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Geetanjali Das, Andry Razakamanantsoa, Dimitri Deneele, Gontran Herrier. Hydraulic performance and microstructure evolution of kneading compacted lime-treated silty soil permeated with low-ionic strength solution.. 20th International Conference on Soil Mechanics and Geotechnical Engineering 2022, May 2022, SYDNEY, Australia. 6 p. hal-03629297

HAL Id: hal-03629297 https://hal.science/hal-03629297v1

Submitted on 4 Apr 2022

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Hydraulic performance and microstructure evolution of kneading compacted limetreated silty soil permeated with low ionic strength solution.

Performance hydraulique et évolution de la microstructure d'un sol limoneux compacté par pétrissage et imprégné d'une solution à faible force ionique.

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ABSTRACT: Durability of lime-treated earthen structures is governed by the hydromechanical performances of the soil. However, fewer studies address the influence of pore solution's chemistry on the mechanism governing the hydraulic and microstructural evolution of the materials. The hydraulic and leaching performances of lime-treated soil, compacted by kneading, and subjected to circulations of demineralized water (DW) or a low ionic strength solution (LW) are studied. The evaluation is made in terms of pore volume flow (PVF), as it considers the soil and pore fluid interactions which govern the leaching mechanism. During the hydraulic conductivity test, 40 PVF of DW is circulated through the specimens within a duration, which is 20 times lower than the one obtained from the LW-subjected soil. Such an accelerated circulation dissolute minerals contained along the flow path in the soil matrix in a short time. This mechanism increases the volume of macropores greater than 500 Å diameter. Increased macropores, thus, increased the magnitude of the hydraulic conductivity in DW-subjected soil. Besides, due to a longer contact duration between the treated soil and the LW than in DW, a greater formation of the cementitious compounds occurred in the former compared to the latter.

RÉSUMÉ : La durabilité des structures en terre traitée à la chaux est régie par les performances hydromécaniques du sol. Cependant, peu d'études abordent l'influence de la chimie de la solution interstitielle sur le mécanisme régissant l'évolution hydraulique et microstructurale des matériaux. Les performances hydrauliques et de lixiviation de sols traités à la chaux, compactés par pétrissage, et soumis à des circulations d'eau déminéralisée (DW) ou d'une solution de faible force ionique (LW) sont étudiées. L'évaluation est faite en termes de débit volumique des pores (PVF), car elle tient compte des interactions entre le sol et le fluide interstitiel qui régissent le mécanisme de lixiviation. Pendant le test de conductivité hydraulique, 40 PVF de DW circulent à travers les spécimens pendant une durée 20 fois inférieure à celle obtenue dans le sol soumis au LW. Une telle circulation accélérée dissout en peu de temps les minéraux contenus le long du chemin d'écoulement dans la matrice du sol. Ce mécanisme augmente le volume des macropores de plus de 500 Å de diamètre. L'augmentation des macropores, ainsi, a augmenté la magnitude de la conductivité hydraulique dans le sol soumis à la DW. En outre, en raison d'une durée de contact plus longue entre le sol traité et le LW que dans le DW, une plus grande formation des composés cimentaires s'est produite dans le premier par rapport au second.

KEYWORDS: lime-treated soil; pore solution; hydraulic conductivity; leaching; macropores

1 INTRODUCTION

Lime treatment of fine-grained soil is a widely implemented technique to improve the engineering properties of soil available in the worksites. Two significant mechanisms occur on adding lime with soil: (a) immediate cation exchange reactions and soil agglomeration, resulting in decreased soil plasticity and improved workability, and (b) pozzolanic reactions, mainly between calcium from lime and dissolved silica and alumina from the soil, resulting in the development of cementitious bonding and hence improving the hydromechanical performances of the soil in the long-term (Baldovino et al. 2019, 2018, Bell 1996, Das et al. 2020,2021, Dhar & Hussain 2019, Diamond and Kinter 1965, Little 1995, Rogers & Glendinning 199, Rosone et al. 2020).

