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

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## 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<sub>fluid</sub>: fluid content*

46 *r: salinity*

47 *EC<sub>fluid</sub>: electrical conductivity of the pore fluid*

48 *EC<sub>bulk</sub>: 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.**

120

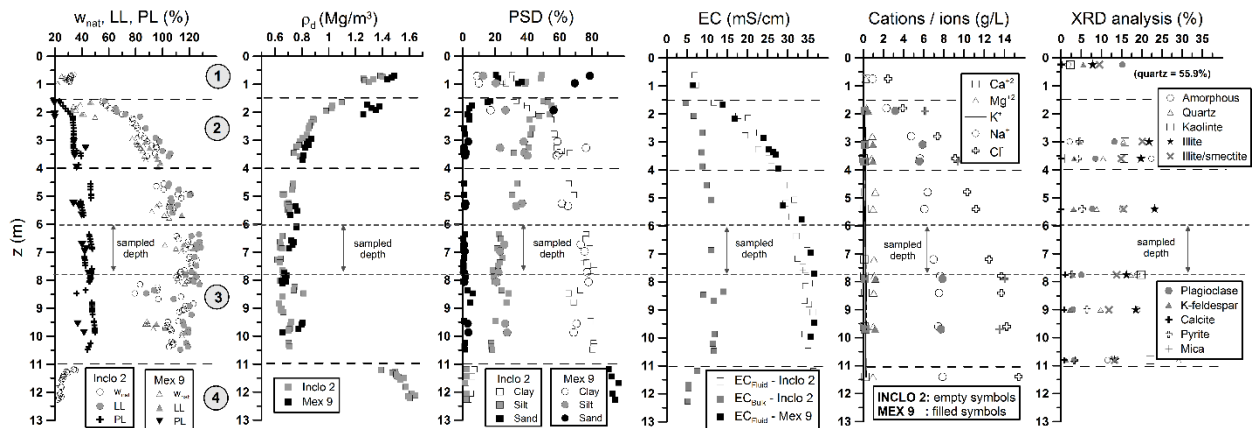
## 121 **2. Materials**

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  $1.50 \text{ Mg/m}^3$  at ground surface  
129 to  $0.70 \text{ Mg/m}^3$  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  $\text{CaCO}_3$ . The bulk electrical conductivity  
136  $\text{EC}_{\text{bulk}}$  increases with depth from 4 to 15 mS/cm whereas the pore fluid conductivity  $\text{EC}_{\text{fluid}}$   
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 <sup>++</sup> (g/l)	Mg <sup>++</sup> (g/l)	K <sup>+</sup> (g/l)	Na <sup>+</sup> (g/l)	Cl <sup>-</sup> (g/l)	Br <sup>-</sup> (g/l)	S <sub>2</sub> <sup>-</sup> (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

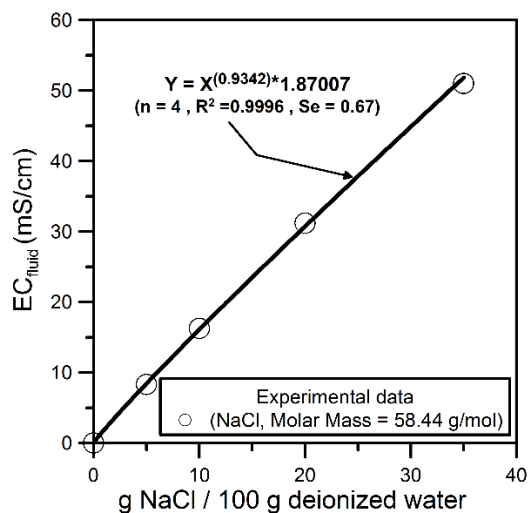
### 160 3. Experimental procedures

#### 161 3.1 Fluid content

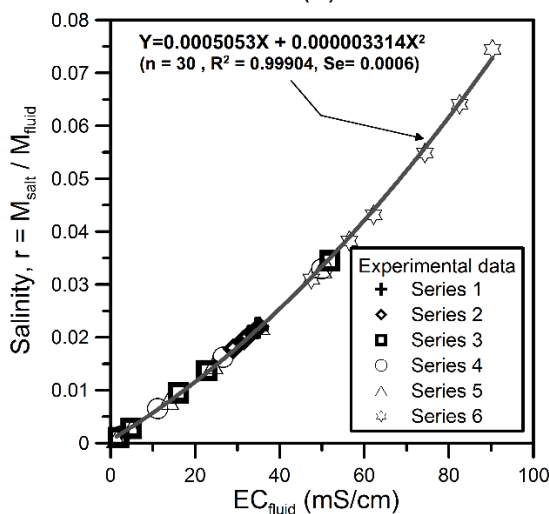
162 In soils that contains salts dissolved in the pore fluid, a correction to the gravimetric water content  
 163 must be applied (e.g. Noorany, 1984; Frydman et al., 2008). The corrected water content (or fluid  
 164 content) is then expressed as (Noorany, 1984):

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

166 where  $w_{\text{fluid}}$  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  $r = M_{\text{salt}} / M_{\text{fluid}}$ . 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  $EC_{\text{fluid}}$   
 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.



