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Short term effects on microstructure of a compacted Kaolin clay induced by lime addition

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Abstract **– Lime stabilization is a widely recognized improvement technique for reusing fine-grained soils unsuitable for earthwork constructions. After adding lime to an unsaturated soil, the two types of reactions take place, short term effects due to change in pore water chemistry and long-term effects due to pozzolanic reactions. In the last decades, attention has mainly been given to the mechanical long-term effects induced by pozzolanic reactions. On the other hand, short-term mechanicals effects have received little attention. The aim of this work is to investigate microstructural changes in the short-term effects and their effect on mechanical behavior of lime-stabilized soils.**

I. INTRODUCTION

Lime stabilization is widely used to improve engineering properties of marginal soils not suitable for use as construction materials in earthworks. The re-use of these soils represents a significant opportunity to reduce the environmental impact of earthworks [1].

Two different chemo-physical reactions take place after the addition of lime, namely cationic exchange and pozzolanic reactions, which develop simultaneously but on different time scales. Cationic exchange between calcium cations made available by lime addition and the hydrogen, sodium and potassium cations of the clay minerals takes place in the short period. In the long term, pozzolanic reactions take place with the development of stable compounds, such as hydrated calcium silicates and aluminates responsible of cementation bounds among soil aggregates, [2] [3]. These two mechanisms are respectively identified as process of modification and process of stabilization of treated soils [4]. At the macroscopic scale, the treated soil shows a different grain size distribution and plasticity, a decrease in compressibility and an increase in shear strength, the latter being strongly dependent on curing time [5].

Significant changes in the electrolyte concentration in the pore water take place after lime addition, influencing both physical and mechanical behavior of the treated soil. The influence of electrolytic concentration and cation valence on the thickness of clay particle double layer and its influence on strength has been demonstrated by [6]. More recently, the dependency of the residual shear strength of clays on the pore water chemistry has been highlighted by [7].

This work presents some results on the effects of short-term reactions on the microstructure of lime-treated clay. These results are part of a wider experimental programme on the physical and mechanical behavior of lime treated clayey soils compacted in saturated and unsaturated state. The attention is focused on the short-term effects due to the addition of divalent exchangeable calcium ions from two different sources, namely calcium oxide (CaO), or quicklime, and calcium chloride ($CaCl_2$). Treated samples were submitted to 1-D compression. MIP tests were then carried out to highlight the effects of calcium ions and its source on soil microstructure and, in turn, on mechanical behavior. The investigation presented here is limited to the saturated conditions and involve lime-added reconstituted kaolin. The rationale behind this study is that aggregates in compacted soils are made of ‘reconstituted’ materials [8]. As a result, an investigation of the response of re-constituted lime-added soils is the first step in the analysis of the response of lime-added unsaturated compacted soils.

II. SOIL AND EXPERIMENTAL PROCEDURE

The soil used for the experimental investigations is the powdered Speswhite Kaolin (English China Clay). Table 1 shows the main properties of the clay.

The minimum amount of quicklime (CaO) required for lime treatment has been established by means of pH measurements on samples prepared with 1:1 solid-liquid ratio [9]. The quicklime contents selected for samples preparation were 0.5%, 0.75%, 1%, 2%, 3%, 5%, and 7% by dry weight of natural soil. The variation of pH with lime content is shown in Fig. 1. Increasing the quicklime percentage, the pH of the pore water strongly increases. The 3% of quicklime eventually increased the pH up to the value of about 12.4, equal to the pH of a $Ca(OH)_2$ saturated solution. Quicklime treated samples for investigations were prepared by hand-mixing quicklime (1% and 3% of CaO), to the dry soil, and then adding demineralized water. The dissolution of the calcium

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Table 1. Physical and chemical properties of Speswhite kaolin.

Index property	Value
Liquid limit	70%
Plasticity Index	38%
Specific gravity	2.6
Natural pH	4.5

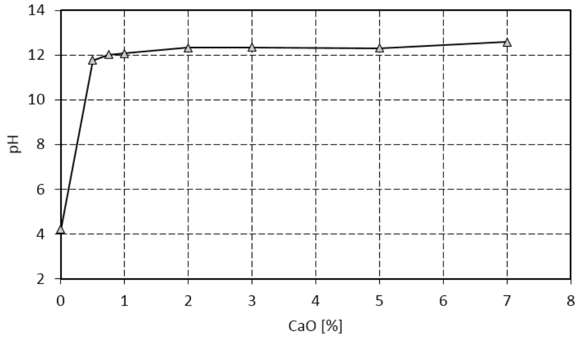

Fig. 1. Variation of pH with quicklime content.

hydroxide obtained after quicklime hydration reaction induces a highly alkaline environment, as evidenced in Fig. 1. In order to reach the same pH value, 12.4, for samples treated with calcium chloride, it has been necessary to add a base in the system. At this purpose, KOH has been chosen for minimizing the effect of the presence of another cation species in the water surrounding the clay particles because of the difference in the hydrates radii [10]. Calcium chloride treated samples were so prepared by hand-mixing the calcium chloride content, 1% of $CaCl_2$, to the dry soil, and then adding alkaline water obtained by dissolution of KOH in demineralized water.