The durability of a lime-treated earth structure is inter-linked with the hydromechanical performance of the structure during its service life. The hydromechanical behaviors of lime-treated soil were shown to be influenced by compaction conditions (Cuisinier et al. 2011, Das et al. 2021, Le Runigo et al. 2011, 2009), lime content (Baldovino et al. 2018, Rosone et al. 2019), and curing conditions (Lemaire et al. 2013, Verbrugge et al. 2011). Specimens compacted at different compaction moisture contents, energies were found to have a different magnitude of hydraulic conductivity (Le Runigo et al. 2009, Mitchell et al. 1965). Le Runigo et al. 2009 stated that lime-treated soil compacted on the wet side of optimum moisture content shows lower hydraulic conductivity than the one compacted on the dry side of optimum moisture content. Besides, the hydraulic conductivity of soil was demonstrated to be a function of macropore amount, which is influenced by the implemented compaction conditions (Ranaivomanana et al. 2018, 2017). Moreover, hydraulic conductivity and leaching of lime-treated soil were shown to increase with increased lime content (McCallister and Petry 1992, Tran et al. 2014). On the other hand, De Bel et al. 2005 stated that the leaching of lime could be minimized by increasing the curing time of treated soil after

compaction, which promoted the formation of cementitious compounds, thus minimizes the hydraulic conductivity.

Though studies were reported regarding several parameters that influence the hydraulic conductivity and leaching performances of lime-treated soil, whether the chemistry of pore solution can also influence these behaviors is a question. The influence of pore fluid chemistry on the hydraulic behavior of natural soil, particularly bentonites, is widely investigated. Bentonites, because of their favorable character as a low permeable material, are extensively used as a liner material in the disposal of the nuclear waste repository (Gapak et al. 2017, Razakamanantsoa et al. 2012, 2016, Sato et al. 2017). Razakamanantsoa et al. (2016) demonstrated the aggressive effect of leachates in the hydraulic behavior of natural bentonites in the long term. Similarly, Demdoum et al. (2020) confirmed the negative effect of landfill leachates on the hydraulic behavior of natural bentonites. Such studies confirm the necessity of considering the impact of pore fluid chemistry during the evaluation of the hydraulic and leaching performances of even lime-treated soil.

In most of the existing laboratory studies, DW was used as a conventional permeant solution to investigate the hydraulic and leaching behaviors of lime-treated soil, whereas in the field, water from a natural source influences such behaviors.

In this context, this study investigates the comparative hydraulic and leaching evolution in lime-treated soil subjected to DW and a LW to evaluate the chemistry of the pore fluid effect. The study was made on lime-treated soil, kneading compacted, at a laboratory scale. The first part of the study shows the differences observed in hydraulic conductivity, followed by leaching. The second part explains the preceding observation based on pore structure evolution.

2 MATERIALS AND SAMPLE PREPARATIONS

Silty soil, obtained from Marche-Les-Dames, Belgium, was treated with quicklime (calcium oxide, CaO). The Lime Modification Optimum (LMO) of the soil was 1 %, which was determined by Eades & Grim (1966) test (ASTM 2006). Lime content higher than the LMO, *i.e.*, 2.5% was used herein since soil treated at LMO was shown to have limited contribution towards long-term strength evolution (Das et al. 2021). Two permeant solutions, *i.e.*, DW and a LW solution composed with DW + 10^{-3} M NaCl were used. The use of 10^{-3} M NaCl was made to clarify the role of low ionic strength solution on the hydraulic performance and the microstructure of lime-treated soil. 10^{-3} M NaCl has been used previously (Razakamanantsoa et al. 2012, 2016, Sato et al. 2017) to highlight the negative effect of leachates on bentonites in the context of landfill.

The pH and Electric Conductivity (EC) of the permeant solutions are provided in Table 1.

Table 1. pH and EC of the permeant solutions

Permeant solutions	рН	Electric Conductivity (µs/cm)
DW	7.40	4
LW	6.53	172

The compaction characteristics of the soil obtained by Standard Proctor compaction as per ASTM D 698-12e2 (ASTM 2012) are presented in Table 2. The soil was first air-dried and then mixed with water to obtain soil paste. This soil paste was then mixed with 2.5% of lime, rested for 1 hour, and then compacted at the optimum moisture content (OMC).

Table 2. Maximum dry density and OMC of the soil				
Soil	Maximum dry density	OMC)		

Soil	dry density (kN/m ³)	(%)
Untreated silty soil	18.4	14.3
Silty soil + 2.5% lime	17.1	18.5

6 specimens, of which 3 untreated and 3 lime-treated of dimensions 0.05m in height and 0.05m in diameter were prepared by kneading (K) compaction. The motive behind conducting kneading compaction is to develop the kneading action the fine-grained soil experience under the pad-foot roller in the field (Das et al. 2021, Kouassi et al. 2000, Williams 1949). The process of kneading compaction was conducted, as demonstrated by Das et al. 2021.

