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Comparative Analysis of Physico-Chemical and Potassium Sorption Properties of Sensitive Clays

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Abstract: The paper analyses physico-chemical and geotechnical characteristics of four Scandinavian sensitive soils formed under different environmental depositional conditions, with the main aims to contribute to the knowledge of sensitive soils and assess the potassium sorption capacity among the investigated soils, as a basic characteristic to evaluate the effectiveness of treatment with KCl and to analyse potassium migration in such soils. The results show that, although the chemical composition of the four soils is very similar, their sensitivity is significantly different. The correlation from literature linking the specific surface, mineralogy, and plasticity in sensitive clays of Eastern Canada, was found to be qualitatively valid for the investigated Scandinavian sensitive clays, too. The highest value of the sensitivity index among the tested soils was found to be related to the lowest cation exchange capacity and to a limited amount of amorphous minerals. These characteristics contribute to explaining the highly sensitive behaviour of that soil affecting the structure formation during the deposition stage. The potassium sorption capacity has been experimentally investigated through batch tests specifically performed on the sensitive soils, as the first step to quantify the maximum sorption capacity and identify the main factors affecting it. The maximum potassium sorption capacity was always lower than that estimated by the cation exchange capacity, and it increased with the cation exchange capacity, plasticity index, and activity of the soils, as well as with the amount of phyllosilicates and amorphous minerals.

Keywords: sensitive clays; microstructure; cation exchange capacity; sorption; potassium

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1. Introduction

Clayey soils exhibiting a fragile structure that completely lose their undrained shear strength upon remoulding are designated as sensitive clays. The degree of sensitivity is quantified by the ratio between the undrained shear strength of an undisturbed specimen and the strength of the same specimen at the same water content but in a remoulded condition [1–3].

The high sensitivity of clays is linked to depositional and post-depositional factors [1]. A primary depositional factor is the inter-particle flocculation, which creates an open microstructure. In clays mainly deposited in a saline marine environment (such as Norwegian quick clays), the high concentration of salt ions reduced the repulsive forces between particles, inducing flocculation. Among post-depositional factors, salt leaching is generally the most significant. After the uplift of clayey deposits due to retreating glaciers, a decrease in the pore water salinity occurred due to leaching of salts through rainfalls and fresh groundwater flow. The open flocculated structure persisted, but the repulsive forces among soil particles increased, resulting in a reduction in both liquid limit and strength when the material was remoulded. Such sensitive clays, with a very low remoulded shear strength ($\leq$0.4 kPa), are termed quick clays according to the classification by Rankka et al. [4].

Owing to their inherent characteristics, high-sensitivity clay deposits are prone to landslide failures. Since leached clays with low salt contents develop high sensitivity, e.g., [1,5], ground improvement methods for sensitive clays have been proposed, consisting of re-introducing high salt contents to reduce the repulsive forces among the clay particles and make the clay more difficult to remould. Potassium salt solutions (KCl) were more effective to the purpose than other major cations (Na^+, Ca^{2+}, Mg^{2+}), mainly due to their smaller hydrated radius resulting in a larger ionic potential, which makes the charge more effective [6–8]. In particular, the installation of salt wells filled with potassium chloride was recently investigated by Helle et al. (2017) in quick-clay hazard areas [8]. The migration of potassium chloride, and consequently the effectiveness of improving the geotechnical properties, was found to depend on several factors, including adsorbed ions, mineral type, and the cation exchange capacity of the clay.

The present paper analyses the physico-chemical and geotechnical characteristics of some Scandinavian sensitive soils formed under different environmental depositional conditions. The aim of the paper is both to contribute to the knowledge of sensitive soils and to assess the potassium sorption capacity of the investigated soils, as basic information to evaluate the effectiveness of treatment with KCl and analyse potassium migration in such soils. In particular, the potassium sorption capacity has been experimentally investigated by batch tests specifically performed on the sensitive soils, as the first step to quantify the maximum sorption capacity and to identify the main factors affecting. To the best of the authors' knowledge, there are no previous studies involving sorption tests on sensitive soils with the aim of evaluating effectiveness of treatments to improve the mechanical characteristics.

