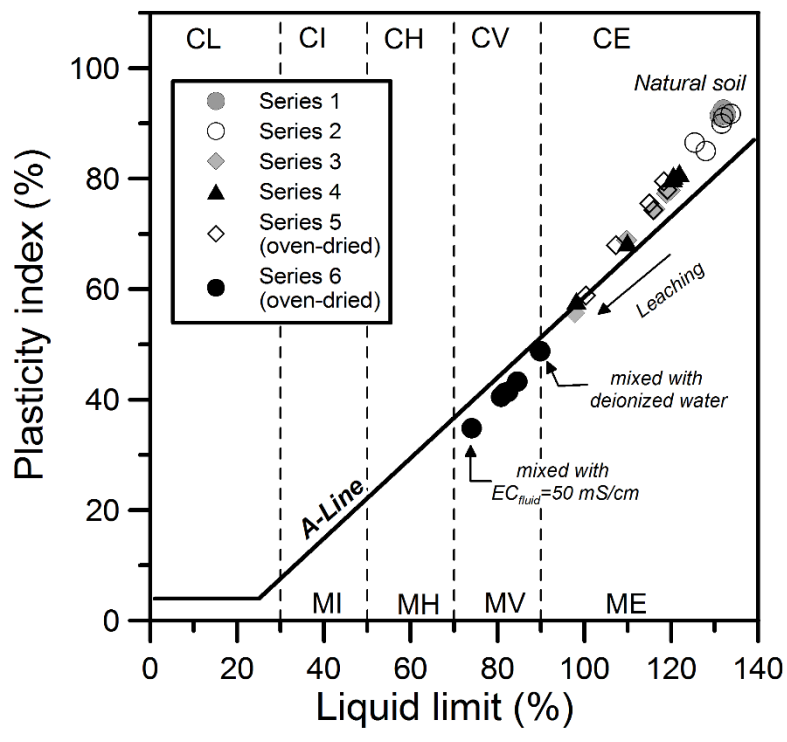
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Fig. 4 Results of the fall cone tests (Note: values of EC reported in the legend refer to EC_{fluid})



(a)



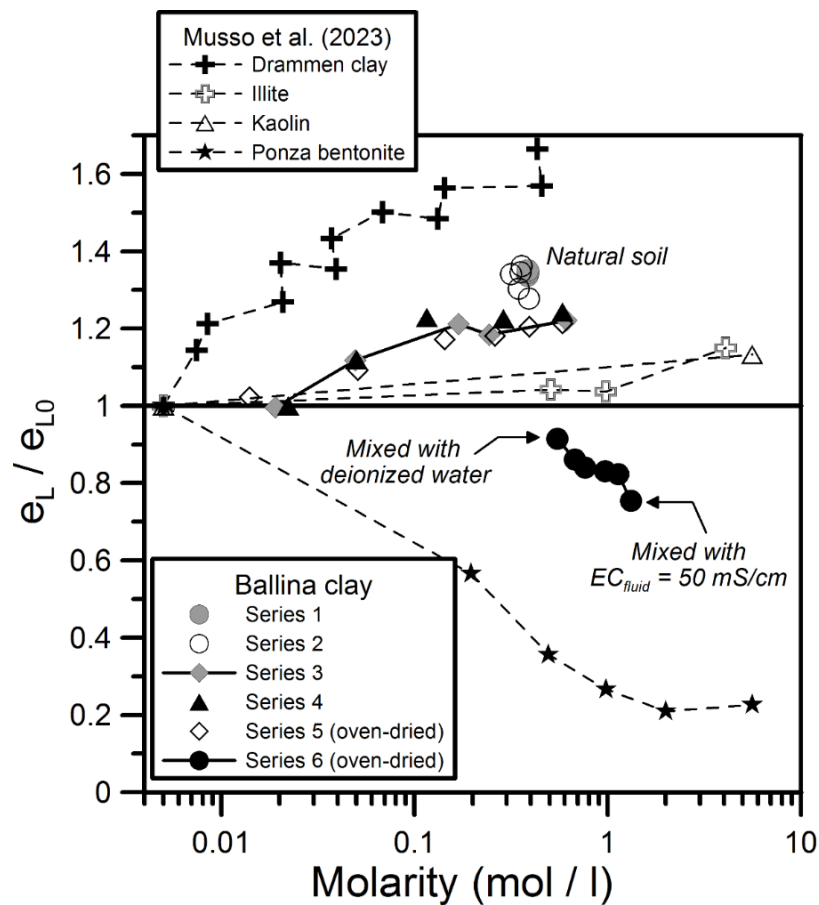
(b)

