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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.

## Notation

*LL: liquid lmit*
*PL: plastic limit*
*PI: plasticity index*
*PSD: particle size distribution*
*w: gravimetric water content*
$w_{fluid}$*: fluid content*
*r: salinity*
$EC_{fluid}$*: electrical conductivity of the pore fluid*
$EC_{bulk}$*: electrical conductivity of bulk’s soil*
*SEM: Scanning Electron Microscopy*
*SAR: solution adsorption ratio*
*I: solution ionic strength*

## 1. Introduction

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

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

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

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

## 2. Materials

The soil tested is Ballina clay, an estuarine soft clay encountered at the National Soft Soil Testing Facility NFTF (Kelly et al., 2017; Pineda et al., 2019) located in the town of Ballina, northern New South Wales, Australia. Detailed *in situ* and laboratory characterisation studies were carried out after the establishment of the NFTF in 2013 (Pineda et al., 2016; Kelly et al., 2017). As observed in Fig. 1, the natural water content increases with depth from 20% ($z \approx 1$ m) up to 120% ($z > 7$ m). The liquid limit LL is typically 10-15% higher than the natural water content whereas the plastic limit PL ranges between 20-53%. Natural dry density reduces from 1.50 Mg/m$^3$ at ground surface to 0.70 Mg/m$^3$ at 4.5 m depth. However, minor changes are noticed between 3 m and 11 m. Despite the differences in sand content (column PSD, Particle Size Distribution) observed above 2 m, the clay fraction is predominant up to 11 m depth, with maximum values around 82%. Soils at Ballina site are composed of amorphous minerals, kaolinite, illite, quartz, interstratified illite/smectite, plagioclase, pyrite, K-feldspar, mica and calcite. Sodium (0.93–8.23 g/L) and chloride (2.5–15.4 g/L) are the most abundant cations and anions in the pore fluid whereas the alkalinity of the pore water lies between 0.6-2.2 g/L of $CaCO_3$. The bulk electrical conductivity $EC_{bulk}$ increases with depth from 4 to 15 mS/cm whereas the pore fluid conductivity $EC_{fluid}$ increases from 7 up to 36 mS/cm (average value below 5 m). This corresponds to salt concentrations between 4–23.5 g/L. The presence of salts during soil deposition promoted a porous fabric composed by aggregates separated by large voids. Four soil layers can be identified in the upper 13 m (see Fig. 1) (Pineda et al., 2019). Layer 1 is a silty-sand alluvial crust. Layers 2 and 3 comprises the upper and lower estuarine silty clay (hereafter Ballina clay). Layer 4 is the silty-sand transition layer which overlies a sand layer and a deeper stiff clay layer.

The soil used in the experiments described below was obtained from high-quality block samples retrieved with the Sherbrooke sampler between 6.0-7.8 m depth. At this level, natural water content ranges between 110–122% whereas clay, silt and sand contents vary between 79-81%, 18-20% and 0-2%, respectively. The soil classifies as clay of extremely high plasticity with soil activity around 1. The organic content at this depth ranges around 1.8%. Tables 1 and 2 summarise the mineralogical composition and the geochemical results reported by Pineda et al. (2016).

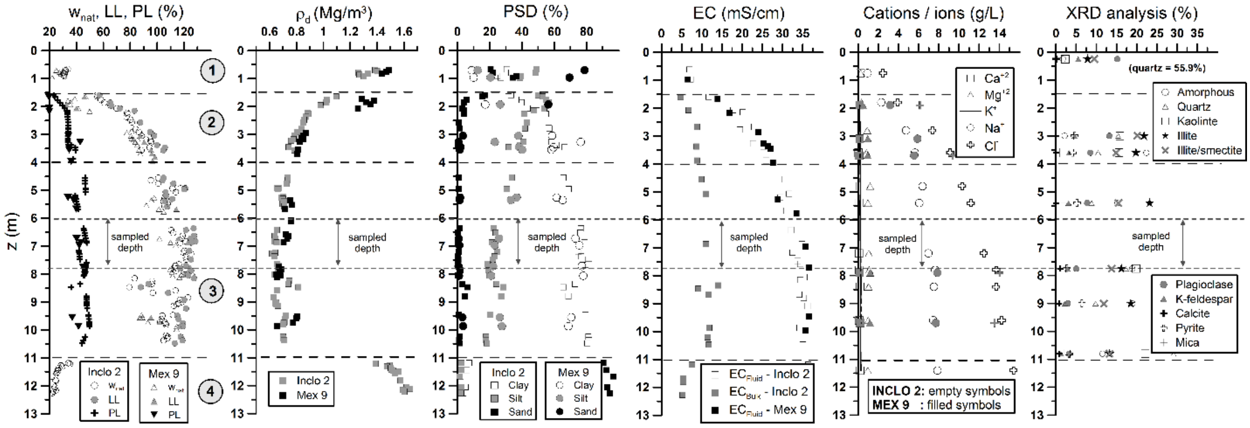


**Fig. 1.** Index properties and geochemical composition of soil deposits at Ballina site (modified from Pineda et al., 2016)