2. Materials

Four different sensitive soils were investigated, three of them taken in the southern part of Finland, close to the cities of Kotka, Perniö, and Kouvola, whereas the fourth soil came from the Tiller site, 10 km south of Trondheim in Norway (Figure 1). Table 1 shows the sampling depth and the water content at sampling of each soil, as well as the main physical characteristics and the relevant testing methods [9,10]. As is typical in sensitive soils, the natural water content is higher than the liquid limit. The liquid index is in the range of 0.9–2.6, with the highest value observed for the Perniö soil, which is the richest in clay fraction.

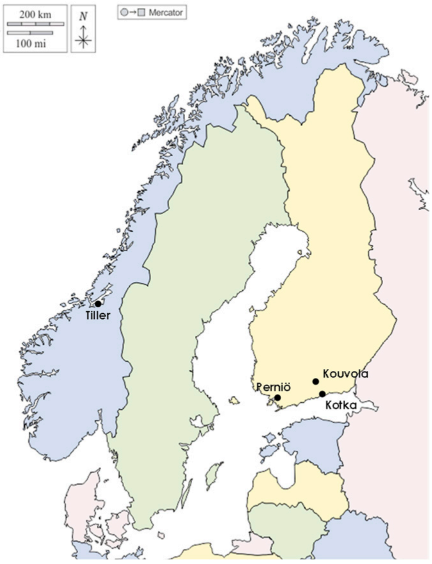

Figure 1. Location of the investigated soils.

Table 1. Properties of the investigated soils.

	Kotka, Finland	Perniö, Finland	Kouvola, Finland	Tiller, Norway
Depth of sampling (m from g.l.)	1.5–3.1 (dry crust)	4.5	1.0–1.5	10.1
Fine fraction, FF (%) [11]	98	83	78	63
Clay Fraction, CF (%) [11]	45	65	24	44
Liquid limit, LL [12]	80	48	28	30
Plasticity Index, IP [13]	38	21	12	10
Natural water content (%) [14]	80–130	82	27–32	35

3. Test Methods

Microstructural and chemical investigations were performed by means of SEM observations and Energy Dispersive X-Ray Spectroscopy (EDS). Observations were carried out with Philips XL20 Microscope (W filament, detector Everhart–Thornley for secondary electrons, solid state detector for backscattered electrons, maximum voltage: 30 kV) after air dewatering of the samples and their gilding by means of Emitech K550 sputter coater. EDS was performed by 20–30 kV voltage.

The mineralogical composition was investigated using X-Ray Diffraction, (XRD) with a Philips diffractometer (PW1730 X-ray generator, PW 1050/70 goniometer, and CuK radiation). The cation exchange capacity (CEC) and the specific surface of the four soils were determined using the methylene blue test [15].

The sensitivity of the soil samples was assessed using the fall cone test, FCT [16], a rapid and straightforward method for empirically determining the undrained shear strength of clays [12]. This test involves a cone placed on the surface of a sample and allowing it to drop under its own weight into the sample. Penetration occurs at a rate assumed to be sufficiently rapid for undrained conditions to be applicable. The undrained shear strength is given as a function of the weight, geometry, and penetration depth of the cone into the sample [17,18]. Sensitivity is defined as the ratio of the undrained shear strength measured on an undisturbed sample to that measured on the same sample after it has been remoulded at the same water content. The 30°-100 g cone was used in testing all the undisturbed samples of the Finnish soil, whereas the 60°-10 g cone was used for the remoulded ones [16].

The sorption capacity of the soils was analysed using batch adsorption tests according to the procedure for fine-grained soils [19] except for the contact time, which was 72 h instead of the standard 24 h to ensure better assurance of chemical equilibrium. A total of 5 g of dry soil was added to 100 mL of a KCl solution at different concentrations (Table 2). The range of KCl concentrations was selected to include the typical concentrations documented in the literature for the treatment of sensitive clays to improve their geotechnical properties, e.g., [7,20]. The final solution was analysed after centrifuging. Double or triple tests were performed on some of the soils when the available quantity was enough.

Table 2. Solutions of K^+ used in batch tests (EC = Electrical Conductivity).