Compacted specimens were then wrapped in plastic films and were cured at ambient laboratory temperature for 28 days.

3 METHODOLOGIES

3.1 Hydraulic conductivity test

At the end of the curing time, 4 specimens were subjected to hydraulic conductivity test. Of the 4 specimens, 1 untreated and lime-treated specimens were subjected to LW and the remaining to DW. The test was conducted using a constant head flexible wall permeameter. The setup of the hydraulic conductivity test was similar to the one used by Ranaivomanana et al. (2017), which includes the injection of permeant solution from the base of the specimen and collection of supernatants from the top. This is done to maintain a uniform flow in the specimen by removing any entrapped air if present. A confining pressure of 88 ± 2 kPa was applied for at least 24-48 hours before the application of hydraulic head pressure to ensure the homogeneity of the stress distribution. The hydraulic head applied was 170 ± 5 cm.

The hydraulic conductivity test was carried out in accordance with the flow conditions laid by Darcy's law. The test involves two phases: saturation and percolation. In the course of the saturation phase, each specimen was allowed to saturate until an inflow volume of permeant solution corresponding to 1 Pore Volume Flow (PVF) enter into the specimen. The volume of pore water required to renew the total water initially present in the soil is called 1 PVF (Katsumi et al. 2008). This is done to ensure the full saturation of the specimens. The concept of PVF was implemented herein to consider the duration of contact of the lime-treated soil with the pore solution, as such duration might contribute to the mechanism of dissolution or precipitation inside the soil. However, such a concept was used in several studies related to natural soil (Katsumi et al. 2008, Shackelford et al. 2000).

At the end of the saturation phase, the percolation phase was initiated, and the permeability coefficient, k was measured as a function of PVF. The hydraulic conductivity test was ended: (a) after 40 PVF of influent percolates through the specimen, and (b) the least 5 values of Electric Conductivity become almost constant.

The pH and ECs of the effluents collected during the percolation phase were measured. A part of the effluents was subjected to chemical analysis by Inductively Coupled Plasma Optical Emission Spectrometry (ICP OES) to determine the concentrations of Calcium (Ca), Silica (Si), and Alumina (Al), as these three elements are the most reactive during the pozzolanic reaction after lime treatment.

3.2 Pore structure determination

The pore structure of samples was analyzed by using Mercury Intrusion Porosimetry (MIP) test. The procedure of MIP test can be obtained from Romero and Simms (2008). The analysis was made on specimens sampled from the core of compacted soil obtained at the end of curing and at the end of the hydraulic conductivity test.

The pore classification was made as per the International Union of Pure and Applied Chemistry (IUPAC) (Rouquerol et al. 1994), which classifies pores based on their pore-width as macropores (> 500 Å), mesopores (20-500 Å), and micropores (< 20 Å).

In this study, the nomenclature used to represent the specimens is as follows: type of soil-compaction methodpermeant solution (percolation periods). For example, 2.5 %-K-DW (4 days) refers to 2.5 % lime-treated kneading compacted soil, percolated by DW for 4 days.

4 RESULTS

4.1 Evolution of hydraulic conductivity

Figure 1 presents the hydraulic conductivity evolution in the lime-treated and the untreated specimens, permeated by DW and LW. The presented hydraulic conductivity value, *i.e.*, the permeability coefficient, k represents the average of the last 10 values of k before reaching 40 PVF.

The k measured in 2.5 % lime-treated kneaded specimen, subjected to LW, was observed to be almost equivalent with the magnitude of k measured from the untreated specimens. Also, the period of percolation to reach 40 PVF was almost equivalent in these specimens, *i.e.*, 80-90 days. On the other hand, the magnitude of k was about more than an order higher for the corresponding specimen, permeated by DW with a percolation time of only 04 days to reach 40 PVF (Figure 1).



Figure 1. Comparative evolution of k in untreated and 2.5 % limetreated kneading compacted soil subjected to DW and LW.

4.2 Evolution of pH and EC in the effluents

The pH and EC of the effluents measured during the percolation phase are presented as a function of the number of PVF in Figure 2.



Figure 2. Evolution of pH and EC in the effluents collected from the DW and LW subjected specimens during the hydraulic conductivity test.

The pH of the effluents gathered from the untreated specimens was around 8 (Figure 2a). The pH of the effluents obtained from the lime-treated soil remained at about 12, irrespective of the type of permeant solutions the specimens were subjected to.