**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 |

**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 |

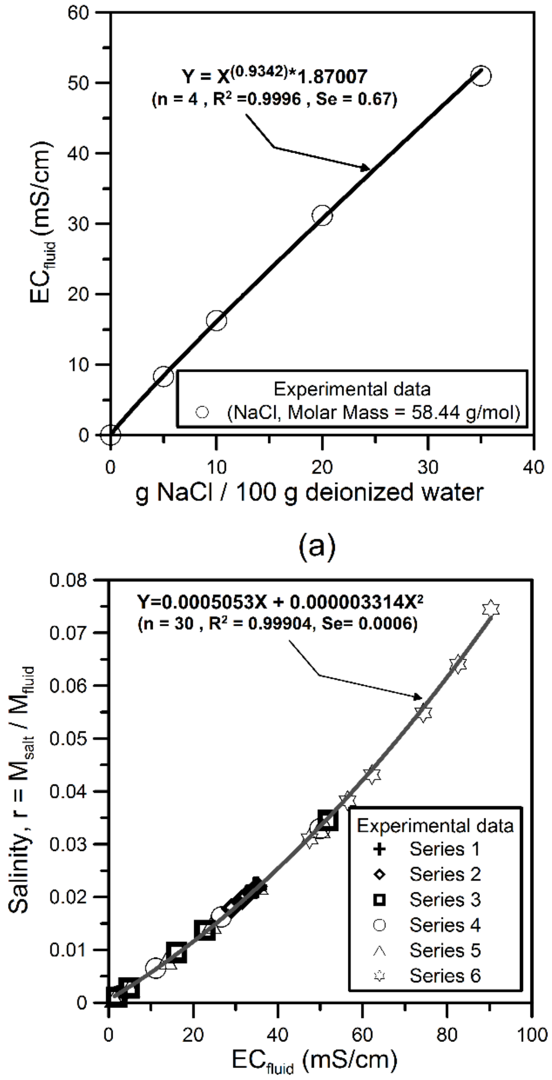
## 3. Experimental procedures

### 3.1 Fluid content

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

$$w_{fluid} = \frac{w}{(1-r)-r*w} \qquad (1)$$

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



(a)

(b)

**Fig. 2** Salinity and pore fluid conductivity

### 3.2 Leaching / salinisation tests

Leaching and salinisation paths were applied to remoulded clay (at natural water content) and oven-dried material (initially mixed with deionized water or saline solutions up to 1.5 times its natural water content) before the estimation of Atterberg limits. Saline solutions were percolated through soil samples by applying a vacuum pressure of 30 kPa, using the experimental setup shown in Fig. 3a. Salt solutions, prepared using the relationships of Fig. 2b, were periodically renewed to avoid changes in the target $EC_{fluid}$. Percolation continued until equilibrium between the electrical conductivity of the reference solution and the percolated fluid was achieved ($EC_{solution} \approx EC_{fluid}$): up to 15 weeks were required to achieve fully leached conditions. The mass of the percolated fluid was measured during this process and used in the geochemical analyses presented below. Each sample was prepared as follows. After placing remoulded clay or clay powder inside PVC containers, gently vibration in a shaking table for 5 minutes allowed to remove the trapped air from the soil mass. Two layers of geotextile were used as porous filters at top and bottom ends. The salt solution was then poured on top of the specimen, whereas the bottom drainage was connected to the vacuum line. Periodic measurements of $EC_{fluid}$ and pH of the percolated fluid were made using an Orion EC (013005MB DuraProbe) electrode and an Orion pH (9165BN) electrode, respectively.

### 3.3 Atterberg limits tests

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

### 3.4 Ion chromatography analysis

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

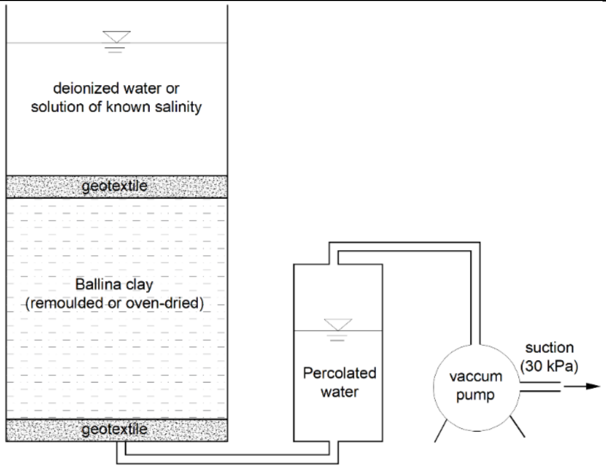
## 4. Experimental program

The influence of pore fluid salinity and oven-drying on soil plasticity was evaluated through six test series summarized in Table 3 and Fig. 3b. Clay specimens were either mixed or percolated with different solutions before evaluating soil's plasticity. In Series 1, which represents the 'natural' conditions, a synthetic solution prepared at *in situ* electrical conductivity ($EC_{fluid}$=35 mS/cm) was added to the remoulded clay at its natural water content. The mixture was then mixed, without preliminarily drying the soil. In Series 2, natural clay was mixed with five solutions with conductivities ranging between 0 (deionised water) to 50 mS/cm (sea water). Although changes in pore fluid salinity occurred in Series 2, controlled leaching/salinisation paths were not imposed. An equilibration period of 48 h was allowed to the clay-fluid mixture before estimating LL and PL. Series 3 involved controlled leaching and desalinisation paths to achieve target conductivities. Remoulded specimens were leached from 35 mS/cm to 25 mS/cm, 15 mS/cm, 5 mS and 0 mS/cm. Salinisation from 35 mS/cm to 50 mS/cm was also applied in Series 3. To check the reversibility of the leaching-salinisation process on soil plasticity, in Series 4 a large volume of remoulded clay was initially fully leached and then sub-samples were subjected to controlled (re)salinisation to 5, 15, 25 and 50 mS/cm. Series 5 and Series 6 tested oven-dried clay. To do so, remoulded clay was first oven-dried at 60°C for 7 days. A large amount of clay powder was then percolated with deionised water in Series 5 until full leaching was achieved. Afterwards, the leached clay was separated in small containers and exposed to controlled re-salinisation to 5, 15, 25, 35 and 50 mS/cm. In Series 6, oven-dried clay was mixed (i.e. controlled leaching-salinisation not intended) with different synthetic solutions ($EC_{fluid}$: 0 → 50 mS/cm). In all cases, an equalisation of 48h was allowed to the clay-fluid mixture before estimating the Atterberg limits. Chemical tests performed on leachate fluid provided the basis for understanding the chemical changes occurring during the leaching and salinisation processes described below.