Solution	Target K^+ Concentration (g/L)	Measured K^+ Concentration (g/L)	EC (µS/cm)
C_1	0.1	0.1	418
C_2	0.2	0.2	753
C_3	0.5	0.536	1674
C_4	1	0.954	3410

The sorption ratio, *S*, was calculated as follows:

$$S = \frac{m_i - m_f - m_e}{m_s},$$

where m_i and m_f are the initial and final masses of K^+ in solution, respectively, m_e is the mass of K^+ eluted from the soil in the batch test with deionized water, and m_s is the dry mass of soil sample. The eluted mass of K^+ was subtracted from the mass lost from the solution for a conservative evaluation of the soil's sorption capacity, supposing that the same eluted mass of potassium occurs independently of its concentration in solution. In any case, the eluted mass of potassium corresponds to at most 10% of the minimum sorption ratio in all the tests.

Since the batch tests were performed on the soil fraction passing through a 2 mm sieve [19], XRD was consistently performed on the same soil fraction, as the main aim was to assess the presence of a specific clay mineral [21].

4. Results and Discussion

4.1. Chemical and Microstructural Investigation

EDS carried out on the soil samples (Table 3) highlighted the presence of silicon, aluminium, and iron as the main components, in very similar proportions. Potassium, calcium, sodium, and magnesium were detected in smaller amounts. In particular, in the Tiller soil, the amount of the bivalent cations, i.e., calcium and magnesium, is slightly higher than in the other two Finnish soils.

Table 3. Chemical composition of the soils (by EDS).

	Weight Composition (%)			
Element	**Perniö, Finland**	**Tiller, Norway**	**Kotka, Finland**	**Kouvola, Finland**
Silicon	52	46	52	55
Calcium	2	5	2	2
Aluminium	16	16	15	16
Iron	14	16	16	15
Potassium	8	6	7	7
Magnesium	4	8	3	2
Sodium	2	3	2	3
Other compounds	<2	<1	<3	0

X-ray diffraction patterns (Figure 2) highlighted some differences in the mineralogical composition of the investigated soils. Amphibole, a double chain inosilicate, and quartz are the only minerals detectable in all the soils of concern. In particular, in the Kouvola soil, the main quartz peak is very pronounced, high and narrow, highlighting a well-crystallised mineral, with large mineral crystals (the width at half height is very small). In addition, the peak intensity is consistent with that of the pure mineral. For this soil, quartz is clearly predominant over other minerals, as evidenced by Rietveld analyses (quartz = 51%; albite = 34%, sanidine = 15% by weight). This is consistent with its greater content of silty and sandy fractions (59% and 19%, respectively) compared to the other soils. Chlorite and Mica are detected in the crystalline phase of all the soils except for the Kouvola soil. Albite, a plagioclase rich in sodium, and sanidine, a potassium feldspar, are detected in the crystalline phase of the three Finnish soils. The mineralogical composition of the crystal phase detected for the four soils of concern is very similar to that observed by Paniagua and L'heureux (2019) when analysing three Norwegian marine sensitive clays by XRD [21], two of which came from the same region as the Tiller soil. They detected muscovite, quartz, and plagioclase as the main components, along with chlorite and K-feldspar.

In addition, the significant upper vertical translation of the Perniö spectrum over the baseline is evidence of a substantial contribution of the amorphous phase in this soil [22,23]. The detected peaks are low and wide, indicating small dimensions of crystals. A contribution of amorphous compounds is also detected in the Kotka and Tiller soils. The spectrum of the soil from Kouvola seems to be unshifted; therefore, no significant presence of amorphous compounds can be identified.

