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On the ICL test in soil stabilization

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***Abstract* - Some considerations upon the Initial Consumption of Lime (ICL) test, arisen from its use in the lime treatment of a pyroclastic soil, are presented. In the investigated material there are no ion-exchanging minerals, such as zeolites. Therefore when this soil comes into contact with lime, no ion exchange processes are expected, and lime consumption is due to pozzolanic reactions. The standard ICL test was performed on this material treated with different lime percentages, and pH value was checked for the following 14 days. Atomic absorption analyses of unconsumed lime have been performed on under-saturated lime solutions of the same soil. Data showed significant consumption of lime, due to pozzolanic reactions, as soon as lime comes into contact with the soil, influencing both Ca^{2+} concentration in solution and pH. Experimental data and theoretical considerations suggest that pH value could be not diagnostic in determining the amount of lime required to satisfy the soil affinity for lime if ion exchanging minerals were present. In fact when pozzolanic reactions develop in the very short term, pH measurement does not allow discerning the amount of lime consumed by pozzolanic reactions and by cation exchange.**

I. INTRODUCTION

Lime stabilization is one of the most popular methods used worldwide to improve mechanical characteristics of clayey soils. Recent studies have investigated the possibility of using this technique for stabilization of pyroclastic soils, generally unsuitable for earthworks in their natural state [9].

The scope of this work is to show some previously undiscussed limitations of the widely used Initial Consumption of Lime (ICL) test [2]. These limitations have arisen from the use of this test in the lime treatment of pyroclastic soils, but it seems that they could also affect the results of ICL test when applied to clayey soils.

The ICL test is one of the most common methods used to determine the minimum amount of lime necessary for soil stabilization (i.e. for the occurrence of pozzolanic reactions). This test was proposed by Eades and Grim [10] and it is based on assumption that cation exchange between soil cations and Ca^{2+} cations, available from dissociation of lime, is completed within one hour. The pH value of lime-soil-water solutions is measured after one hour of curing; if such value is as high as 12.4 (corresponding to the pH of a lime saturated solution) or slightly lower, it is assumed that lime is still available for pozzolanic reactions. The maintenance of highly alkaline conditions is necessary for the dissolution of clay silicates, making silica and alumina available for pozzolanic reactions [3]. If after one hour curing the solution pH is lower than 12, it is considered that lime have reacted with the soil and then calcium silicates hydrates cannot be formed. This occurrence is commonly interpreted by assuming that the lime percentage added to the soil was not high enough to satisfy its cation exchange capacity. This would be confirmed by the fact that soil plasticity reaches its minimum when the lime percentage is high enough to maintain the pH at a stable value of 12.4 [10]; this percentage corresponds to the lime fixation point (LFP) as defined by Hilt and Davidson [11].

Rogers et al. [13] amended the standard ICL test pointing out the practical drawbacks deriving from pH measurements. The Authors have shown how the pH is relevantly depending on the measurement method, affecting the ICL values; they pointed out also the effects of lime slaking during storage, altering the reactivity of the system and the pH measurements. They finally have suggested to consider the evolution of pH over time rather than concentrating on a single pH value. Some inconsistencies in the ICL test results applied to clayey soils were also shown by Cambi et al [5].

As previuously stated, in this work ICL test was performed on pyroclastic soils. In these materials the fine fraction can be very variable and generally the efficiency of lime treatment mostly depends on the pozzolanic reactions, especially when exchanging minerals, such as zeolites, are absent. This is the case of the pozzolana considered in this study, known as Pozzolana Nera (PN), belonging to the stratigraphic profile of the Colli Albani volcanic complex, in the south-east area of Rome [6], [7].

II. MATERIALS AND METHODS

Previous studies on the mechanical behaviour of lime treated PN soil [9] showed the high reactivity of this material to lime and the relevant mechanical improvement when the soil was treated with 10% of lime by dry weight

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of soil. Such lime percentage provided the maintenance of a stable and highly alkaline pH for long time [8].

In this work the PN was characterized by means of X-ray diffractograms, to determine its mineralogical composition. The standard ICL test was then performed on samples of PN, treated with different percentages of lime. The pH of solutions was measured after one hour curing, as required by the standard test, and then checked for the following 14 days. For allowing a deeper comprehension of the chemico – physical processes occurring after the addition of lime to PN, atomic absorption analyses of unconsumed lime have been performed on PN samples treated with lime.

