EVOLUZIONE DEI FENOMENI FISICO-CHIMICI INDOTTI DALLA STABILIZZAZIONE A CALCE DI UNA PIROCLASTITE DELL'ITALIA CENTRALE

CHEMO-PHYSICAL AND MECHANICAL INVESTIGATION OF THE EFFECTS INDUCED BY LIME TREATMENT ON A PYROCLASTIC SOIL

Cambi C., Cecconi M., Guidobaldi G., Pane V.
Università di Perugia
costanza.cambi@unipg.it; manuela.cecconi@unipg.it; giulia.guidobaldi@studenti.unipg.it;
vincenzo-pane@unipg.it

Russo G., Vitale E.
Università di Cassino e del Lazio Meridionale
giarusso@unicas.it

Deneele D., Paris. M.
Institut des Matériaux Jean Rouxel (IMN), Université de Nantes, CNRS
Dimitri.Deneele@cnrs-imn.fr

Abstract

A zeolite-rich pyroclastic soil coming from Central Italy was chosen for an extensive experimental study aimed to investigate the effects induced by lime addition on the soil geotechnical and chemo-mineralogical properties. Conventional direct shear tests, performed on raw and lime treated soil samples, showed an increase of both shear strength properties and dilatancy resulting from lime addition. The chemical, physical and mineralogical evolution of the soil-lime system was monitored over time by X-Ray Powder Diffraction, ThermoGravimetry and Nuclear Magnetic Resonance. The multi-scale study approach allowed to investigate the link between the microstructural features and the observed mechanical improvements. The geotechnical beneficial effects are mostly due to soil high reactivity to lime addition, quick triggering of pozzolanic reactions and formation of secondary phases.

1. Introduction

The necessity to reduce costs and environmental effects of large infrastructures promoted the development of methods based on treatment and reuse of local material, unsuitable in their natural state because of the low geotechnical features. In earthwork practice, problematic clayey soils are commonly treated with lime stabilization, inducing mechanical improvements. Pyroclastic soils, widely spread in Central and Southern Italy, are not used in the construction field. Even though, natural pozzolanas are known to be highly reactive to calcium oxide addition, their exploitation is limited by the intrinsic complexity in terms of nature, heterogeneity, microstructure features and saturated and unsaturated hydro-mechanical behaviour. The suitability of pyroclastic soil to lime treatment was recently studied by Cecconi e Russo (2013) and Russo et al. (2016), who focused on the influence on the engineering features of parameters such as lime per cent by weight, compaction energy, initial water content and curing time. The present study aims to highlight the mechanical and chemo-mineralogical effects of lime treatment on a zeolite rich pyroclastic soil, thoroughly investigating the relationship between macro and micro modifications following the addition of calcium hydroxide. The considered pyroclastic soil, coming from the Orvieto cliff (Central Italy), is
particularly abundant in Chabazite, a calcium enriched zeolite. A thorough characterization of the chemo-physical- mineralogical features of the raw soil was carried out and it is discussed in a companion paper presented to this IARG2017. Direct shear tests on reconstituted samples of raw and lime treated soil with different percentages of lime (2% and 5% Ca(OH)₂) at increasing curing times (24 h, 7 and 28 days) allowed a quantitative analysis of the mechanical improvement. The interpretation of the mechanical behaviour was supported by the chemo-physical outcomes. To emphasize the system reactivity and make more evident the system evolution, samples undergoing microstructural analysis were prepared with high lime content.

2. Experimental procedures and equipment

The material was gathered in blocks from the sampling site (see Cecconi et al., 2017a), crumbled by hand in a mortar until passing the sieve 450 μm and oven-dried for 24 h at 60 °C. Hydrated lime, containing more than 95% of Portlandite (Ca(OH)₂), was used for the treatment. Direct shear tests were performed on remoulded samples of raw and lime treated OR soil. The ground dry soil was mixed with two different lime percentages, namely 2% and 5% by dry weight of soil, and 20% of distilled water. The material was compacted in a standard 60 × 60 mm shear boxes in n.3 layers by using a hollow cylindrical mallet of mass 850 g, sliding along a vertical bar. The adopted procedure is the same used for the preparation of oedometer samples (see Cecconi et al., 2017). For the compaction of each soil layer, the mass dropped from 0.40 m of height (n.10 consecutive blows). The samples were sealed in plastic bags to avoid contact with atmosphere, and stored at room temperature for 24 hours, 7, 28 days of curing before testing. Direct shear tests were performed at a displacement rate of 0.133 mm/min. All tests were performed in unsaturated conditions, but no suction measurements were performed before and during the tests; different increasing stress levels were applied, namely 50 kPa, 100 kPa, 200 kPa.