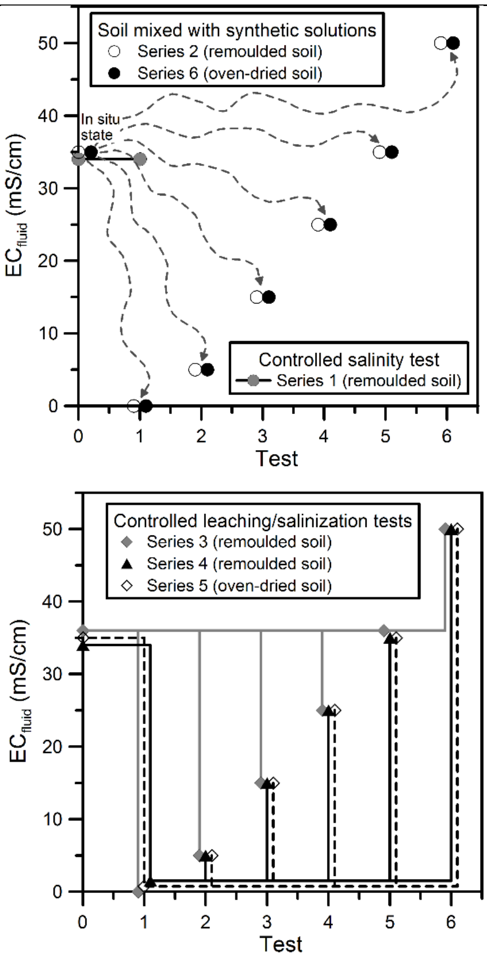
**Table 3** Test series carried out to evaluate salinity and drying effects on soil plasticity

| 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.*



(a)



(b)

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

## 5. Results

### 5.1 Liquid limit and plastic limit

Fig. 4 shows the corrected water content (fluid content)-cone penetration plots obtained from the fall cone tests. Results from Series 2, where remoulded clay was mixed with solutions between 0 → 50 mS/cm, plot closer to the results obtained for the natural soil (Series 1). Results for remoulded clay exposed to leaching (Series 3 and 4) are located below the line for natural soil (Series 1). There, the fully leached clay defines the lower boundary. Contrasting results are observed in oven-dried specimens (Series 5 and 6). Interestingly, oven-dried clay exposed to controlled leaching and re-salinisation (Series 5) shows the same behaviour as the remoulded clay tested in Series 3 and Series 4. However, the opposite response is observed for oven-dried clay mixed with NaCl solutions (Series 6). The specimen mixed with synthetic sea water (50 mS/cm) defines the lower bound for oven-dried clay. The flatter slope of the plots for oven-dried clay in Series 6 indicates a decrease in its water retention capacity.

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

The effects of oven-drying on LL is evaluated by comparing results from Series 5, where oven-dried clay was fully leached prior to re-salinisation, with those from Series 4. Interestingly, LL measured in Series 5 is only 6% lower than that measured in Series 4. Minor modifications in soil fabric seem caused by oven-drying at 60°C in cases where both remoulded clay and oven-dried soil are equilibrated at the same salinity (conductivity) prior to fall cone testing. However, this is not the case when salt solutions are mixed with oven-dried clay that has not been previously leached (Series 6). There, the increase in salinity promotes the reduction in LL. LL drops to 90% when the oven-dried clay is mixed with deionised water and it reduces further to 74% when the clay is mixed with solution at 50 mS/cm. The comparison between Series 2 (natural soil) and Series 6 (oven-dried soil) indicates that oven-drying strongly reduces the water retention capacity of the soil (LL reduces by around 60%). Such a behaviour is explained by the densification of clay aggregates, a mechanism that becomes more evident with the increase in soil salinity. Because in practice soils are commonly oven-dried prior to testing and the chemistry of the pore fluid is rarely evaluated, results from Series 6 are valuable to elucidate the consequences of neglecting these two phenomena. The comparison of the LL obtained in Series 6 using deionised water against the value for the natural soil (Series 1) represents the maximum error expected in practice. An error of 42% in LL is observed. If the chemistry of the pore fluid is controlled but the soil is previously oven-dried, larger reduction in LL may occur. Note that the value of LL for natural soil is not recovered when the soil is previously subjected to leaching, re-salinisation or oven-drying. Fig. 5a also presents the variation of fluid content at plastic limit (hereafter PL) with $EC_{fluid}$ for the six series tested in this study. Interestingly, PL is almost unaffected by changes in pore fluid concentration, leaching/salinisation paths and oven-drying. An average value of 41% represents all the scenarios covered in this study. The negligible sensitivity of PL to changes in salinity and oven-drying is consistent with previous studies on active and non-active clays (e.g., Muir Wood, 1990).