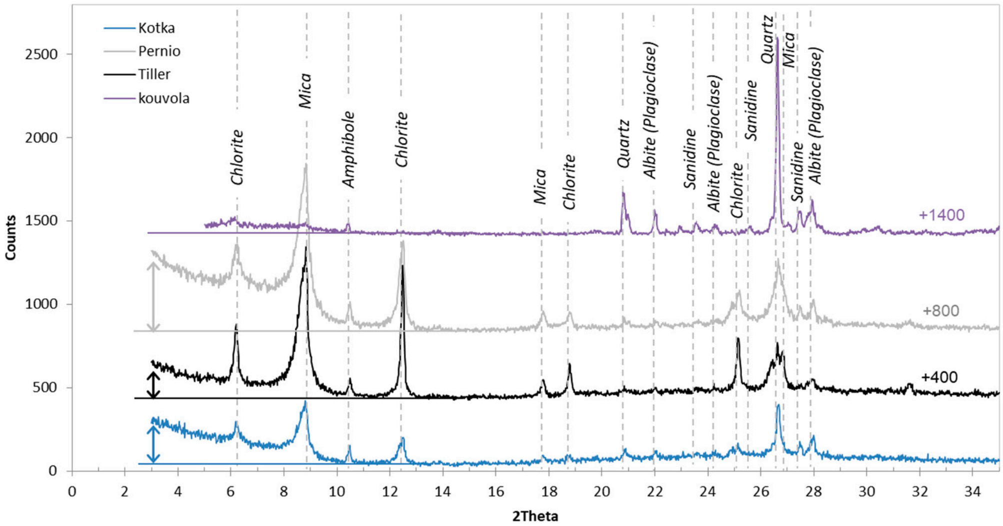

Figure 2. X-ray diffraction spectra of the studied soils.

Micrographs of the soils are shown in Figure 3. The main mineralogical constituents of the soils, as revealed by X-ray diffraction, are evident also in the SEM images, confirming the findings of the XRD analyses. Mica is marked with M in Figure 3a,f and is observed in all the soil samples: in Figure 3a, the layered structure and regular cleavage planes, typical of Mica crystals, are well detectable, reaching more than 60 micron in size. Additionally, typical rosette-like structures of chlorite (ChL), formed as clusters of plates, are widely distributed across all the observed samples, sometimes occurring in agglomerated form, filling the space between other minerals. Amphiboles (A in Figure 3d) and plagioclase (PL in Figure 3b) are also detected in SEM images. Amphiboles, inosilicate minerals, are present as small (2–4 micron in length), needle-like crystals, as a result of monoclinic crystallisation. The elongate plagioclase structures in Figure 3b are constituted by lamellar albite twinning and their length is approximately 20–30 microns. In the Kotka soil sample, euhedral pyritic grains (Py in Figure 3e) are sporadically present, as highlighted by the presence of sulphur and Iron as the only constituents when EDS is directly performed on the crystals. The pyritic grains, approximately 1 micron in size, are gathered together to form large, localised groups. Sanidine is also easily identifiable in Figure 3e.

Diatoms are systematically detected in the observed samples (Figure 4). They are centric diatoms, usually present in freshwater environments.

4.2. Sensitivity and Specific Surface

Based on Rankka's classification of sensitivity [4], soils from Kotka and Kouvola are defined as medium-high sensitive soils, while the Perniö and Tiller clays are considered highly sensitive. Nonetheless, the sensitivity index of the Tiller soil is very high compared with other values, mainly due to the low remoulded strength value (Table 3) when compared with the values for Finnish soils. This occurrence connotes the Tiller soil as the most sensitive among those tested. As previously noted, the sensitivity and remoulded strength values offer limited information related to the potential activation of landslides, with the remoulding energy emerging as a crucial factor in this context.

In order to assess this differentiation, specific surface values were determined by the methylene blue adsorption method (spot test), which is a quick, reliable, economical, and easy-to-perform method to appreciate the presence of clay minerals [24]. In particular, specific surfaces can capture the combined effects of particle size and slenderness, which can influence the arrangement of clay particles and, therefore, the behaviour of clayey soils.