In order to highlight the pozzolanic activity of the system, for microstructure analyses, the ratio between dry weight of lime and OR soil was kept equal to 1:1. Soil and lime were thoroughly mixed and distilled water was added with a water/solid ratio equal to 1. The samples were stored in sealed plastic containers to avoid carbonation of lime, and cured for 1, 7, 14, 28 days in a controlled environment at constant humidity and room temperature. Before testing, the specimens were dehydrated by freeze-drying technique (Delage and Pellerin, 1984). X Ray Diffraction (XRD), Thermogravimetric Analysis (TGA), 29Si Nuclear Magnetic Resonance Spectroscopy (29Si NMR) analyses were performed.

3. Results

3.1. Chemo-physical evolution of lime treated OR soil

The bulk mineralogical composition of OR soil was determined by X-Ray Powder Diffraction (black spectrum in Figure 1, see Guidobaldi et al., 2017). Its quantitative analysis reveals the prominent presence of the zeolite Chabazite (46.3%), whose abundance is furtherly proved by DTG and NMR spectra, heavily affected by this calcium enriched zeolite. The presence of Chabazite - from DTG results is indicated by two typical mass loss at 75 °C and 150 °C (see the black line in Fig. 2), suggesting the presence of two different water absorption sites (Stakebake, 1984). In NMR spectrum (black spectrum, Fig. 3) Chabazite characteristic peaks (Trush and Kuznicki, 1991) are clearly recognizable at −87.61 Q4(4Al), −94.0 Q4(3Al), −99.4 Q4(2Al), −104.8 Q4(1Al), −110 ppm Q4(0Al).
X-Ray Powder Diffraction spectra (Fig. 1) of treated OR samples at increasing curing times (namely 1, 7, 14, 28 days) show that, due to high amount of added lime, reflections of unconsumed Portlandite are steadily detected until 28 curing days. The intensity of Portlandite peaks decreases over time, suggesting that the reactions taking place in the system involve a consistent amount of lime. Chabazite reflections show an evident time-dependent intensity decrease. The formation of a new mineralogical phase, namely Monocarboaluminate hydrate (C4AICH11), is recognizable since 7 days of curing and the intensity of the peak steadily increases in time. The results obtained from thermogravimetry are consistent with XRD outcomes. Figure 2 shows that after treatment the two main peaks of Chabazite slowly decrease in time. The formation of hydrated produces a progressive increase of the % mass loss in the range of temperature of 100 °C. Starting from 14 days the formation of new hydrated phases ~300 °C (typical temperature range of C-A-H) (Farage et al., 2003; Vedalakshmi et al., 2003) is detected. The interpretation of 29Si NMR spectra allowed an insight into the secondary phases structure (Fig. 3). Initially, the hydrates are mainly composed by Q1, while Q2 as well as Q2(1Al) polymerization takes place for longer curing times.
3.2. Mechanical improvement: shear strength properties of lime treated OR soil

Direct shear tests were carried out on raw and treated samples of OR soil (Ca(OH)$_2$=2%, 5%). Figures 4 (a, b, c) show the stress-strain behaviour observed for treated compacted samples (see Table 1) after increasing curing times, namely 24 h, 7 and 28 days. The results have been represented as a function of vertical confining stress ($\sigma_v$=50, 100 and 200 kPa). The observed behaviour clearly depends on lime percentage (2, 5%), on curing time (1, 7, 28 days) as well as on the applied stress level (50 ÷ 200 kPa). For the whole range of investigated stress levels, all treated samples exhibit a brittle behaviour denoted by the attainment of a peak resistance and following softening towards a stable condition at which volumetric strains still develop at approximately constant shear stresses. This behaviour is systematically associated to soil dilatancy. At increasing stress level ($\sigma_v$=200 kPa), the brittleness of the material increases while, on the other hand, the tendency of the soil to dilate reduces; this occurrence is rather remarkable for the samples treated with the largest amount of lime (5% Ca(OH)$_2$). With regard to the influence of curing time on the shear strength, from the inspection of Figure 4, it can be noted that for the samples treated with lower lime-percentages (2% Ca(OH)$_2$) the maximum values of the peak shear strength are attained within a week (7 days) from lime addition, independently of applied stress level. On the other hand, for larger lime amounts (5%), the longer is the curing time, the higher is the increase of the peak shear strength. Also, for these samples treated with the addition of high lime percentage, the drop of shear strength is definitively abrupt, differently from that observed for samples treated with 2% Ca(OH)$_2$.