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

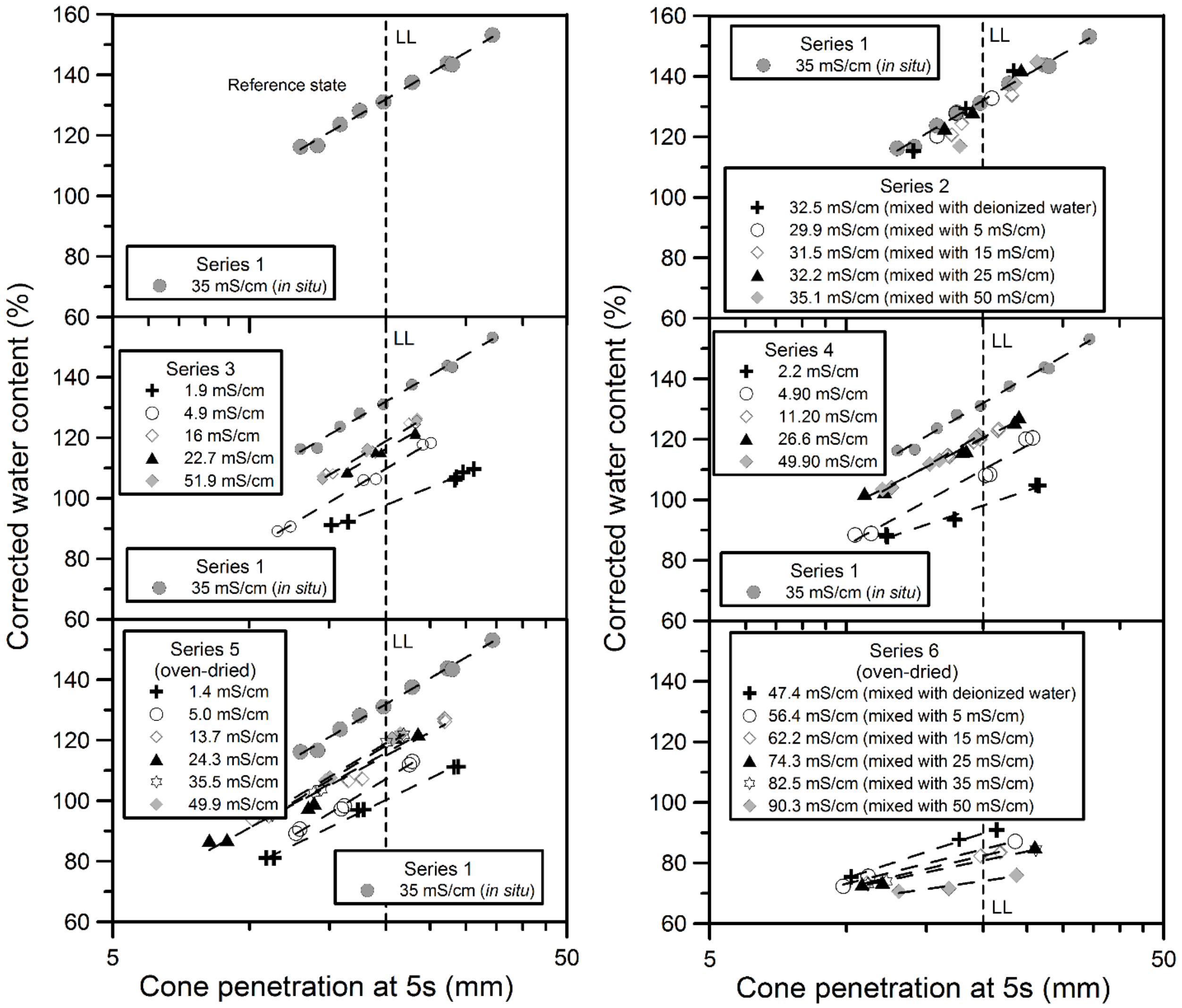
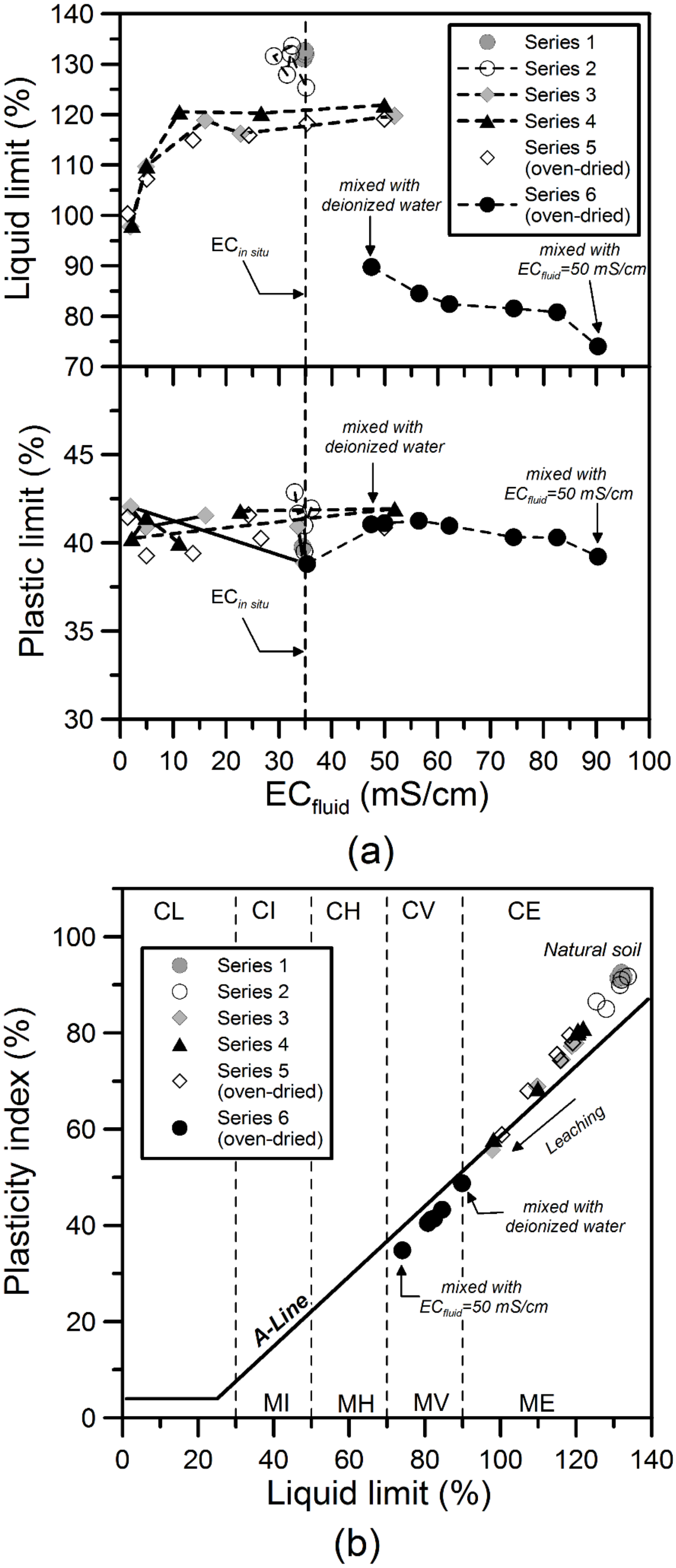