(a)



(b)

**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  $EC_{\text{fluid}}$ . 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  $EC_{\text{fluid}}$  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  $30^\circ$  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,  $EC_{\text{bulk}}$ ,  $EC_{\text{fluid}}$  and pH were measured as follows.  $EC_{\text{bulk}}$  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  $15 \text{ cm}^3$  of pore fluid required to measure  $EC_{\text{fluid}}$ . 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



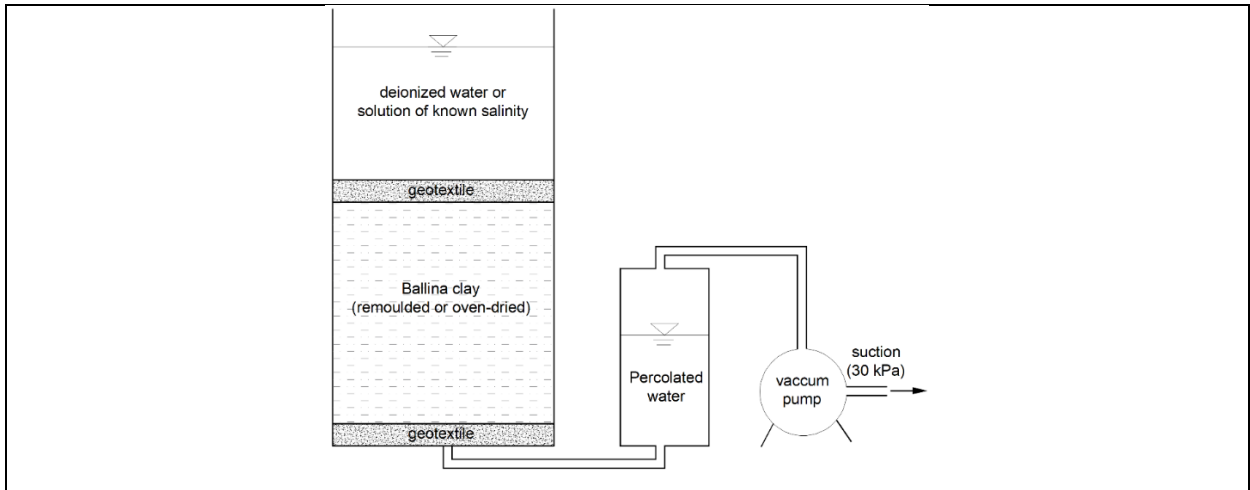
218 'natural' conditions, a synthetic solution prepared at *in situ* electrical conductivity ( $EC_{fluid}=35$   
 219 mS/cm) was added to the remoulded clay at its natural water content. The mixture was then  
 220 mixed, without preliminarily drying the soil. In Series 2, natural clay was mixed with 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