All samples were prepared at water content higher than the liquid limit. The prepared samples were cured for 24 hours allowing the hydration of quicklime, or the dissolution of calcium chloride. Samples for 1-D compression tests have been formed directly into the oedometer ring. Samples were consolidated up to 2200 kPa and then unloaded. At the end of the unloading path, each sample was extruded from the ring, weighted and divided in two parts, one for measuring the initial and final water content [11] and the other one to perform the MIP tests.

Samples dehydration before MIP tests was performed following the freeze-drying method, [12] [13].

The oedometer tests on quicklime (QL) and calcium chloride (CC) treated samples are summarized in Table 2, where initial water content w_0, initial void ratio e_0, final water content w_f, final void ratio e_f, for each sample are reported. All the samples were submitted to loading paths in a very short curing time (two days). The samples prepared for MIP observations were taken directly at the end of the unloading path, so their water content and void ratio are the water content and void ratio measured at the end of the oedometer tests reported in Table 2.

Table 2. Tests on quicklime and calcium chloride treated samples.

ID	w_0 [%]	e_0	w_f [%]	e_f
QL1	100	2.64	45.9	1.20
QL2	100	2.51	45.3	1.18
CC1	100	2.57	44.9	1.17

III. RESULTS AND DISCUSSION

A. Oedometer tests

The results of oedometer tests on 1% and 3% CaO (QL1 and QL3 respectively) samples are reported in Fig. 2. At very low curing time (two days), the percentage of quicklime does not affect the compressive behaviour during loading sequence.

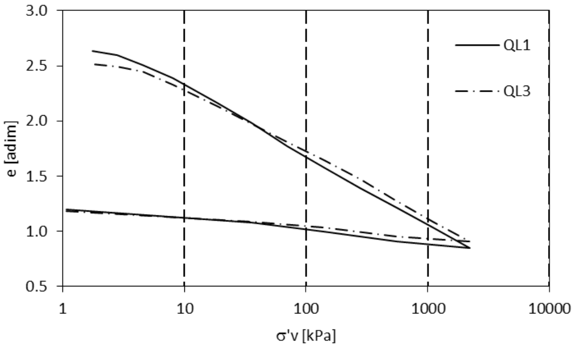

Fig. 2. Oedometer tests on lime-treated samples (1% and 3% CaO, QL1 and QL3 respectively).

The very alkaline environment induced by the dissolution of calcium hydroxide influences the pH dependent charge of the kaolin particles [14], favouring deprotonation at the edges of soil particles. Consequently, the soil particles are negatively charged both on surface and edge sites, [15]. The negative charge increases the cation exchange capacity (CEC) of the clay, favouring the adsorption of Calcium, [16] [17]. Around the lime fixation point [18], the CEC is no longer increased and the negative sites are completely covered of calcium ions, as confirmed by no further differences measured in the CEC at increasing lime percentages, [17]. It is likely that the 1% of CaO supports enough Calcium ions to completely cover the negative sites induced by the alkaline environment. As a result, the decrease of the interparticle repulsion forces takes place decreasing the compressibility of the soil as well as observed for 3%

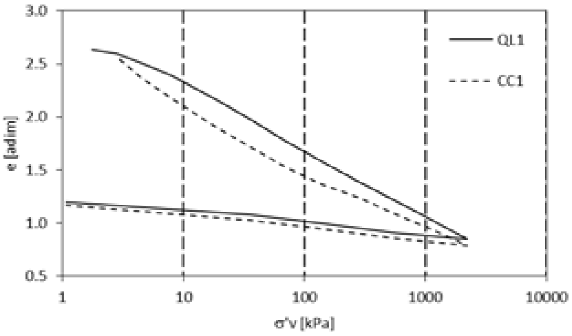

Fig. 3. Oedometer tests on 1% lime treated and 1% calcium chloride treated samples.

CaO treated samples.