X-ray diffraction analyses allowed to define whether the high reactivity of PN soil with lime could be due to the presence of ion-exchanging minerals, such as zeolites. The results of X-ray analyses have been reported in Table 1, where it is shown that zeolites are not present in PN soil.

Table 1. Mineralogical composition of the examined pozzolana

phase	weight %
Biotite	2.57
Analcime	4.80
Leucite	27.22
Muscovite	5.26
Sanidine	1.82
Anortite	2.70
Calcite	0.11
Augite	16.18
Albite	6.70
Illite	4.07
Gypsum	1.87
Amorphous	*26.7*

Differently from clayey soils, when PN comes into contact with lime, ion exchange processes involving Ca^{2+} cations are therefore not expected.

Following the ICL standard test, soil-lime-water solutions were prepared with 25 g of dry soil added with lime between 2% and 6% of dry weight of soil. In order to avoid that lime slaking could alter the solution [13], hydrated lime was used to prepare the soil-lime mixture, in accordance with Eades and Grim [10]; 100 ml of water were then added to each mixture to form the solution, which is therefore supersaturated in lime (lime solubility in water at 20 °C is 0.173g/100ml). The pH of solutions was measured after one hour curing and then checked for the following 14 days.

Atomic absorption analyses were carried out to determine the unconsumed lime. To this purpose, four soil-lime-water solutions were prepared, under-saturated in lime, by using 0.4 g of lime and an amount of dry PN soil such that lime percentages were equal to 2%, 5%, 10% and 20%. 500 ml of distilled water were added to each mixture. The concentration of lime was then lower than its solubility, and the product of ions concentration was lower than lime K_{ps}. Table 2 shows quantity details of soil-lime-water solutions.

Table 2. Water, lime and soil quantities used to prepare the solutions unsaturated in lime

Water (*ml*)	Lime (*g*)	pozzolanic soil (*g*)	$Ca(OH)_2$ weight %
500	0.4	20	2
500	0.4	8	5
500	0.4	4	10
500	0.4	2	20

The amount of Ca^{2+} cations in solution (expressed in ppm) was evaluated at different curing times.

III. RESULTS

Results of the standard ICL test are shown in Table 3, where pH values of the soil-lime-water solutions for one hour of curing and for longer curing times have been reported.

Table 3. Values of pH for pozzolana-lime-water solutions after one hour curing.

$Ca(OH)_2$ (weight %)	1 h	1 day	2 days	6 days	8 days	14 days
2	11.76	12.3	12.14	11.99	11.96	11.47
3	11.83	12.4	12.26	12.11	12.08	11.81
4	11.85	12.48	12.33	12.16	12.16	11.96
5	11.88	12.49	12.37	12.20	12.17	12.09
6	11.90	12.5	12.39	12.21	12.18	12.16

Fig. 1 shows the pH values recorded after one hour curing for the standard solutions (lime percentage ranging between 2% and 6%).

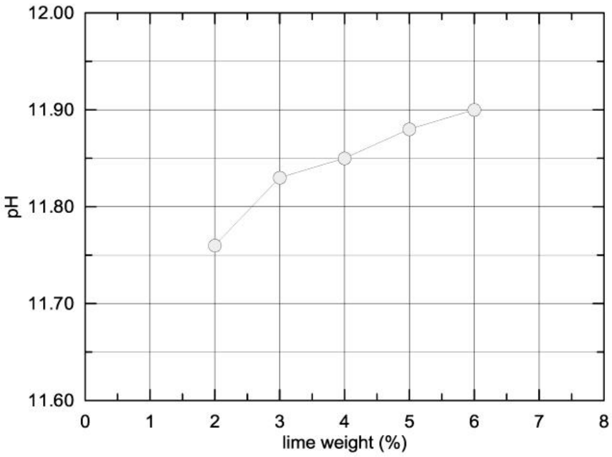

Fig. 1 - Value of pH for pozzolana-lime-water solutions after one hour curing.