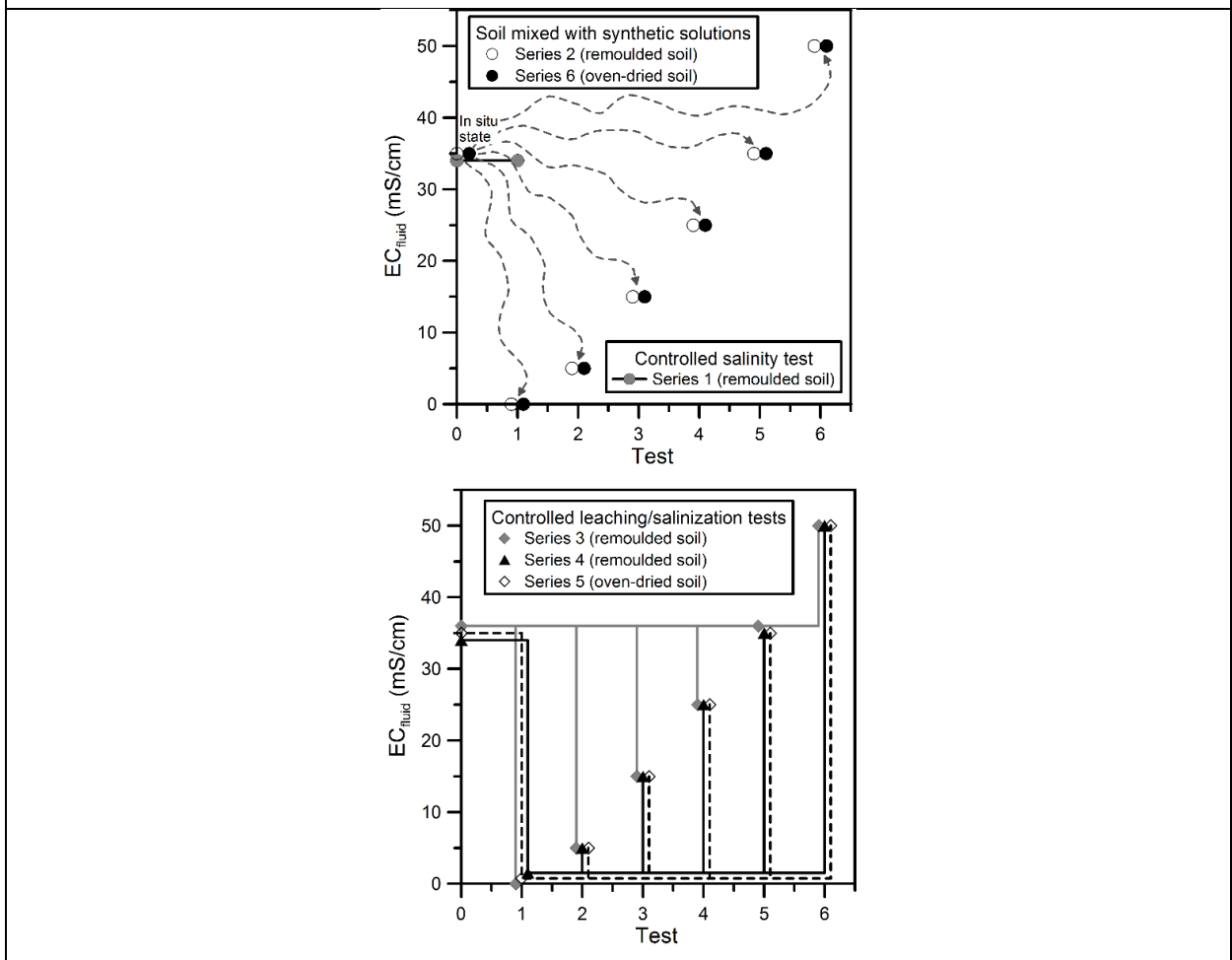
Series	Soil	Comments
1	Remoulded (natural water content)	Clay mixed with $EC_{fluid} = 35^*$ mS/cm
2		Clay mixed with $EC_{fluid}: 0, 5, 15, 25$ and 50 mS/cm
3		Clay leached to 0, 5, 15 and 25 mS/cm. Salinisation to 50 mS/cm
4		Clay fully leached followed by re-salinisation to 5, 15, 25, 35 and 50 mS/cm
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.

240  
 241  
 242



(a)



(b)

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

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.