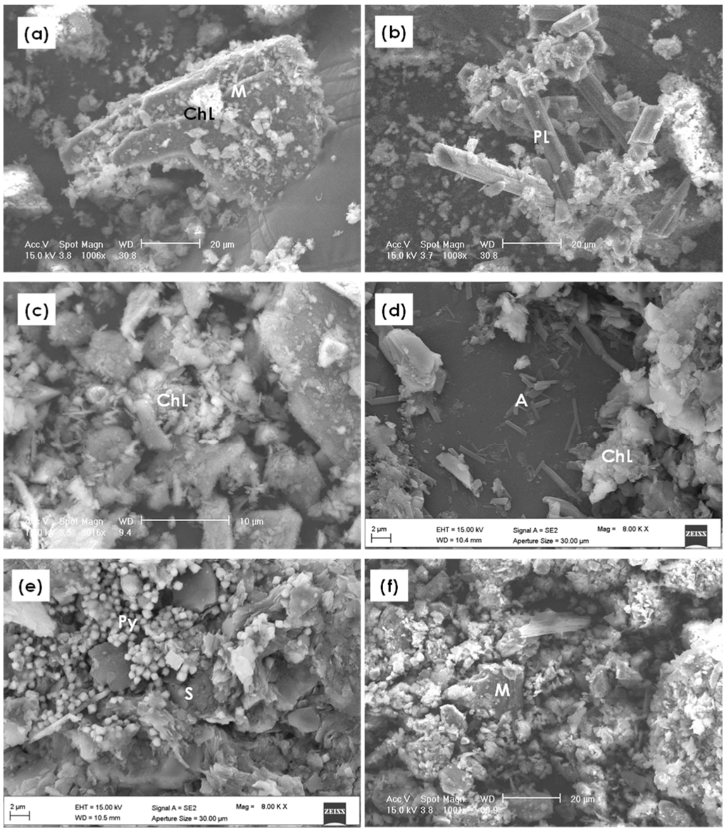

Figure 3. SEM images of the investigated soils: (**a**,**f**) Perniö soil; (**b**,**d**,**e**) Kotka soil; (**c**) Tiller soil. PL—Plagioclase, ChL—Chlorite, M— Mica, A— Amphibole, Py— Pyrite, S—Sanidine.

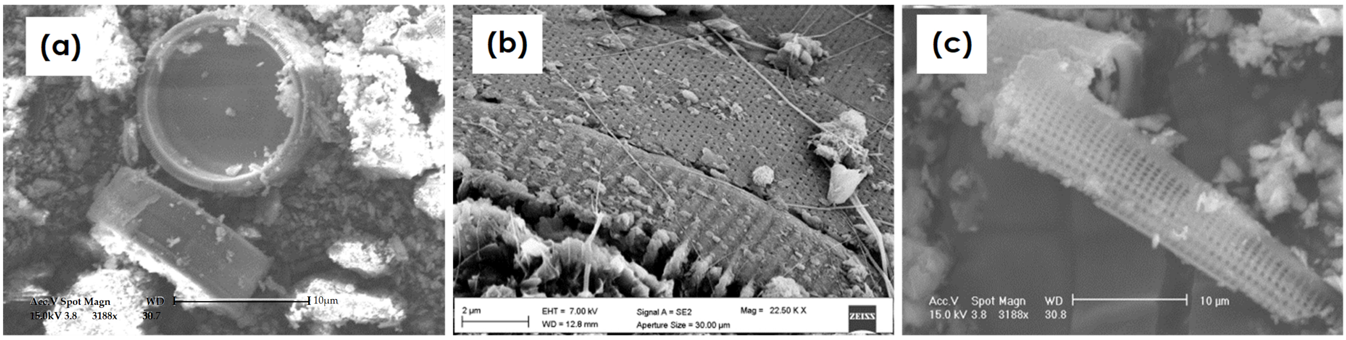

Figure 4. Micrographs of diatoms: (**a**,**b**) Kotka soil; (**c**) Perniö soil.

All the examined soils exhibit specific surfaces that significantly exceed 1 m^2/g, which represents a threshold value triggering sedimentation and fabric formation [25]. These processes are influenced by environmental factors such as pH and ionic concentration in the pore fluid, resulting in shrinkage, stiffening under unsaturated conditions, and mechanical–chemical coupling. Locat et al. (1985) established a correlation between the measured specific surface, mineralogy, and plasticity in sensitive clays of Eastern Canada [24]. Specifically, their findings indicate that higher specific surfaces correspond to high amounts of phyllosilicates and amorphous minerals (P). The results obtained by their correlation, based on the specific surfaces measured on the investigated soils, are listed in Table 4.

Table 4. Sensitivity values and results from methylene blue test (CEC = Cation Exchange Capacity).