310

311

Fig. 5 Variation of liquid limit and plastic limit with EC_{fluid} (a). Soil classification (b)

312 To compare the influence of pore fluid salinity on LL for soils with different compositions it is more
 313 convenient to express LL in terms of void ratio rather than gravimetric water content (e.g., Nagaraj
 314 & Srinivasa Murthy, 1983, 1986; Nagaraj et al., 1993, 1994), provided that salinity is considered
 315 in the calculations. Fig. 6 shows the variation of void ratio at liquid limit e_L with the molarity of the
 316 NaCl solution obtained on Ballina clay specimens. These results are compared against data
 317 collected by Musso et al. (2023) for Drammen clay (Norway), Ponza bentonite (Italy), illite and
 318 illite/smectite mixtures, and kaolinite. e_L is normalised in Fig. 6 using the value for soil saturated
 319 with distilled water ($EC_{fluid} \approx 0$ mS/cm). Table 4 summarises the main properties for those soils
 320 which covers active and non-active clay minerals. e_L increases with pore fluid salinity in soils
 321 where illite and kaolinite fractions are dominant. e_L increases more than 60% in Drammen clay
 322 when mixed with sea water. In pure illite and kaolinite, the increase in e_L is negligible for molarities
 323 lower than 1 mol/l. Larger molarities cause an increase in e_L up to 15%. For Ballina clay at natural
 324 state, e_L 1.33 (i.e. 33% higher than the fully leached soil). When the clay is subjected to leaching-
 325 salinisation or oven-drying/leaching/re-salinisation, the increase in e_L with salinity is lower than
 326 20%. The oven-dried clay tested in Series 6 behaves in a similar way as soils with high proportion
 327 of active minerals (montmorillonite), i.e. e_L reduces with salinity. This contrasting behaviour is
 328 associated here to changes in soil fabric as discussed below.



329
 330 **Fig. 6** Evolution of normalized void ratio at liquid limit e_L with NaCl concentration (modified from
 331 Musso et al., 2023)

332 **Table 4.** Index properties and void ratio at liquid limit saturated with distilled water for active and
 333 non-active soils (from Musso et al., 2023)

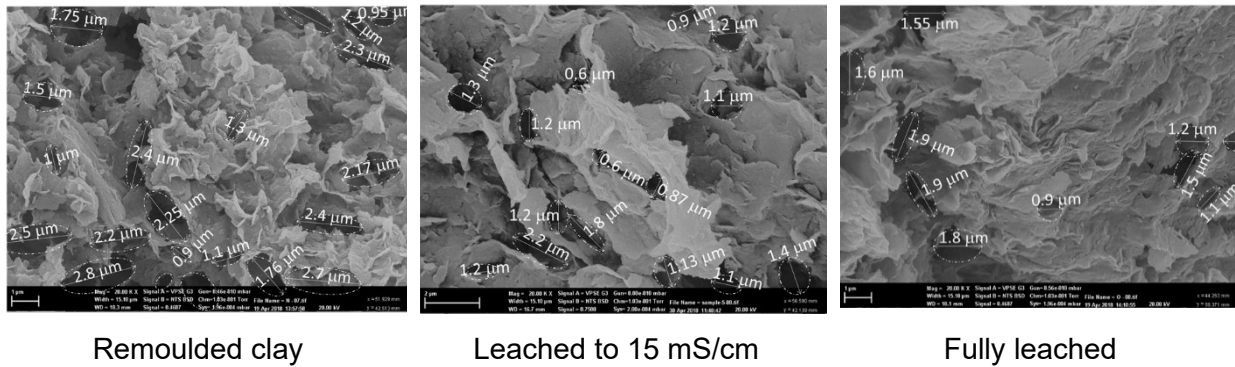
Soil	Main minerals	LL (%)	PL (%)	Activity	e_{LO}	Reference
Kaolin	Kaolinite 75-80% Illite 8-10%	50	38	0.20	1.37	Di Maio and Fenelli (1994)
Illite	Illite > 95%	83	33	0.75	2.26	Musso et al. (2023)
Drammen clay	Illite Chlorite	32	23	0.18	0.88	Torrance (1974)
Ponza bentonite	Montmorillonite 80% Kaolinite 20%	390	76	3.90	10.80	Di Maio (1996)