Fig. 3 shows the oedometer compression curves of samples prepared with 1% of calcium chloride ($CaCl_2$) and 1% of calcium oxide (CaO) are reported and compared. The short term effects observed on the samples, in terms of initial void ratio and compressibility, were very similar, whatever the source of calcium cations. The same concentration induced the same effects in terms of particles arrangement. At the end of the load path, the two samples showed a similar final void ratio. Then similar considerations about compressibility can be applied in this case also for samples treated by calcium chloride

B. MIP tests

In Fig. 4 the results of MIP tests on 1% and 3% lime treated samples are reported in terms of cumulative intrusion void ratio (eMIP) and intrusion volume frequency (ΔeMIP/Δ(logd)) as function of entrance pore diameter (d). The different calcium concentrations seems not influence the pore-size density function: the two distributions are substantially mono-modals with the most frequent pore entrance diameters localized around 0.2 µm. After loading and un-loading steps, the fabric of the two samples is very similar, irrespective on the different concentration of calcium oxide induced by the treatment which seems to not influence the particles arrangement.

Similar results have been obtained using calcium chloride as source of calcium ions, as shown in Figure 5. The pore size density function, after the loading and unloading path, is monomodal with the most frequent pore entrance diameter localized around 0.2 µm. The high electrolytic environment induced by the addition of potassium hydroxide and the presence of calcium ions induced by the dissociation of calcium chloride have similar effects on the particles association induced by calcium oxide. The addition of a flocculant agent as calcium oxide in a high electrolytic environment reduces the potential energy and favours the interactions between the particles [19]. The resulting soil fabric is flocculated with higher initial void ratio respect to the one obtained with a dispersing agent [20].

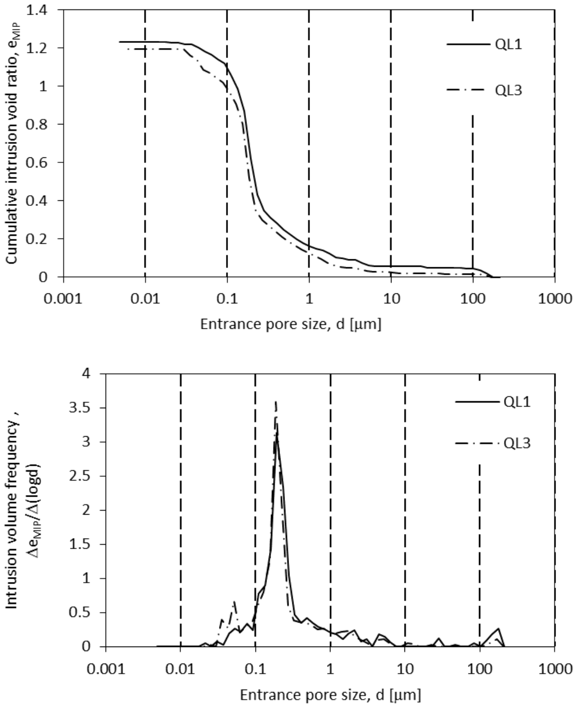

Fig. 4. MIP tests on lime treated (1% and 3%) samples.

IV. CONCLUSIONS

In the paper some results of a study on the physical and mechanical behaviour of a lime treated saturated and unsaturated kaolin (Speswhite kaolin) have been reported. The attention has been focused on the short term effects in terms of microstructure and compressibility behaviour of the treated soils at different lime percentage. Two different sources of calcium ions were then used to treat the samples, namely calcium oxide and calcium chloride, to understand if the particle arrangement depend on the source of calcium ions.

The results showed no substantial differences both in compressibility behaviour and fabric of the samples treated with 1% and 3% of CaO. Since the calcium ions tend to cover the negative site charge on the soil particles induced by alkaline pH, the concentration induced by 1% of CaO is sufficiently high to lower the repulsive forces between adjacent particles. The observation could justify the similar results showed by samples treated with 3% of CaO.

By adding calcium ions from a different source (calcium chloride instead of calcium oxide), the effects induced on the initial void ratio and on the compressibility are very similar due to similar particles association: the dispersant action induced by the alkaline environment is reduced introducing calcium ions in the pore water independently from the particular source.

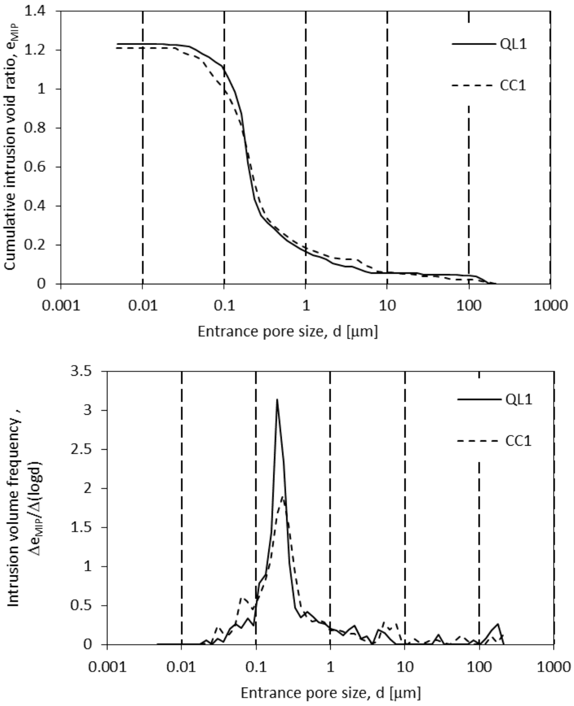

Fig. 5. MIP tests on 1% lime treated and 1% calcium chloride treated samples.