According to Figure 2b, the trend in the evolution of EC was similar for the untreated and the lime-treated specimens subjected to both types of solutions. The loss in EC was maximum up to about 10 PVF, and then the trend of decrease in EC became almost stable.

The initial EC of DW was 4 μ s/cm (as seen in Table 1). The measured EC from the effluents gathered from the untreated soil subjected to DW was significantly higher than the initial EC of the DW (Figure 2b). At the same time, this relative difference in EC was observed to be comparatively lower in the effluents collected from the LW submitted specimens. Besides, though the overall trend of evolution of EC was almost similar for both DW and LW leached lime-treated soil, the attained EC was obtained in only 04 days for the DW subjected soil while it took 80 days for the LW subjected soil to reach the same level.

4.3 Evolution of Ca, Si, and Al in the effluents

The concentration of *Ca*, *Si*, and *Al* elements leached from the lime-treated soil was measured from the effluents collected during the hydraulic conductivity test up to 40 PVF. The measured concentration is presented in Figure 3 as a function of PVF. The evolution of these elements was compared with the maximum concentration of the same measured in the effluent of the untreated soil, which was 100 mg/l, < 0.2 mg/l, and 8 mg/l for *Ca*, *Al*, and *Si*, respectively.



Figure 3. Comparative evolution of Ca (a), Al (b), and Si (c) in the effluents obtained from the untreated and 2.5 % lime-treated kneading compacted soil.

According to Figure 3a, 2.5% lime-treated kneaded specimen, leached by DW, showed a relatively greater loss in calcium when compared to the corresponding LW leached specimen. In the DW-subjected specimen, the concentration of Ca measured in the effluents were even lower than 100 mg/l, which is the maximum Ca concentration measured in the effluents of the untreated specimen leached by DW. Thus, this indicates that in addition to the loss of Ca coming from lime, particularly available around the flow path, a part of soluble calcium was also lost from the soil. However, the Ca leached from the corresponding LW submitted specimen was relatively lower and remained higher than 100 mg/l. The trend in the evolution of the concentration of Al measured in the effluents obtained from the lime-treated soil subjected to both types of solutions remained almost the same (Figure 3b). The obtained concentration of Al varied in the range of 4-6 mg/l during the hydraulic conductivity test. However, this concentration was much higher than what was measured in the effluents obtained from the untreated soil (< 0.2 mg/l). Thus, it can be said that the obtained Al in the effluents probably comes from the Al released from the soil due to increased soil pH or from the cementitious compounds formed during curing.

Figure 3c shows a significant loss in Si within the first 5 PVF, and the loss was then stabilized in both types of percolated limetreated soil. However, the maximum concentration of Simeasured was 8 mg/l from the effluents of the untreated soil, which was much higher than the one measured in the lime-treated soil's effluent. Thus, the obtained Si probably comes from the soluble Si present in the silty soil or from the cementitious compounds available in the soil.

Besides, although the observed trend in the loss of all the above minerals was almost similar in both types of percolated specimens with respect to PVF, it varies widely in the duration of percolation time. In DW permeated specimen, the above loss in minerals was achieved in only 4 days of percolation. On the other hand, it took 80 days to undergo an equivalent loss of similar minerals from the LW-subjected specimen.

4.4 Pore structure modifications

Hydraulic conductivity of compacted soil was demonstrated to be the function of macropores (Hunt and Sahimi 2017, Le Runigo et al. 2009). In this aspect, a comparative evaluation of the percentage of pore volumes greater and lower than 500 Å, obtained by MIP, was made for the unleached (after 28 days of curing) and the DW- and LW-leached specimens in Figure 4.



Figure 4. Comparative evolution of the percentage of pore volume in 2.5 % lime-treated unleached and DW- and LW-leached kneading compacted soil by MIP

According to Figure 4, the total percentages of macropores volume of diameter greater than 500 Å was maximum in the 28 days cured specimen (unleached). For the DW-and LW-leached specimens, these macropores' volume was lowered by about 8% and 12%, respectively. However, LW-subjected specimens showed the minimum presence of macropores.

Besides, the evolution of mesopores was comparatively maximum for the LW-subjected specimen. These mesopores volume were 5% and 12% higher in the LW-leached soil compared to the DW-leached and untreated soil, respectively.

5 DISCUSSIONS

The results evidenced that lime-treated kneaded soil subjected to pore fluids exhibiting different chemistry showed different magnitude of hydraulic conductivity and characteristics of leaching.