Finally, it is worth to note that at larger horizontal displacements ($\delta x$ = 7 mm), when a stable condition is attained, both the raw samples and the treated samples with 2% Ca(OH)$_2$ exhibit – approximately – the same shear strength; this is true for all the investigated stress levels. This finding can be also extended to the treated soil at 5% Ca(OH)$_2$ but only for short curing time, up to 7 days, and large vertical confining stress (200 kPa).

<table>
<thead>
<tr>
<th>$w$ (%)</th>
<th>$G_s$ (-)</th>
<th>$\gamma$ (kN/m$^3$)</th>
<th>$\gamma_d$ (kN/m$^3$)</th>
<th>$S_r$ (%)</th>
<th>$e$ (-)</th>
</tr>
</thead>
<tbody>
<tr>
<td>22</td>
<td>2.43</td>
<td>15.8 ± 1.53</td>
<td>13.0 ± 1.25</td>
<td>60.7 ± 12.9</td>
<td>0.88 ±0.18</td>
</tr>
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*Table 1. Physical properties of OR reconstituted samples*
4. Discussion and concluding remarks

A thorough investigation of the chemo-physical evolution of the lime-soil-water system was performed with the purpose of linking such evolution to the macroscopic mechanical improvements obtained for 2% and 5% lime treated OR soil. In order to highlight the ongoing modifications at particle level, soil and lime were mixed in the same weight proportions. Since the very short time, the highly alkaline environment induced by lime addition favours silica and alumina dissolution which in turn, coupled with the availability of calcium ions, leads to the formation of secondary hydrates phases. The newly formed crystalline phases were identified by XRD only after 7 days of curing, whereas they could be identified at the very short term by DTG. The consumption of existing mineralogical phases is detectable since the very short term. The height of the peaks decreases simultaneously with the growth of hydrated phases. The observations suggest that Chabazite and the amorphous phase play an active role in the evolution of the lime-soil system and hydrates formation. A further confirmation of the kinetic and the features of the system evolution is given by the quantitative interpretation of the results obtained from thermogravimetric analyses in Figure 5, where the mass losses of treated samples at increasing curing time in the range of temperatures typical of Portlandite and hydrated phases have been reported. The decrease of Portlandite mass loss in the very short term is relevant (about 17%) and continues during the time interval considered. The increase of mass losses in the temperature range of hydrates phases indicates that increasing amount of hydrates are forming over time, with particular high rate in the short term. The formation of cementitious compounds derived from the pozzolanic activity is the most relevant effect due to lime addition. The role of these new formation phases is revealed by the noticeable increase of soil dilatancy for the treated material which, in turn, is responsible of its brittle behaviour observed upon shearing. Therefore, the main findings allow the following conclusions to be drawn:

- Portlandite, Chabazite and the amorphous phase are the main phases involved in pozzolanic reactions induced by lime addition;
- secondary hydrated phases are formed since the very short time in amorphous state, as highlighted by comparing diffractometric and thermogravimetric analyses;
- the recognized secondary phases are mainly C-A-S-H, even though other forming cementing
Figure 5. Lime treated OR soil. Mass loss percentage due to dehydration of secondary hydrated phases and Portlandite consumption at increasing curing time

- phases have been identified, as evidenced by 29Si NMR spectroscopy;
- the mechanical behaviour of treated soils depends on applied stress level, amount of added lime and curing time, and is brittle and dilatant; these features are more evident for higher lime contents, longer curing times and larger stress levels;
- the formation of hydrated secondary phases affects the observed mechanical behaviour upon shearing. In particular, the amount of lime, and therefore of newly formed hydrated phases, increases the peak shear strength; the dilatant aliquot of the shear strength is increased by the formation of hydrated secondary phases, giving rise to bonding effects between grains;
- for considered curing time intervals, friction angles of the lime treated soil are not influenced by the amount of lime added to the sample and hence by the amount of secondary phases, presumably because of their low crystallization degree.

Bibliografia


Cambi C., Cecconi M., Guidobaldi G., Pane V., Russo G., Vitale E., Deneele D., Paris M.