For each studied solution, the pH measured after one hour curing never reached the saturated lime solution pH (12.4), although the amount of lime used was larger than the maximum lime solubility. The results highlighted also an increase of pH with increasing lime percentages.

Fig. 2 shows the evolution of pH of the different solutions in the following 14 days.

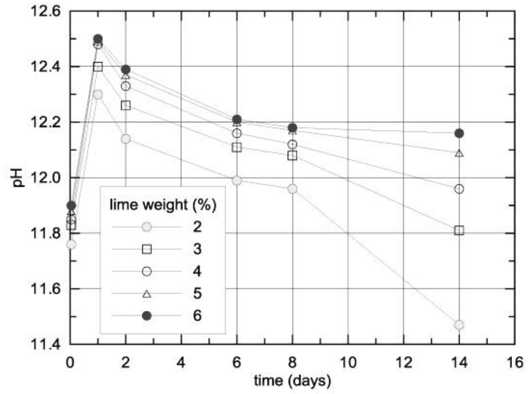

Fig. 2 - Evolution of pH for pozzolana-lime-water solutions over 14 days.

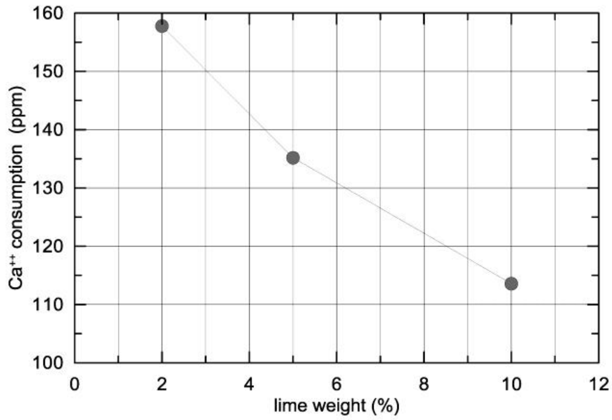

Fig. 3 - Ca^{2+} consumption after one hour curing for different lime percentages

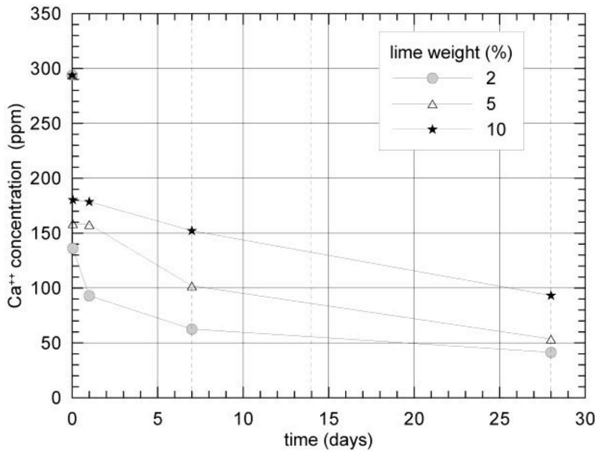

Fig. 4 - Consumption of Ca^{2+} cations with time for different lime percentages.

For the studied solutions, the pH reaches approximately the values of the saturated lime solution after one day of curing. Afterwards the pH drops for the next 14 days: the lower the lime percentage in the mixture, the faster the decrease in pH. These results suggest that lime consumption in the first hour of curing is very fast and, even though the solutions are super-saturated in lime, the pH does not reach the expected 12.4 value within the first hour in any of the examined solutions. Since there are no ion-exchanging minerals, lime consumption can only be ascribed to pozzolanic reactions. After one day of curing, the pH of the solutions is increased up to the value expected for a saturated lime solution (Figure 2), before dropping again as the reactions continue. This suggests that lime consumption is slowed down, so that pH can reach the value expected for a saturated lime solution, until lime dissolution is completed.

To validate the indications obtained by pH measurements, atomic adsorption analyses were carried out to determine the amount of Ca^{2+} ions in the solutions at different times. As stated previously, for this purpose few solutions under-saturated in lime were prepared. Since the solutions were prepared by using the same amount of lime and water, an equal initial concentration of dissolved Ca^{2+} cations were provided for each solution, equal to 294 ppm. Fig. 3 and Fig. 4 show respectively the Ca^{2+} consumption after one hour of curing for each solution and the evolution of Ca^{2+} concentration in the following 28 days.