334
 335 **5.2 Changes in soil fabric**

336 The effects of pore fluid salinity and oven-drying on soil fabric are evaluated via Scanning Electron
 337 Microscopy (SEM) and particle size distribution (PSD) analyses. SEM tests were performed in a
 338 field emission microscope (Zeiss Sigma VPFESEM) on specimens previously subjected to the
 339 freeze-drying process (Delage et al., 2006). Fig. 7 shows SEM microphotographs (20000x) for
 340 specimens from Series 3 (i.e. remoulded clay initially leached and then re-salinized to target
 341 values). Three stages are shown in this figure: (i) remoulded (natural) state, (ii) leached to 15
 342 mS/cm, and (iii) fully leached conditions. An open fabric without clear patterns of fabric anisotropy
 343 is observed at remoulded (natural) state. Particle aggregation is also evident at this magnification.
 344 Macro pores (inter-aggregates) around 1.3 - 2.5 μm can be distinguished. Leaching to 15 mS/cm,
 345 promotes some tendency for the particles to arrange in a preferential direction although the
 346 footprint of the natural open fabric is visible. Dominant macro-pore sizes range around 2.2 μm
 347 whereas pores intra-aggregates vary between 0.7–1.3 μm . Full-leaching promotes stacking of
 348 soil particles with reduction of inter-aggregate and intra-aggregate pores. The reduction in pore
 349 size as well as the aggregation observed in Fig. 7 is consistent with the reduction in e_L with
 350 leaching shown in Fig. 6. Although SEM pictures for remoulded clay subjected to re-salinisation
 351 are not available, the behaviour of LL shown in Fig. 5a indicates that modifications in soil fabric
 352 observed in Fig. 7 may be largely reversible.

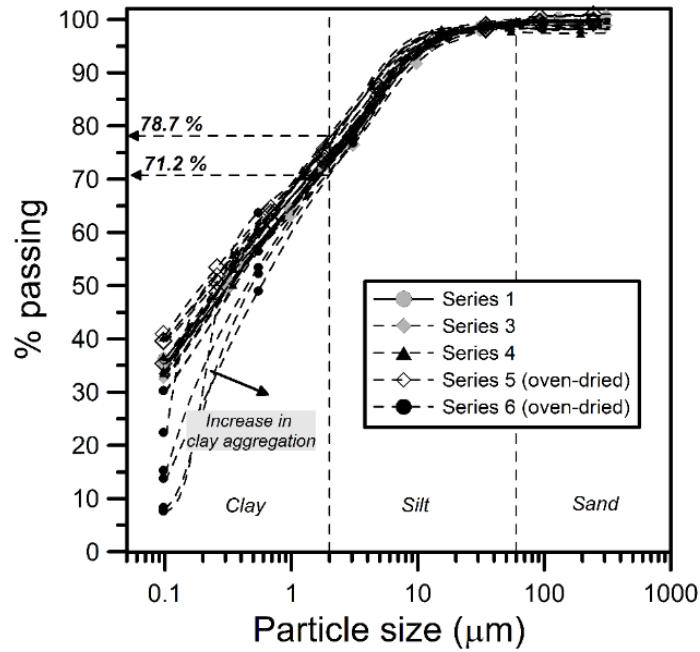
353
 354 To evaluate the influence of oven-drying on soil fabric, Fig. 8 compares the PSD curves estimated
 355 post fall cone testing. PSDs were estimated on oven-dried soil (60°C) using a particle size
 356 analyser (Sedigraph III, Micromeritics®). Whereas the procedure for sample preparation,
 357 including the mixing of the dry soil with the dispersant, may cause modifications in the natural soil
 358 fabric, its effect is assumed the same for all tested specimens. Hence, differences between PSDs
 359 should reflect the influence of previous salinity and drying paths. Fig. 8a shows changes in clay
 360 content ($\% < 2 \mu\text{m}$) between 71.2 – 78.7 % without clear link with the previous salinity and drying