	Kotka, Finland	**Perniö, Finland**	**Kouvola, Finland**	**Tiller, Norway**
Sensitivity index (-)	15–40	37–71	31	270–280
Fall cone remoulded strength (kPa)	0.5–0.9	0.3	5.0–7.0	0.1
Specific Surface (m^2/g)	115.8	109.6	77	54.2
CEC (meq/100 g)	14.9	14.1	8.6	7.0
P (%)	51	49	34	27

Referring to the values in Table 4, with a focus on the three clayey soils of Kotka, Perniö, and Tiller (as the soil from Kouvola is mainly a silty soil), it is evident that the soils from Kotka and Perniö exhibit the highest specific surface values, resulting in higher p values. Consequently, these soils display a greater abundance of amorphous compounds, along with significant peaks for Mica and Chlorite (Figure 2). The specific surface of the Tiller soil is the lowest among the sensitive soils of concern, corresponding to a P value of 27%, with the relative X-ray spectrum showing the smallest presence of amorphous compounds, although mica and chlorite are present. In any case, the specific surface and CEC of Kotka and Perniö are twice that of Tiller. The lower CEC value observed in the Tiller sample may help explain its high sensitivity index, along with low remoulded strength and remoulding energy. This lower CEC value likely led to a comparatively limited extent of ionic exchange reactions between ions on soil particles and ions during its deposition in the marine environment, in contrast to the soils from Kotka and Perniö (originated in brackish water). Consequently, Tiller exhibited a less stable flocculated structure after formation and was susceptible to remoulding. To add to the merit, Bentley and Smalley (1978) were the first to observe that high sensitivity corresponded to low concentrations of Na^+ and K^+ in the interstitial water [26], indicating that the role of amorphous material is probably important in the development of sensitivity, promoting the open structure of the clay. Later, Yong et al. (1979) indicated that a high content of amorphous material correlates with low sensitivity [27], and Locat (1982) stated that amorphous materials can also act as a cementing agent during sedimentation, keeping the flocculated structure open [28].

The combined use of the methylene blue test and XRD can give useful information to understand the sensitive behaviour of Finnish and Norwegian clay.

4.3. Potassium Sorption Capacity

Figure 5 shows the potassium sorption isotherms of the investigated soils, along with the values of the electrical conductivity of the equilibrium solutions and the maximum sorption capacity (S_{max}) calculated on the basis of the CEC value of each soil (Table 4), in the hypothesis of complete saturation with potassium of the exchangeable sites of the soil.

Notwithstanding an appreciable scattering, the investigated soils show an increase in the sorption capacity with potassium concentration up to 0.2–0.4 g/L, after which the trend is almost constant, except for the Kotka soil (Figure 5a). For this last soil, the experimental data at the highest concentration gave two very different values of the equilibrium concentration and, therefore, of the S values (S = 3.50 mg/g and S = 0.34 mg/g). Considering that the EC values of the two final equilibrium solutions are almost the same (Figure 5d) while 0.34 mg/g is even equal to the S value obtained at the testing lowest equilibrium concentration, it is reasonable to assume that S = 0.34 mg/g is a value affected by an unidentified measurement error. For this reason, such a value is not considered in the analysis of the results. Therefore, considering the only reliable value of S at the highest equilibrium concentration, the Kotka soil shows an increase in the sorption capacity above the equilibrium concentration of 0.4 g/L.