**Fig. 4** Results of the fall cone tests (Note: values of EC reported in the legend refer to $EC_{fluid}$)



(a)

(b)

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

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

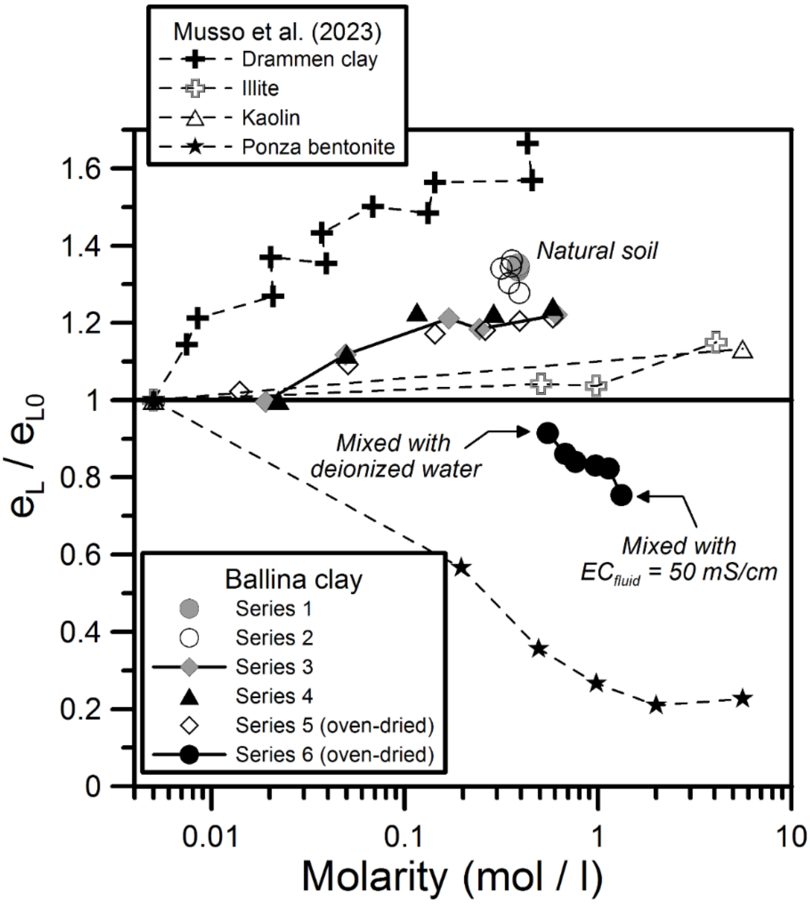


**Fig. 6** Evolution of normalized void ratio at liquid limit eL with NaCl concentration (modified from Musso et al., 2023)

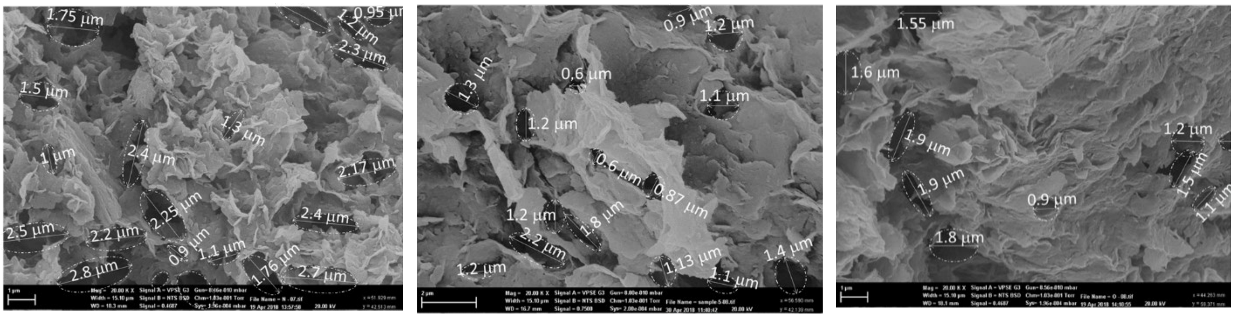
**Table 4.** Index properties and void ratio at liquid limit saturated with distilled water for active and non-active soils (from Musso et al., 2023)

| Soil | Main minerals | LL (%) | PL (%) | Activity | $e_{L0}$ | Reference |
|---|---|---|---|---|---|---|
| Kaolin | Kaolinite 75-80%<br>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<br>Chlorite | 32 | 23 | 0.18 | 0.88 | Torrance (1974) |
| Ponza bentonite | Montmorillonite 80%<br>Kaolinite 20% | 390 | 76 | 3.90 | 10.80 | Di Maio (1996) |