361 paths. However, the inspection of the fractions below 2 μm shows an interesting trend, particularly
 362 for specimens from Series 6 where oven-dried clay was mixed with salt solutions. A progressive
 363 increase in particle size within the clay fraction is observed. This behaviour, which is indicative of
 364 clay aggregation, is clearer in Fig. 8b where fractions passing 0.1 μm are plotted against EC_{fluid} .
 365 Samples tested in Series 6 show a strong decrease in the proportion of particles lower than 0.1
 366 μm from 30% to 7% with the increase in EC_{fluid} . In other words, the proportion of very small particle
 367 sizes decreases when the soil is oven-dried due to the tendency of those particles to aggregate.
 368 This behaviour is more evident with the increase in soil salinity.

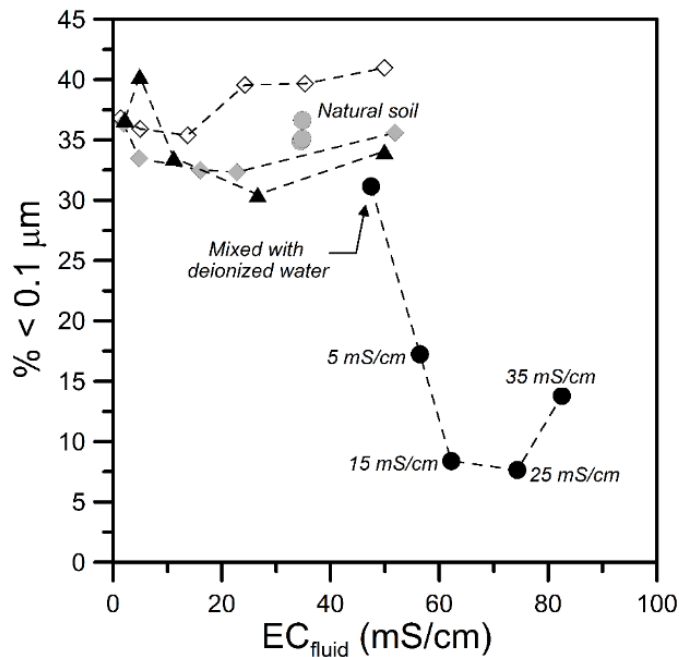


369 **Fig. 7** SEM analysis on remoulded and leached Ballina clay

370



(a)



(b)

371
372 **Fig. 8** Particle size distribution of tested specimens (a). Variation in fraction $< 0.1 \mu\text{m}$ with EC_{fluid}
373