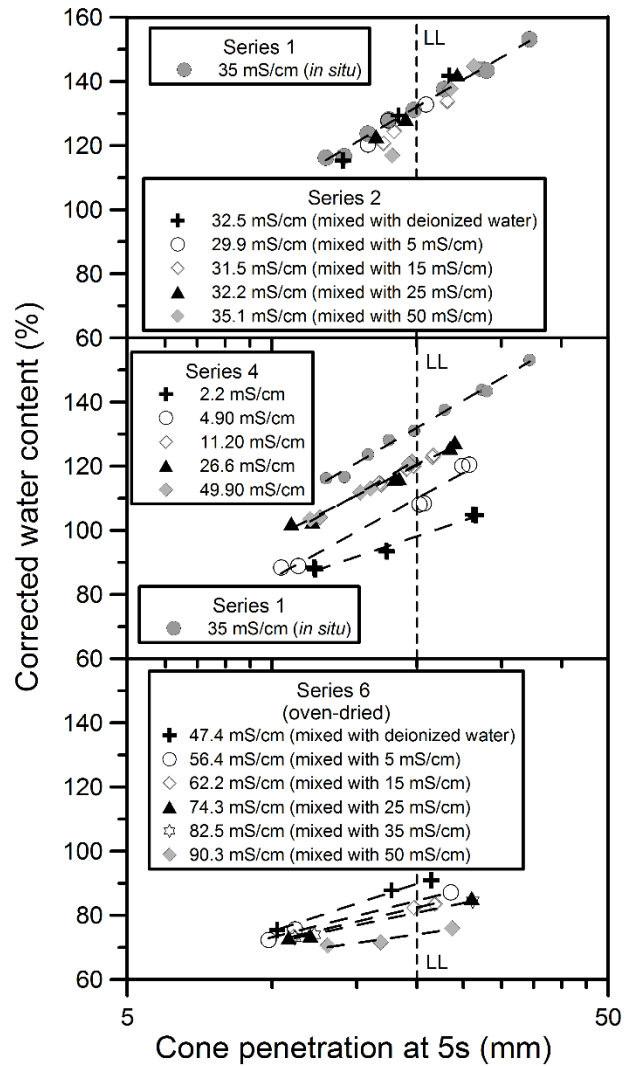
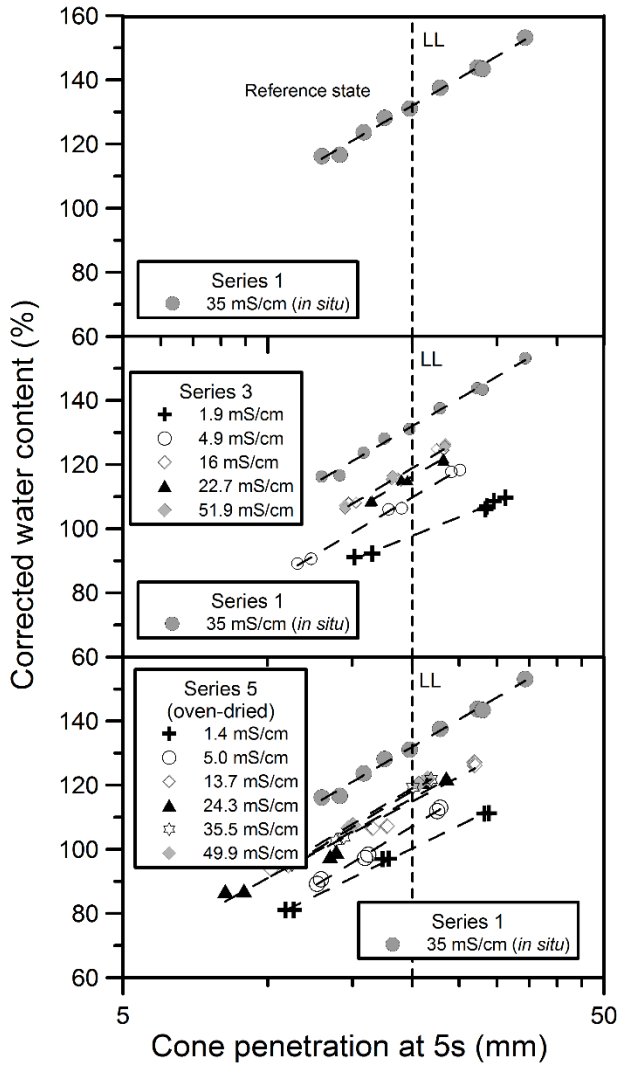
257  
258 Fig. 5a shows the fluid content at LL estimated according to Eq. 1, as a function of the measured  
259  $EC_{fluid}$ . The LL for natural soil (Series 1) is  $132 \pm 0.6 \%$  (average from 3 tests). The correction for  
260 salt content causes an increase in LL up to values around 139%. Apart 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 ( $133 \pm 1 \%$ ). 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).