## 5.2 Changes in soil fabric

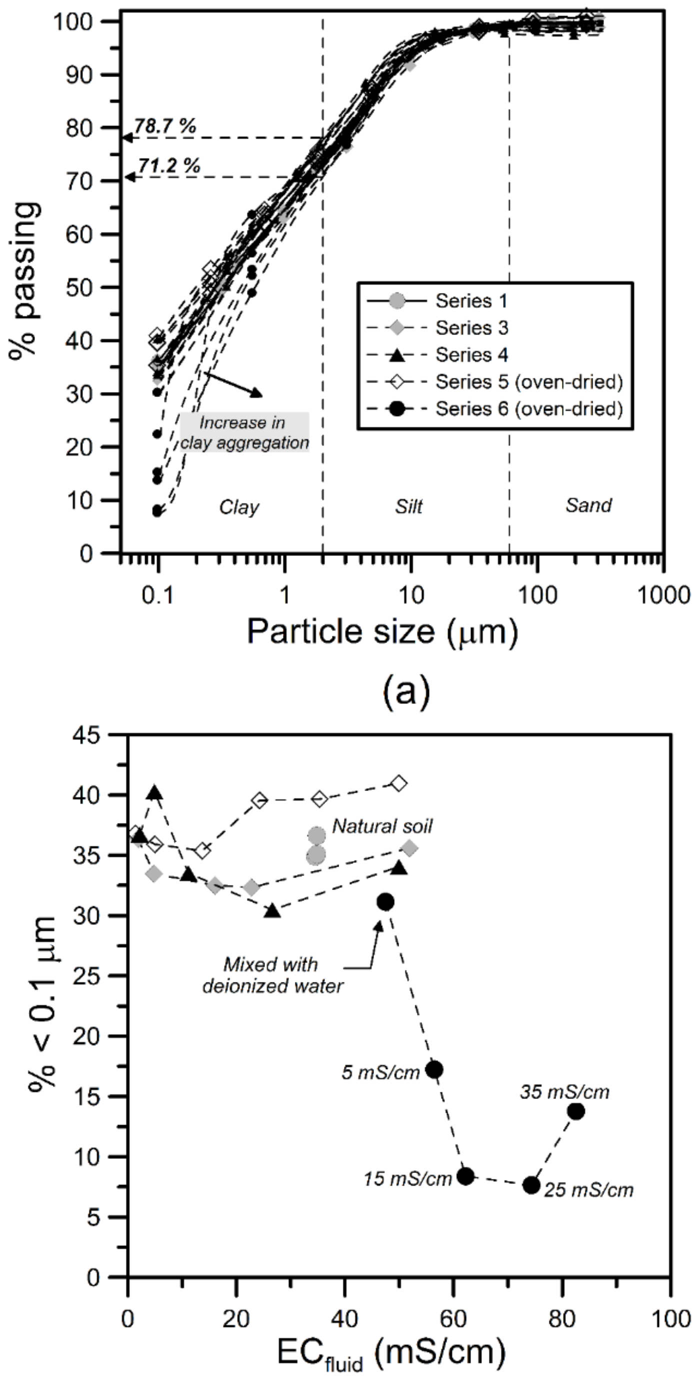
The effects of pore fluid salinity and oven-drying on soil fabric are evaluated via Scanning Electron Microscopy (SEM) and particle size distribution (PSD) analyses. SEM tests were performed in a field emission microscope (Zeiss Sigma VPFESEM) on specimens previously subjected to the freeze-drying process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

To evaluate the influence of oven-drying on soil fabric, Fig. 8 compares the PSD curves estimated post fall cone testing. PSDs were estimated on oven-dried soil (60°C) using a particle size analyser (Sedigraph III, Micromeritics®). Whereas the procedure for sample preparation, including the mixing of the dry soil with the dispersant, may cause modifications in the natural soil fabric, its effect is assumed the same for all tested specimens. Hence, differences between PSDs should reflect the influence of previous salinity and drying paths. Fig. 8a shows changes in clay content (%<2 $\mu$m) between 71.2 – 78.7 % without clear link with the previous salinity and drying paths. However, the inspection of the fractions below 2 $\mu$m shows an interesting trend, particularly for specimens from Series 6 where oven-dried clay was mixed with salt solutions. A progressive increase in particle size within the clay fraction is observed. This behaviour, which is indicative of clay aggregation, is clearer in Fig. 8b where fractions passing 0.1 $\mu$m are plotted against $EC_{fluid}$. Samples tested in Series 6 show a strong decrease in the proportion of particles lower than 0.1 $\mu$m from 30% to 7% with the increase in $EC_{fluid}$. In other words, the proportion of very small particle sizes decreases when the soil is oven-dried due to the tendency of those particles to aggregate. This behaviour is more evident with the increase in soil salinity.