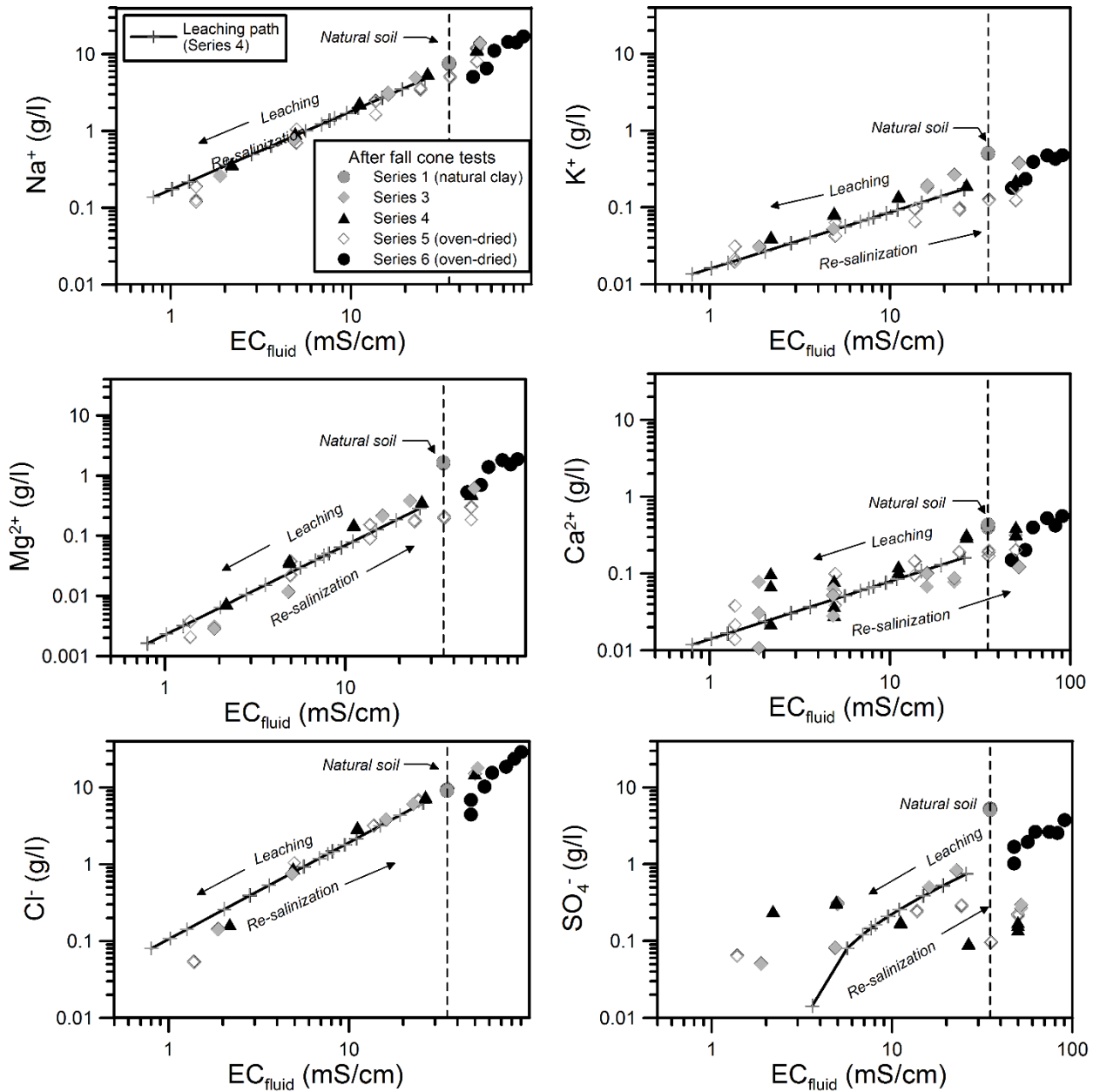
374 **6. Changes in pore fluid composition upon leaching / salinisation paths**

375 Results from chemical analyses performed to evaluate the changes in pore fluid dissolved cations
376 and anions are shown in Fig. 9. The dashed vertical line represents the pore fluid salinity for
377 natural soil, used here as reference. For the natural soil
378 (Series 1) sodium ($\text{Na}^+ \approx 7.55 \text{ g/L}$) is the major cation in the pore fluid. Other major cations present
379 are potassium ($\text{K}^+ \approx 0.51 \text{ g/L}$), magnesium ($\text{Mg}^{2+} \approx 1.63 \text{ g/L}$) and calcium ($\text{Ca}^{2+} \approx 0.42 \text{ g/L}$).
380 Chloride is the predominant anion ($\text{Cl}^- \sim 9.20 \text{ g/L}$) followed by sulphate ($\text{SO}_4^{2-} \sim 5.22 \text{ g/L}$). The

381 large proportion of soluble SO_4^- in the pore fluid at natural state (5.22 g/L, i.e. around 70% the
382 total of Na^+) agrees well with the predominance of Na^+ and the presence of K^+ (Tabatabai, 1987).
383 In Fig. 9 grey crosses represent the variation in cation and ions upon leaching for the clay used
384 in Series 4. Na^+ reduces linearly with EC_{fluid} to 0.137 g/L at the end of leaching. This represents
385 a reduction of 98.2% with respect to initial value. Monovalent and divalent cations reach values
386 of $\text{K}^+=0.014$ g/L, $\text{Mg}^{2+}=0.002$ g/L, $\text{Ca}^{2+}=0.011$ g/L. Similarly, leaching causes almost complete
387 removal of $\text{Cl}^- =0.080$ g/L (~99.1%) and $\text{SO}_4^{2-} = 0.014$ g/L (~99.7%). It is important to note that
388 the remoulded clay subjected to partial leaching in Series 3 (empty diamonds) follows the same
389 trend. Percolation of remoulded clay in Series 3 with pore fluid prepared at 50 mS/cm
390 (salinisation) increases the proportion of $\text{Na}^+=13.87$ g/L (~84 %) but also decreases $\text{Mg}^{2+}=0.615$
391 g/L, $\text{K}^+=0.38$ g/L and $\text{Ca}^{2+}=0.122$ g/L. A remarkable reduction in SO_4^{2-} (~0.287 g/L) is observed.
392 Remoulded clay subjected to re-salinisation after full leaching in Series 4 (triangles) follows the
393 same trend of the remoulded clay exposed to leaching in Series 3, thus indicating large degree
394 of reversibility during leaching/salinisation paths. Sulphates SO_4^{2-} are the exception to this trend,
395 with 96% reduction upon re-salinisation.