The results showed a higher Ca^{2+} consumption (in absolute value) during the first hour for lower lime contents. This datum is consistent with the lower pH values measured after one hour curing for lower lime concentrations. In the following days, the lime concentrations measured for different solutions followed approximately the same trend, and lime consumption slowed down. Therefore, it can be stated a good correlation between pH and Ca^{2+} consumption measurements.

When lime consumption is very fast, such as in the first hour, lime dissolution is not fast enough for the pH to reach the pH expected for a saturated lime solution. When lime consumption slows down pH increases up to about 12.4 since the consumption of lime is compensated by lime dissolution.

IV. DISCUSSION

Cation exchange and pozzolanic reactions are the main physico – chemical processes induced by the addition of lime. Cation exchange occurs when ion-exchanging minerals are present in the soil, such as in clays, consuming only Ca^{2+} cations, while the entire lime molecule is involved in pozzolanic reactions.

As previouly stated, in the ASTM D6276-99a test, the soil-lime-water solution is prepared by adding 100 ml of water to 25 g of dry soil, mixed with hydrated lime between 2% and 6% of dry weight of soil. The minimum amount of lime added to the soil is therefore 0.5 g, guaranteeing the solution super-saturated in lime (lime solubility in water at 20 °C is 0.173g/100ml and its solubility product constant K_{sp} is 4.68×10^{-6}). The solution is alkaline enough to guarantee the dissolution of silica and alumina from silicates, the pH of a saturated lime solution being 12.4.

In the standard test, the pH of solution is measured after one hour curing. As the pH corresponds to the (OH^-) concentration, its value is only influenced by lime consumption due to pozzolanic reactions, as cation exchange does not affect (OH^-) concentration [1]. Since the solution is always super-saturated in lime, the presence of undissolved lime should guarantee the maintenance of pH at 12.4 until lime dissolution is complete, unless lime consumption due to lime-soil reactions is faster than lime dissolution. Values of pH lower than 12.4 in the first stage of lime-soil reactions seem to be attributable to fast reactions involving the whole lime molecule, and influencing the (OH^-) concentration.

When ion-exchanging minerals are present, in the first stages of lime-soil reactions is then difficult to establish, from pH measurements, the amount of lime consumed by pozzolanic reactions and the amount consumed by cation exchange. Experimental evidences from the investigation performed on treated PN showed that the lime consumption due to pozzolanic reactions is already significant in the very first stage of the treatment.

CONCLUSIONS

The interpretation of ICL test on a lime treated pyroclastic soil has been discussed. Experimental data showed that a significant consumption of lime takes place when lime comes into contact with silicate (within one hour) influencing both the solution Ca^{2+} concentration and pH. In materials where ion-exchanging minerals are not present, such as the PN soil, the consumption of lime can only be ascribed to pozzolanic reactions. In the investigated material pozzolanic reactions are immediate, similarly to other clays containing active minerals, such as montmorillonite [3], [4], [12]. These indications, together with the considerations exposed above, suggest that pH of the solution in the standard ICL test could be not diagnostic in determining the amount of lime required to satisfy the soil affinity for lime when ion-exchanging minerals are present. In presence of such minerals, it is difficult to establish, on the basis of pH measurements, the amount of lime consumed by pozzolanic reactions and the amount consumed by cation exchange. Indeed these two processes seem to occur simultaneously.

Despite of the wide use of this test in practice, the aforementioned data and considerations highlight a further flaw of the standard ICL test, to be added to the already pointed out practical drawbacks [13], [5]. For materials similar to the PN soil, the ICL test should not be interpreted conventionally. Nevertheless, by checking the evolution of pH values over an extended time interval, a useful indication on the development of pozzolanic reactions can be obtained.

Alternative tests for determining the minimum lime percentage for the activation of pozzolanic reactions should be taken into account in presence of exchanging minerals. Due to the rapid execution and simplicity, Cambi et al. (2012) suggested the use of the methylene blue absorption (MBA) test in determining the lime affinity of clayey soils, showing its higher accuracy with respect to the ICL test.

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