Remoulded clay Leached to 15 mS/cm Fully leached

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



(a)

(b)

**Fig. 8** Particle size distribution of tested specimens (a). Variation in fraction < 0.1 μm with EC$_{fluid}$

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

Results from chemical analyses performed to evaluate the changes in pore fluid dissolved cations and anions are shown in Fig. 9. The dashed vertical line represents the pore fluid salinity for natural soil, used here as reference. For the natural soil (Series 1) sodium ($Na^+ \approx 7.55$ g/L) is the major cation in the pore fluid. Other major cations present are potassium ($K^+ \approx 0.51$ g/L), magnesium ($Mg^{2+} \approx 1.63$ g/L) and calcium ($Ca^{2+} \approx 0.42$ g/L). Chloride is the predominant anion ($Cl^- \sim 9.20$ g/L) followed by sulphate ($SO_4^{2-} \sim 5.22$ g/L). The large proportion of soluble $SO_4^-$ in the pore fluid at natural state (5.22 g/L, i.e. around 70% the total of $Na^+$) agrees well with the predominance of $Na^+$ and the presence of $K^+$(Tabatabai, 1987). In Fig. 9 grey crosses represent the variation in cation and ions upon leaching for the clay used in Series 4. $Na^+$ reduces linearly with $EC_{fluid}$ to 0.137 g/L at the end of leaching. This represents a reduction of 98.2% with respect to initial value. Monovalent and divalent cations reach values of $K^+$=0.014 g/L, $Mg^{2+}$=0.002 g/L, $Ca^{2+}$=0.011 g/L. Similarly, leaching causes almost complete removal of $Cl^-$ =0.080 g/L (~99.1%) and $SO_4^{2-}$ = 0.014 g/L (~99.7%). It is important to note that the remoulded clay subjected to partial leaching in Series 3 (empty diamonds) follows the same trend. Percolation of remoulded clay in Series 3 with pore fluid prepared at 50 mS/cm (salinisation) increases the proportion of $Na^+$=13.87 g/L (~84 %) but also decreases $Mg^{2+}$=0.615 g/L, $K^+$=0.38 g/L and $Ca^{2+}$=0.122 g/L. A remarkable reduction in $SO_4^{2-}$ (~0.287 g/L) is observed. Remoulded clay subjected to re-salinisation after full leaching in Series 4 (triangles) follows the same trend of the remoulded clay exposed to leaching in Series 3, thus indicating large degree of reversibility during leaching/salinisation paths. Sulphates $SO_4^{2-}$ are the exception to this trend, with 96% reduction upon re-salinisation.

Interestingly, the change in cations and anions for the clay tested in Series 5 (oven-drying followed by full leaching and re-salinisation) follows a similar trend as the remoulded clay from Series 4 not exposed to oven-drying. Large variation in $EC_{fluid}$, cations and ions is observed in Series 6 where oven-dried clay was mixed (no percolated) with solutions of variable salinities. In this case, $Na^+$ : 6.0–21.6 g/L, $K^+$ : 0.157–0.607 g/L, $Mg^{2+}$ : 0.49–2.5 g/L, $Ca^{2+}$ : 0.145–0.74 g/L, $Cl^-$ : 4.45–29 g/L, and $SO_4^{2-}$ : 1.03–3.78 g/L. Although oven-drying has the same effect on the clay tested in Series 5 and Series 6, i.e. precipitation of salts initially dissolved in the pore water as well as clay aggregation, those effects vanish when the oven-dried clay is percolated for several weeks with deionized water (Series 5) to full leaching prior to re-salinization. Long leaching times promoted particles disaggregation. The trend between salinity and liquid limit found for Series 5 is thus the same of the non oven-dried specimens, consistent with what is expected for non-expansive clays, where the increase in salinity promotes a face to edge to edge arrangements. On the other hand, disaggregation was not obtained by simply mixing the soil powder with distilled water and letting the slurry equilibrate for 48 hours (Series 6). In such case, the effect of the increase in salinity appears to be only a decrease in the repulsive forces between particles, which likely only promoted a decrease of the repulsive forces between particles (e.g. Mitchell and Soga, 2005) and thus further aggregation.