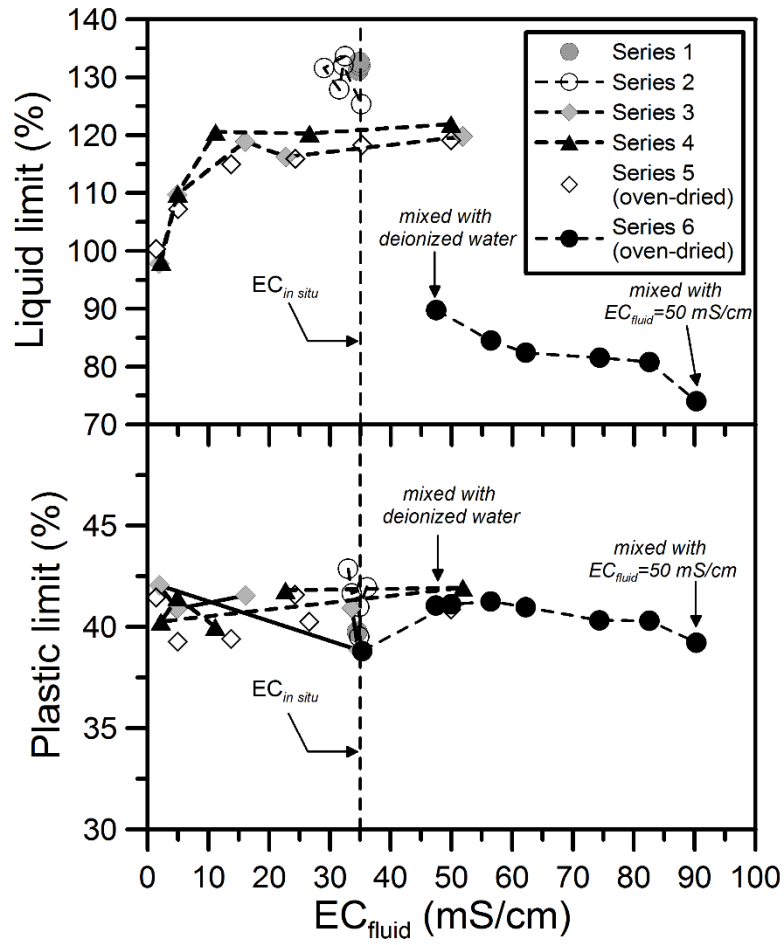
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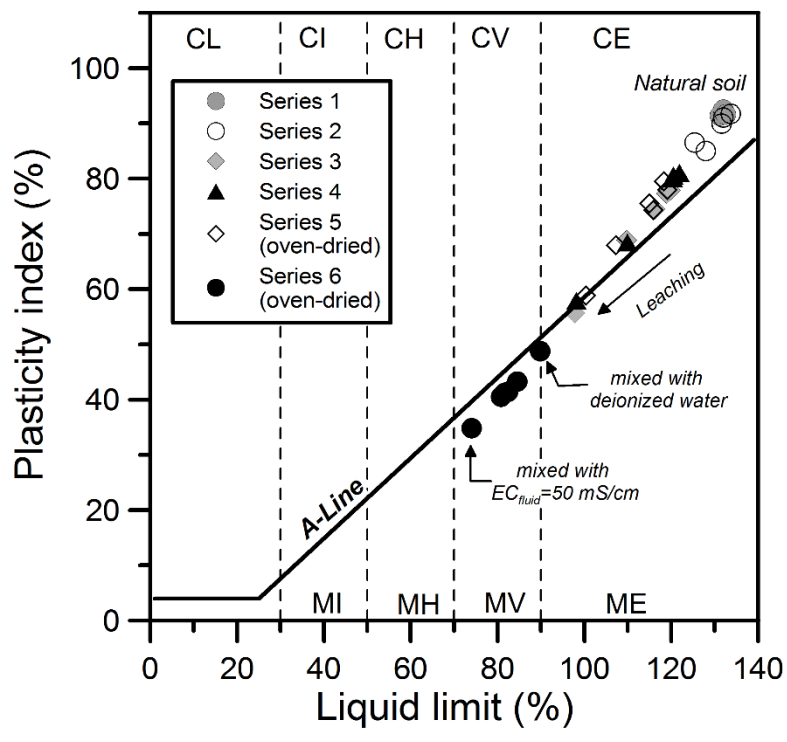
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**Fig. 4** Results of the fall cone tests (Note: values of EC reported in the legend refer to  $EC_{fluid}$ )



(a)



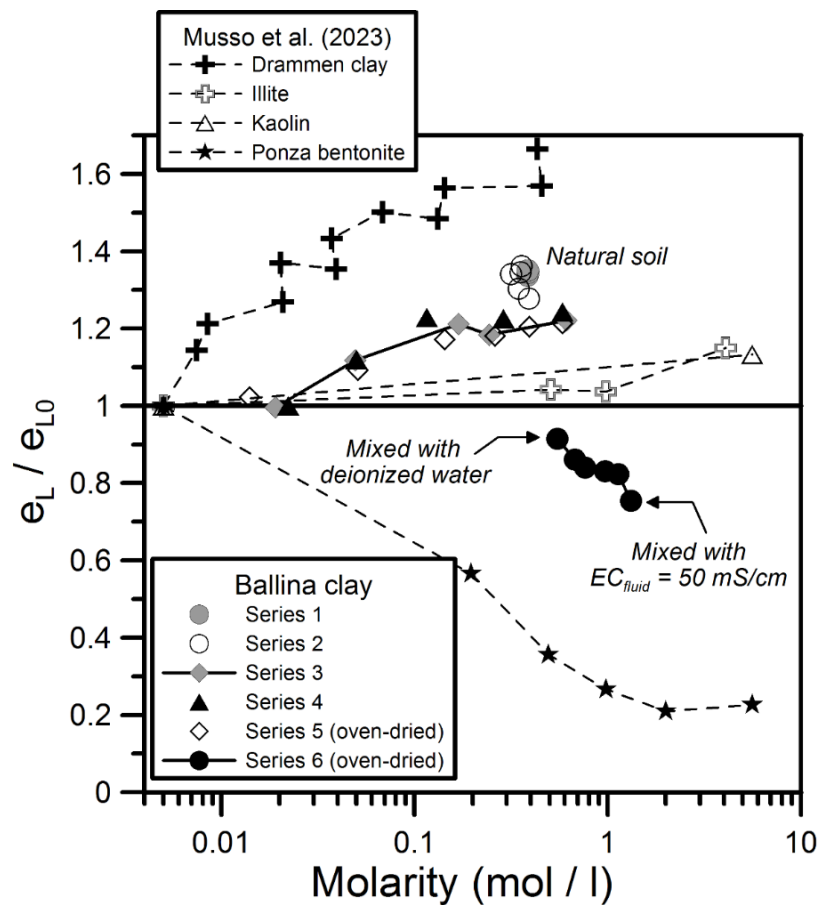
(b)