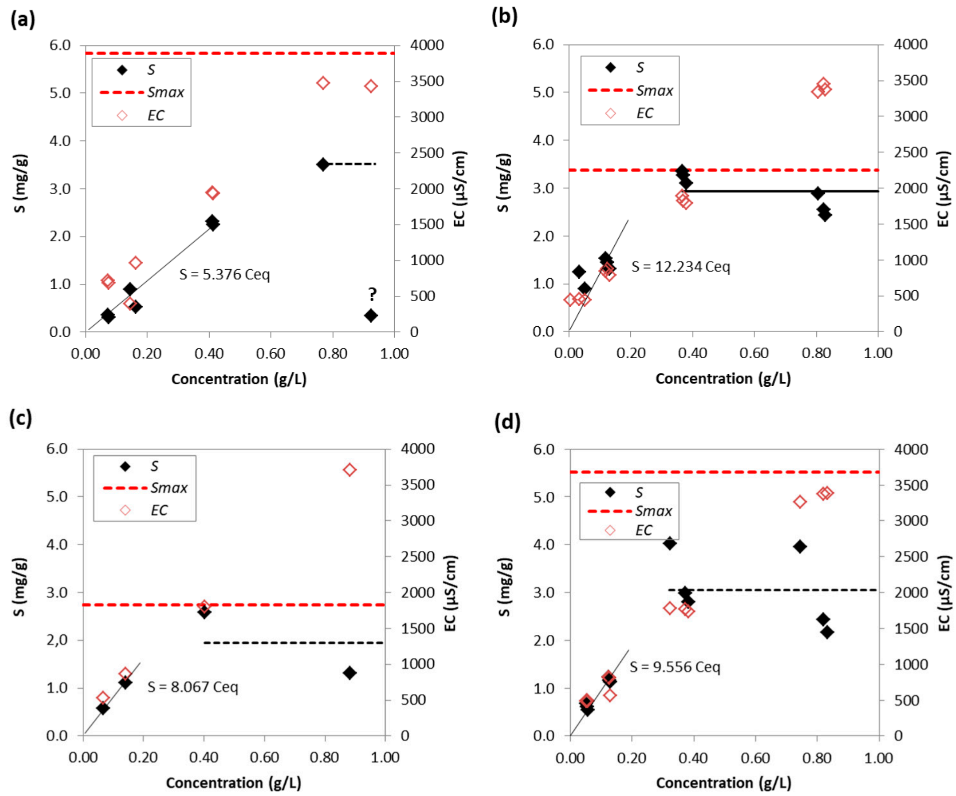

Figure 5. Batch test results: (**a**) Kotka; (**b**) Kouvola; (**c**) Tiller; (**d**) Perniö.

Since neither the Langmuir nor the Freundlich sorption isotherms fit the experimental data well, the results of the batch tests on all the investigated soils were analysed considering a linear isotherm at low concentrations and a constant sorption for high concentration values (Figure 5). In particular:

- for the Perniö, Kouvola, and Tiller soils, the values of S at the equilibrium concentrations exceeding 0.35 g/L, S_{lim}, were assumed to be equal to the average value of the corresponding experimental data;
- in the case of the Kotka soil, S_{lim} was assumed equal to the highest S value detected;
- for all the investigated soils, the values of S lower than S_{lim} were fitted by the linear isotherm, as typically occurs at low range of concentrations:

$$S = k_d\, C,$$

where C is the equilibrium concentration and k_d is the distribution coefficient [L^3/M].

The values of S_{lim} were always lower than the relevant values of S_{max} (S_{lim}/S_{max} = 55%–87%). This means that part of the exchangeable sites is already occupied by potassium (Table 3) and/or that exchangeable cations other than K^+ are more tightly adsorbed and cannot be displaced, at least with reference to the investigated range of potassium concentrations.

The values of S_{lim} are plotted in Figure 6 as a function of the cation exchange capacity, plasticity index, activity, A (A = PI/CF) and P (values in Tables 1 and 4). As expected, the higher the CEC, the higher the S_{lim} value (Figure 6a), as the mass of potassium that can be adsorbed is greater. The S_{lim} value also increases with the plasticity index (Figure 6b) and with the activity (Figure 6c), both of which reflect the quality and quantity of the clay fraction in soils, as well as with the P value (Figure 6c), which takes into account of reactive amount of minerals (phyllosilicates and amorphous minerals) in soils.

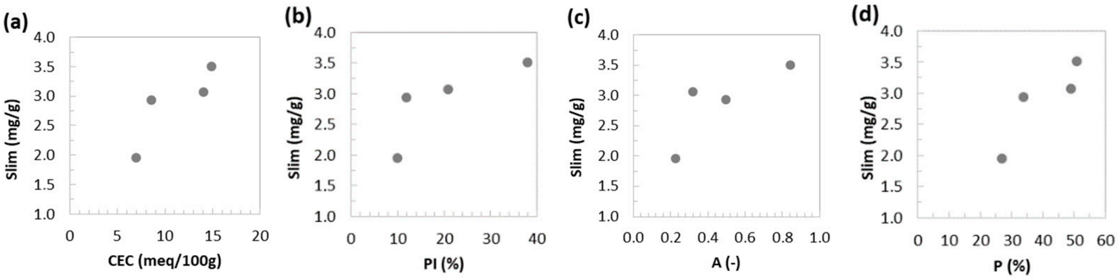

Figure 6. Limit potassium sorption capacity of the investigated soils, S_{lim}, as a function of (**a**) cation exchange capacity; (**b**) plasticity index; (**c**) activity; and (**d**) *P* value.