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REFERENCES

[1] P.Croce, G.Russo, “Reimpiego dei terreni di scavo mediante stabilizzazione a calce”. Proc. XXI CNG, L’Aquila, 2002, pp. 387-394, Patron.

[2] J.L.Eades and R.E. Grim, “A quick test to determine lime requirements for lime stabilization”, H.R.R, 1966, n. 139, pp. 61-72.

[3] G.Glenn and R.Handy, “Lime-clay mineral reaction products” in “Stabilization of Soil with Lime and Fly Ash”, 1963, Bull. N° 29, H.R.B, Wash., pp. 70-82.

[4] C.D.F.Rogers and S.Glendinning “Modification of clay soils using lime”, 1996, In Lime Stabilisation, C.D.F. Rogers, S. Glendinning and N. Dixon (eds.), 99-112, Thomas Telford.

[5] P.Croce, G.Russo, “Experimental investigation on lime stabilised soils”. Proc. XIII Eur. Conf. of Soil Mech. and Geotech. Eng. (ECSMGE), 2003, Prague.

[6] R.Young “A physic-chemical approach to analysis of clay soil behavior”. Symp. on Found. Eng.. Indian Inst. Science, 1961, A6: 1-12.

[7] R.W.Anson and A.B.Hawkins, “The effect of calcium ions in pore water on the residual shear strength of kaolinite and sodium montmorillonite”, 1998, Géothecnique 48, No. 6, 787-800.

[8] A.Tarantino, “Unsaturated soils: compacted versus re-constituted states”, Proc. 5th Int. Conf. on Unsaturated Soils, (E.E. Alonso and A. Gens, eds.) Barcelona, Spain, 6-8 Sept. 2010, vol. 1, 113-136.

[9] ASTM D 4972. “Standard Test Method for pH of Soils1”, ASTM, 1998, Annual Book of ASTM standard, Section 4: Construction, vol 04.09.: Soil and rock (II)

[10] A.G.Volkov, S.Paula, D.W.Deamer. “Two mechanisms of permeation of small neutral molecules and hydrated ions across phospholipid bilayers”, Bioelectrochemistry and Bioenergetics 42, 1997, pp. 153-160

[11] ASTM D 2216. “Test Method for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass3”, ASTM, 1998, Annual Book of ASTM standard, Section 4: Construction, vol 04.09.: Soil and rock (II).

[12] P.Delage, & F.M.Pellerin. “Influence de la lyophilisation sur la structure d’une argile sensible du Québec”. Clay Minerals, 1984, vol. 19 pp. 151-160.

[13] A.Tarantino, and E.De Col, “Compaction behaviour of clay.” Géotechnique, 2008, vol. 58, pp.199-213.

[14] Y.-H.Wang and W.-K.Siu “Structure characteristics and mechanical properties of kaolinite soils. I. Surface charges and structural characterizations” Can. Geotech. J., 2006, n°43, pp 587–600.

[15] C.Ma and R.A.Eggleton “Cation exchange capacity of kaolinite”, Clays and clay minerals, 1999, Vol. 47, No. 2, pp. 174-180

[16] A.R.Ferris and W.B.Jepson “The exchange capacities of kaolinite and the preparation of homoionic clays”. Journal of Colloid Interface Science, 1975, No.51, pp. 245-259.

[17] F.G.Bell “Lime stabilization of clay minerals and soils”. Eng. Geology, 1996, No.42, pp 223-237.

[18] G.H.Hilt and D.T.Davidson. “Lime fixation in clayey soils”. Highway Research Board, 1960, Bulletin 262, 20–32.

[19] A.Sridharan, S.Rao and G.Rao. “Shear strength characteristics of saturated Montmorillonite and Kaolin clays”. Soils and Foundations, 1971, vol. 11 n°3, pp.1–22.

[20] A.Sachan and D.Penumadu. “Effect of Microfabric on Shear Behavior of Kaolin Clay”. Journal. of Geotech. and Geoenv. Eng., 2007, Vol. 133, No. 3. pp 306–318.