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)

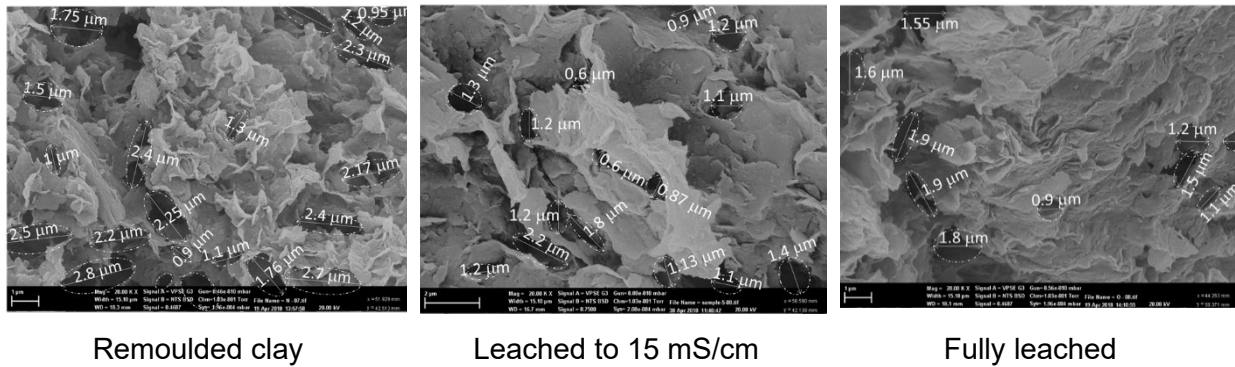
334  
 335 **5.2 Changes in soil fabric**

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  $\mu\text{m}$  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  $\mu\text{m}$   
 347 whereas pores intra-aggregates vary between 0.7–1.3  $\mu\text{m}$ . 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

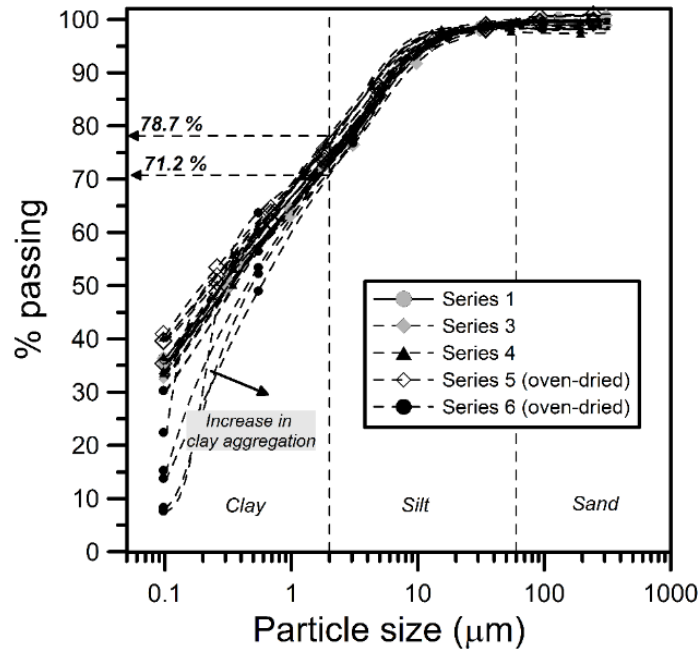


361 paths. However, the inspection of the fractions below 2  $\mu\text{m}$  shows an interesting trend, particularly  
 362 for specimens from Series 6 where oven-dried clay was mixed with salt solutions. A progressive  
 363 increase in particle size within the clay fraction is observed. This behaviour, which is indicative of  
 364 clay aggregation, is clearer in Fig. 8b where fractions passing 0.1  $\mu\text{m}$  are plotted against  $\text{EC}_{\text{fluid}}$ .  
 365 Samples tested in Series 6 show a strong decrease in the proportion of particles lower than 0.1  
 366  $\mu\text{m}$  from 30% to 7% with the increase in  $\text{EC}_{\text{fluid}}$ . 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.