It is important to point out that the utmost value of S_{lim} in Figure 6 (3.50 mg/g) could be higher, as discussed previously (Kotka soil, Figure 5a). In this case, the increasing trends in Figure 6 would be however confirmed.

With reference to k_d, the results show no evident trends with the previously considered geotechnical and mineralogical factors. The different affinities of the investigated soils for potassium could be due to variations in the morphology of the clay surface exchange sites, which results in a different accessibility for potassium adsorption at low concentrations.

From a practical point of view, the results show that a potassium equilibrium concentration in solution of at least about 0.4 g/L is necessary to obtain the limit sorption capacity. It is important to point out that the actual conditions of in situ-treated soils can be less favourable to sorption than those of the batch tests, mainly for the lower accessibility of the exchangeable sites of the clay particles. Therefore, the values of S_{lim} evaluated by batch testing can be considered the upper limit of the effective sorption capacity of a soil. For the same reason, the equilibrium potassium concentration necessary to reach the limit sorption capacity could be higher than 0.4 g/L. In this regard, it is important to point out that research performed on bentonites [29] and on mixtures of cement and bentonite [30] found that the sorption capacity of cations, evaluated by batch tests, was very similar to the results obtained from column tests on entire samples, i.e., under hydraulic and chemical conditions more similar to the actual conditions of the soil than those of the batch tests. However, simple diffusion tests and column tests with potassium solutions are planned in the near future on one of the investigated sensitive soils to assess the potassium sorption behaviour in conditions similar to those of on-site treatments and to analyse the influence of the soil density on its potassium sorption capacity and effective diffusion coefficient.

5. Conclusions

Four Scandinavian sensitive soils of different origin, sensitivity, and remoulded strength values have been investigated with the main aims: to contribute to the knowledge of sensitive soils and to assess the potassium sorption capacity among the investigated soils as a basic characteristic for evaluating the effectiveness of treatment with KCl to improve their mechanical characteristics. The chemical composition of the soils resulted very similar, while X-ray diffraction highlighted significant differences in the mineralogical composition.

Based on the findings, the following conclusions can be drawn.

Soils from Kotka and Perniö exhibit the highest specific surface values and corresponding *P* values, showing presence of amorphous compounds as well as significant peaks for Mica and Chlorite. In contrast, the Tiller soil has the lowest specific surface among the sensitive soils, thus the lowest *P* value.

The cation exchange capacity (CEC) of Kotka and Perniö is twice that of Tiller, which may help explain their high sensitivity index, along with low remoulded strength and remoulding energy.

Tiller's lower CEC value likely led to a comparatively limited extent of ionic exchange reactions between ions on soil particles and ions during its deposition in the marine environment, in contrast to the soils from Kotka and Perniö (originating in brackish water).

Consequently, Tiller exhibited a less stable flocculated structure after formation and was susceptible to remoulding.

The combined use of the methylene blue test and XRD can provide valuable information to understand the sensitive behaviour of Finnish and Norwegian clay.

The limit potassium sorption capacity of the investigated soils resulted in the range of 0.6–0.9 of the maximum value estimable on the basis of the cation exchange capacity. An equilibrium potassium concentration in solution of at least 0.4 g/L was necessary to obtain the limit sorption capacity. Since the actual conditions of in situ treatments can be less favourable to sorption than those of the tests performed (batch tests), the assessed values of S_{lim} can be considered the upper limit of the effective sorption capacity of a soil. For the same reason the equilibrium potassium concentration necessary to reach the limit sorption capacity could be higher than 0.4 g/L.

The limit potassium sorption capacity was found to increase with the cation exchange capacity, plasticity index, and activity of the soils, as well as with the amount of both phyllosilicates and amorphous minerals.

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