396
397 Interestingly, the change in cations and anions for the clay tested in Series 5 (oven-drying
398 followed by full leaching and re-salinisation) follows a similar trend as the remoulded clay from
399 Series 4 not exposed to oven-drying. Large variation in EC_{fluid} , cations and ions is observed in
400 Series 6 where oven-dried clay was mixed (no percolated) with solutions of variable salinities. In
401 this case, $\text{Na}^+ : 6.0\text{--}21.6$ g/L, $\text{K}^+ : 0.157\text{--}0.607$ g/L, $\text{Mg}^{2+} : 0.49\text{--}2.5$ g/L, $\text{Ca}^{2+} : 0.145\text{--}0.74$ g/L,
402 $\text{Cl}^- : 4.45\text{--}29$ g/L, and $\text{SO}_4^{2-} : 1.03\text{--}3.78$ g/L. Although oven-drying has the same effect on the
403 clay tested in Series 5 and Series 6, i.e. precipitation of salts initially dissolved in the pore water
404 as well as clay aggregation, those effects vanish when the oven-dried clay is percolated for
405 several weeks with deionized water (Series 5) to full leaching prior to re-salinization. Long
406 leaching times promoted particles disaggregation. The trend between salinity and liquid limit
407 found for Series 5 is thus the same of the non oven-dried specimens, consistent with what is
408 expected for non-expansive clays, where the increase in salinity promotes a face to edge to edge
409 arrangements. On the other hand, disaggregation was not obtained by simply mixing the soil
410 powder with distilled water and letting the slurry equilibrate for 48 hours (Series 6). In such case,
411 the effect of the increase in salinity appears to be only a decrease in the repulsive forces between
412 particles, which likely only promoted a decrease of the repulsive forces between particles (e.g.
413 Mitchell and Soga, 2005) and thus further aggregation.

414



415
416 **Fig. 9.** Changes in ion concentrations for each test Series
417

418 To evaluate changes in soil fabric upon leaching and salinisation **paths**, the classification
419 proposed by the U.S. Salinity Laboratory Staff (Richards et al., 1954) was adopted here. This
420 compares the sodium adsorption ratio SAR against the solution ionic strength I . The SAR refers
421 to the ratio of the amount of cations contributed to a soil by Na^+ to that of contributed by Ca^{2+} and
422 Mg^{2+} (e.g. Mitchel and Soga, 2005):
423

$$424 \text{ SAR (meq/l)}^{0.5} = \frac{\text{Na}^+}{[(\text{Ca}^{2+} + \text{Mg}^{2+})/2]^{0.5}} \quad (2)$$

425 The solution ionic strength I is calculated as the sum of the molarities of each **dissolved species**
426 multiplied by the square of its charge (Atkins et al., 2018):

$$I = \frac{1}{2} \sum_{i=1}^n c_i z_i^2 \quad (3)$$

where c_i is the molar concentration of ion i (mol/l) and z_i represents its charge.