As mentioned in section 3.1 that lime-treated soil subjected to hydraulic conductivity test underwent two phases, *i.e.*, saturation and percolation phase, respectively. In the present case, during the saturation phase, the development of cementitious bonding was enhanced due to constant contact of soil-lime with the pore solution and also due to the limited flow of the pore solution. The preceding statement was evident by the decrease and increased in macropores and mesopores, respectively, in the leached specimens compared to the unleached (Figure 4). Thus, a rise in hydromechanical performance can be expected.

Once the percolation phase was initiated, the renewal of pore water occurred. During the percolation phase, the EC measured in the effluent obtained from the DW subjected untreated soil was relatively higher than the initial EC present in the DW (Figure 2b). On the other hand, this feature was less significant in the corresponding LW-subjected soil. In the lime-treated soil, the time required to renew 40 PVF was 20 times lower in the DW-subjected soil compared to the corresponding LW-subjected soil (Figure 1). Thus, a quick renewal of the pore solution occurred in the DW-subjected, which created accelerated leaching of Ca, Si, and Al, as evident from Figure 3. The aboveobserved difference in the chemical characteristic of effluent obtained between DW-and LW-subjected soil is attributed to the electronically unstable nature of DW. DW being devoid of any ions, leached more minerals in a short time in the process of attaining its required ionic equilibrium. Thus, enhanced leaching of lime components under the influence of DW, particularly available around the flow path, triggered an increase in macropores of diameter greater than 500 Å (Figure 4). As a result, the hydraulic conductivity of LW-subjected soil was similar to the untreated soil, whereas it was about more than an order higher in the DW-subjected soil (Figure 1).

Based on the above discussion, it was confirmed that the analysis of the hydraulic performances of the lime-treated soil against pore solution is not only a function of permeation time but must include the mechanism of leaching and the rate of contact of the pore fluid within the soil matrices. Thus, the analysis is required to be made in terms of PVF, where the number of times the pore water is renewed can be measured.

Besides, the pozzolanic reactions developed between soillime and pore water depend on the duration of contact between both the components. As a result, due to the longer contact of LW with the soil during the hydraulic test, a greater generation of cementitious compounds occurred. This was evident by the evolution of a greater number of mesopores in the LW-subjected specimens (Figure 4). Such an evolution is beneficial for the long-term durability of lime-treated soil.

6 CONCLUSIONS

This study investigates the evolution of hydraulic conductivity and leaching mechanism in lime-treated compacted silty soil on being subjected to demineralized water and low-mineralized solution. Based on the investigations, the following conclusion was derived:

1) Demineralized water increased the hydraulic conductivity and accelerated the leaching mechanism in lime-treated soil compared to the low-mineralized solution. Demineralized water, being devoid of any ions, dissolute a relatively greater concentration of lime components, particularly available around the flow path. This increased the macropores of diameter greater than 500 Å resulting in increased hydraulic conductivity.

2) The analysis of the hydraulic performances of the lime-treated soil against pore solution is required to be made as a function of pore volume flow. Measuring pore volume flow considers the number of times the pore water is renewed in the lime-treated soil, which governs the mechanism of leaching influenced by the pore solution chemistry.

3) The duration of contact between the soil components and the permeant solution impact the development of pozzolanic reactions during the hydraulic conductivity test. A longer contact of LW with the soil component enhanced the evolution of cementitious compounds. Such an evolution contributes towards the long-term durability of lime-treated soil.

Thus, the study showed that the 10^{-3} M of NaCl concentration added was sufficient enough to modify the hydraulic, leaching performance, and microstructure of lime-treated soil. Based on these findings, it is important to consider the chemistry of the permeant solution chosen at a laboratory scale. The selected permeant solution must be representative of the pore water that the structure is likely to be subjected to in the field. This can help to give an accurate prediction of the long-term hydraulic performance of in-situ hydraulic structures.

7 FUTURE STUDY

Further studies are going on in the laboratory to understand if such a difference in the permeant solution can also impact mechanical behavior in the long term. Besides, these behaviors will also be compared with lime-treated soil prepared at different lime contents.

8 ACKNOWLEDGEMENTS

This work was financially supported by Association Nationale de la Recherche et de la Technologie with grant N°2018/0219 and Lhoist Southern Europe with grant N°RP2-E18114. The authors are very thankful to the research team of Université Gustave Eiffel, and Lhoist R&D for their great support in performing laboratory experiments and technical supports.

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