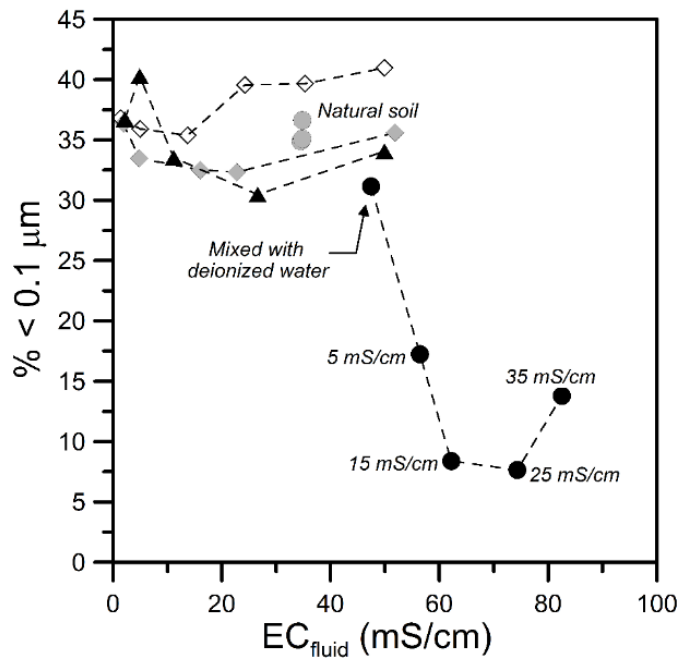


369 **Fig. 7** SEM analysis on remoulded and leached Ballina clay

370



(a)



(b)

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

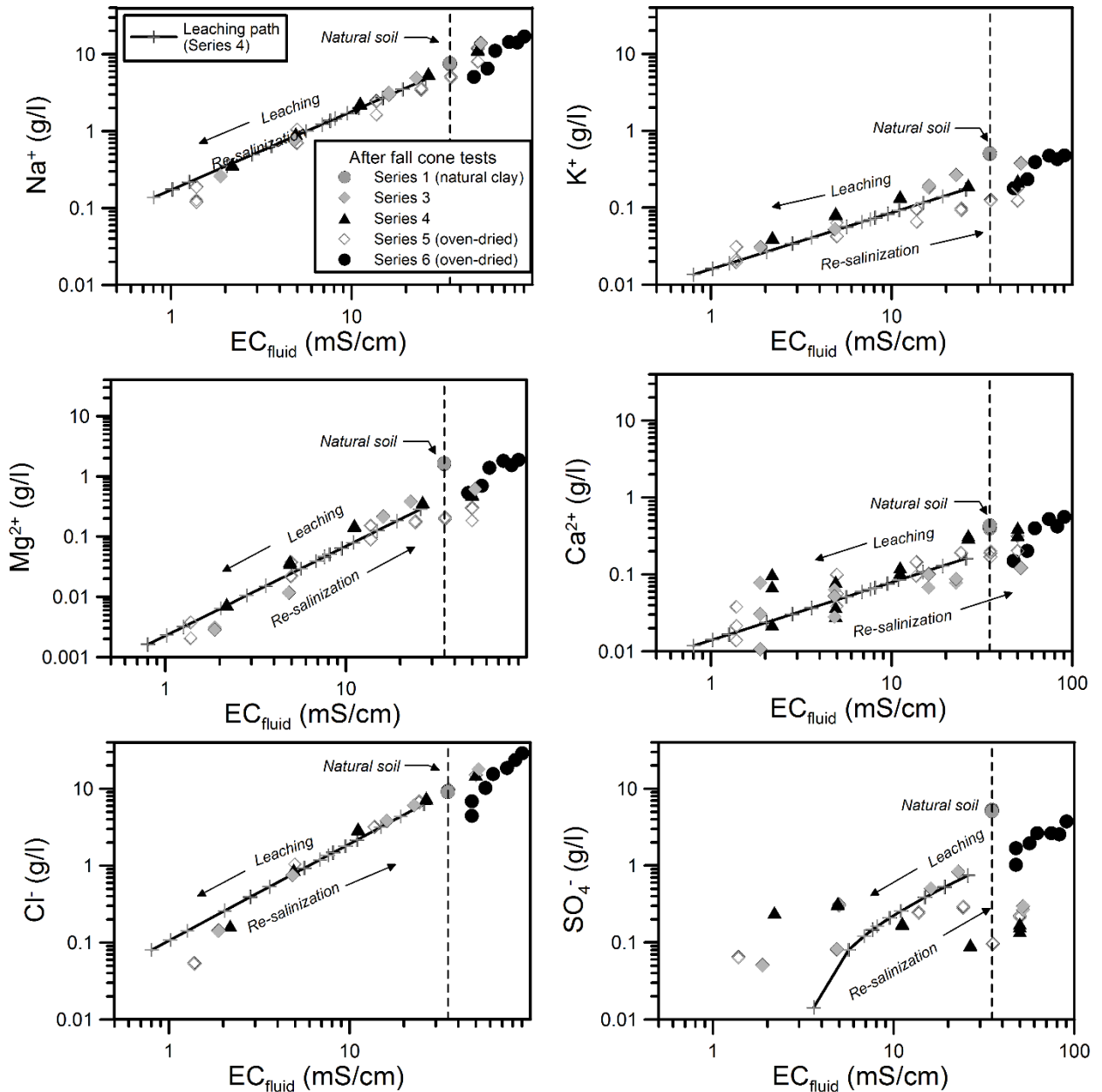
374 **6. Changes in pore fluid composition upon leaching / salinisation paths**