Fig. 10 shows the variation of the SAR and LL against the solution ionic strength for Series 1, Series 3, Series 4 and Series 5. Four categories of soil fabric are defined in this 2 according to Richards et al. (1954): (1) *normal flocculated*, (2) *sodic dispersed*, (3) *saline flocculated*, and (4) *saline-sodic flocculated*. Non-saline fabrics (Zones 1 and 2) are separated from saline arrangements (Zones 3 and 4) by $I = 0.04$ mol/l ($EC_{\text{fluid}} = 4$ mS/cm). A SAR of 13 meq/l distinguishes between *normal flocculated* and *saline flocculated* fabrics (Zones 1 and 3) from *sodic dispersed* and *saline-sodic flocculated* arrangements (Zones 2 and 4). The natural soil (Series 1), which has $I = 0.55$ mol/l and SAR = 38 meq/l, is characterised by a saline-sodic flocculated fabric (Zone 4). This implies large predominance of both soluble salts and exchangeable Na^+ . As shown in Fig. 9, leaching with deionized water or saline solutions causes a rapid reduction in Mg^{2+} and Ca^{2+} in comparison to Na^+ . This explains the increase in SAR shown in Fig. 10 for $I > 0.1$ mol/l ($EC_{\text{fluid}}=10$ mS/cm) with respect to the natural soil. The *saline-sodic flocculated* fabric is preserved upon leaching or salinisation paths. Nevertheless, for $I < 0.04$ mol/l, the fabric becomes *normal flocculated* (Zone 1). The transition from *saline-sodic flocculated* to *normal flocculated* fabrics indicates that repulsion forces promoted by the large proportion of Na^+ in the natural soil are not predominant at fully leached conditions which, in turn, facilitate aggregation of soil particles. This description is consistent with the SEM results shown in Fig. 7. With less pore space for water absorption, the reduction in liquid limit observed in Fig. 5a is justified. Results for Series 5 presented in Fig. 10 shows that changes in soil fabric caused by leaching are largely reversible upon re-salinisation which is consistent with the small differences in LL shown in Fig. 5a for Series 3, Series 4 and Series 5. Despite of this, greater values of SAR are observed for Series 3, 4 and 5 at ionic strengths similar to that for the natural soil (0.55 mol/l). This is explained by the lower proportions of Ca^{2+} and Mg^{2+} measured during re-salinisation at $EC_{\text{fluid}} \geq 35$ mS/cm (see Fig. 9). Whereas the reduction in divalent cations during re-salinisation (particularly Mg^{2+}) may contribute to the reduction in LL after leaching and re-salinisation to $EC_{\text{in situ}}$ in Series 4 and Series 5, another factor to be considered is the severe decrease in SO_4^- shown in Fig. 9.