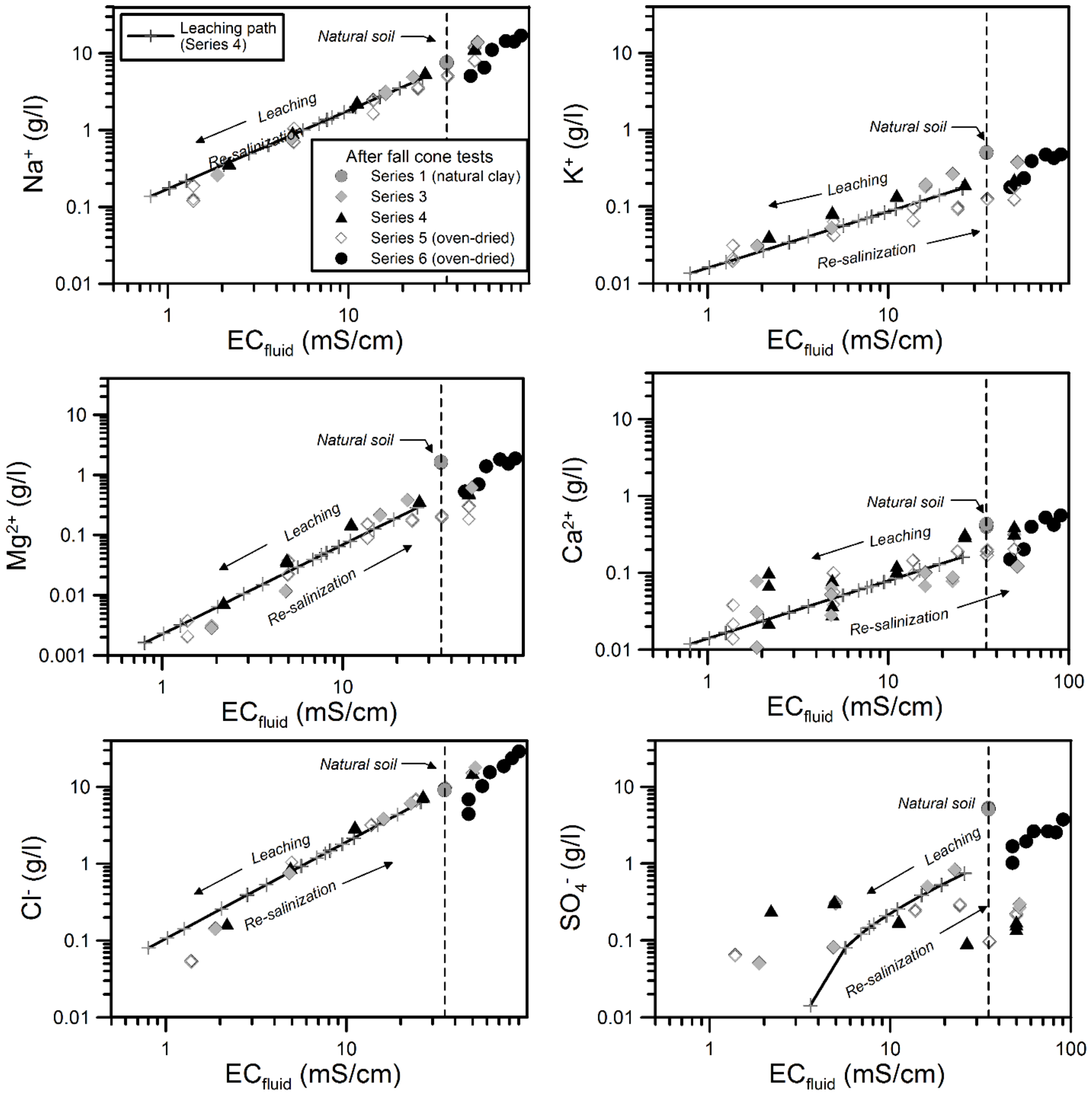


**Fig. 9.** Changes in ion concentrations for each test Series

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

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

The solution ionic strength *I* is calculated as the sum of the molarities of each dissolved species 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 ($EC_{fluid}$ = 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 ($EC_{fluid}$=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 $EC_{fluid}$≥ 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 $EC_{in\ situ}$ in Series 4 and Series 5, another factor to be considered is the severe decrease in $SO_4^-$ shown in Fig. 9.

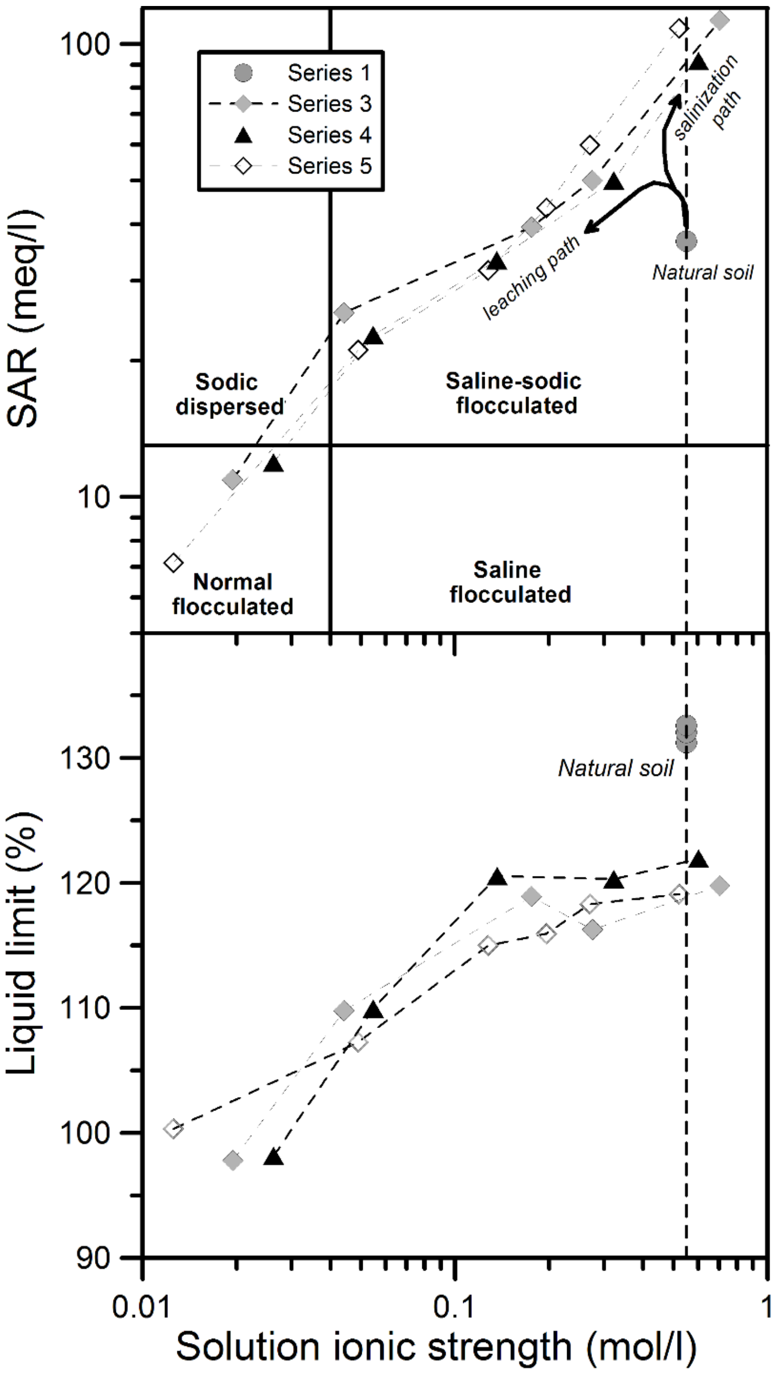


**Fig. 10.** Solution ionic strength vs. associated other parameters

## 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.

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

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

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