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  $\text{SO}_4^-$  in the pore fluid at natural state (5.22 g/L, i.e. around 70% the  
382 total of  $\text{Na}^+$ ) agrees well with the predominance of  $\text{Na}^+$  and the presence of  $\text{K}^+$  (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.  $\text{Na}^+$  reduces linearly with  $\text{EC}_{\text{fluid}}$  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  $\text{SO}_4^{2-}$  (~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  $\text{SO}_4^{2-}$  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  $\text{EC}_{\text{fluid}}$ , 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  $\text{Na}^+$  to that of contributed by  $\text{Ca}^{2+}$  and  
422  $\text{Mg}^{2+}$  (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):

427 
$$I = \frac{1}{2} \sum_{i=1}^n c_i z_i^2 \quad (3)$$

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

429

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

457

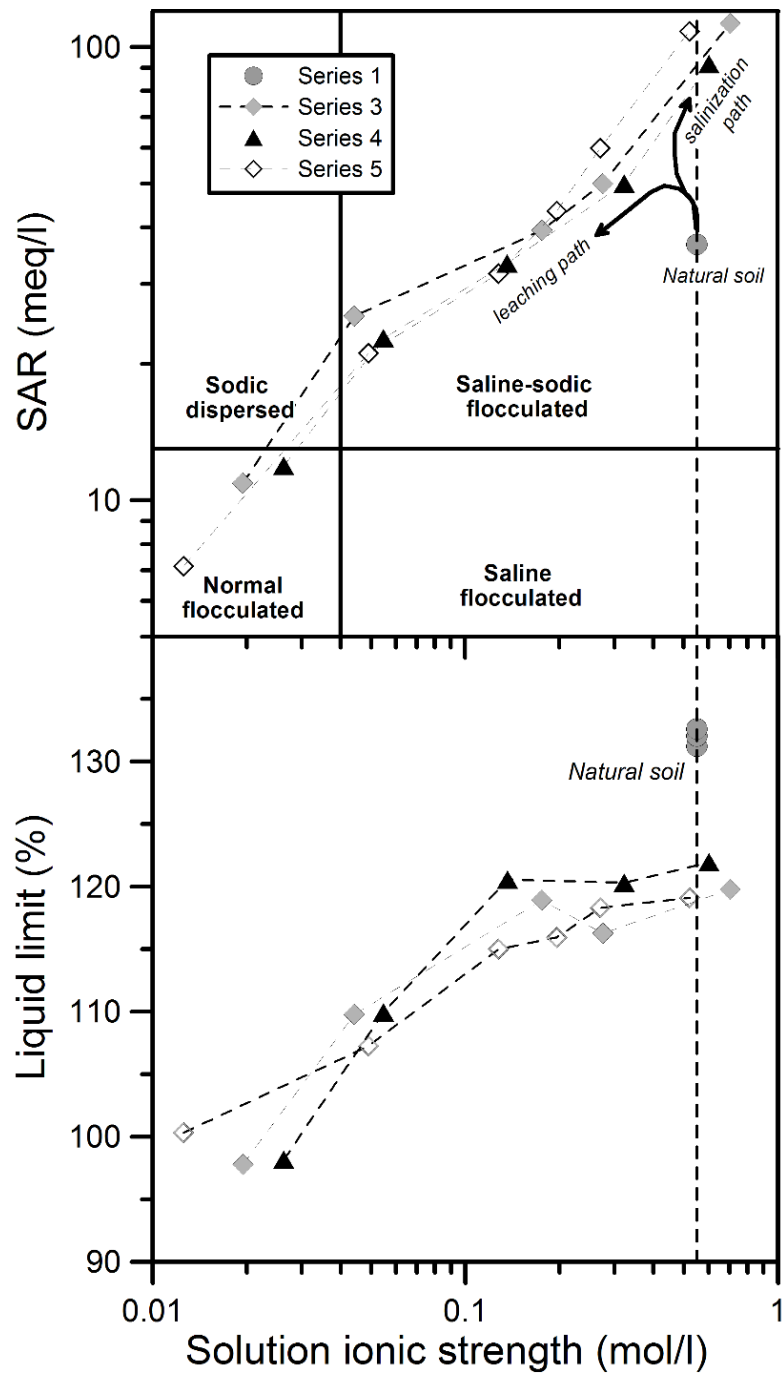


Fig. 10. Solution ionic strength vs. associated other parameters

## 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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