457

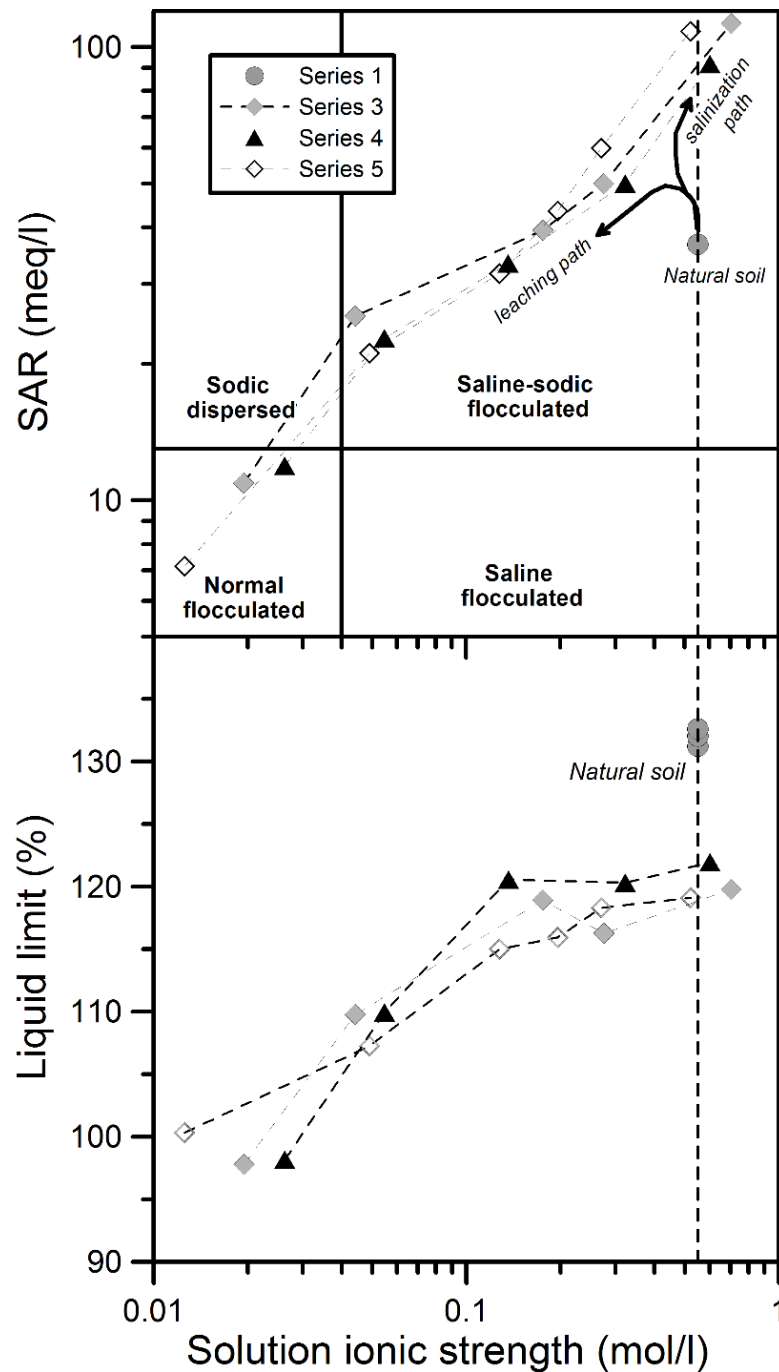


Fig. 10. Solution ionic strength vs. associated other parameters

8. Conclusions

From the experimental study described above, the following aspects deserve to be remarked:

1. leaching of salts from the natural clay, represented by a salinity around 0.7 times the salinity of sea water, causes a reduction in liquid limit up to 35%. This behaviour is in agreement with the response reported for the very sensitive low-plasticity silts from Scandinavia as well as some natural soft clays of high-plasticity from Japan.

- 468 2. leaching effects are largely reversible upon (controlled) salinisation paths. However, the
469 liquid limit for the natural clay is not achieved, attributed here to the deficit in Mg^{2+} , Ca^{2+} ,
470 K^+ (due to the composition of the synthetic solution used in the study) but also in SO_4^{4-}
471 (which seems to be related to other phenomena).
- 472 3. Leaching changes the structural arrangement of the clay from an initially saline-sodic
473 flocculated to a normal flocculated fabric. SEM analysis shows that full-leaching promotes
474 the stacking of soil particles with reduction of inter-aggregate and intra-aggregate pores..
- 475 4. Two contrasting behaviours are observed when the natural soil is oven-dried prior to
476 testing. If the salts in the oven-dried soil are fully leached then the liquid limit follows the
477 same trend observed for the remoulded clay upon salinisation. No influence of oven-
478 drying on the estimated liquid limit is observed in this case. In contrast, when the oven-
479 dried soil is mixed (no percolated) with salt solutions the measured liquid limit reduces
480 dramatically. In fact, the liquid limit decreases further with increasing the salinity of the
481 solution. The comparison of the PSD curves estimated after fall cone suggest that the
482 reduction in LL observed in oven-dried soil is due to the strong decrease in the proportion
483 of particles smaller than $0.1 \mu m$, which is explained by the tendency of the clay particles
484 to promote 'stacks'. Results for oven-drying soil mixed with deionized/tap water have
485 practical relevance because they provide the maximum expected variation (error) when
486 standard methods for the estimation Atterberg limits are adopted.
- 487 5. The plastic limit shows to be insensitive to leaching, which also agrees with available
488 literature for pure clays and natural soft soils.

489

490 The results obtained in this study show that salinity and oven-drying history might have relevant
491 effects on the index properties, on which several empirical correlations used in the engineering
492 practice rely to estimate soil parameters. Most of the correlations available in the Soil Mechanics
493 literature have been obtained by determining the plastic limit on oven-dried soils mixed with
494 distilled water. This should ensure their applicability if changes in pore water salinity are not of
495 concern. Their use should be made with some care if changes in pore water salinity are of
496 concern, as the impact on the fabric (and then on the liquid limit) imparted by oven-drying and
497 mixing might be opposite to the one expected to occur on the natural clay.

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