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Enza Vitale, Dimitri Deneele, Giacomo Russo. Multiscale analysis on the behaviour of a lime treated bentonite. *Procedia Engineering*, 2016, VI Italian Conference of Researchers in Geotechnical Engineering, CNRIG2016 - Geotechnical Engineering in Multidisciplinary Research: from Microscale to Regional Scale, 22-23 September 2016, Bologna (Italy), 158, pp.87-91. 10.1016/j.proeng.2016.08.410 . hal-01775078

**HAL Id: hal-01775078**

**<https://hal.science/hal-01775078v1>**

Submitted on 27 Apr 2018

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VI ITALIAN CONFERENCE OF RESEARCHERS IN GEOTECHNICAL ENGINEERING –  
Geotechnical Engineering in Multidisciplinary Research: from Microscale to Regional Scale,  
CNRIG2016

## Multiscale analysis on the behaviour of a lime treated bentonite

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### Abstract

An insight into the microstructural evolution of clay-lime-water systems and its influence on the hydro-mechanical behaviour of treated bentonite has been presented. The time-dependent mineralogical and microstructural changes induced by lime addition on a natural bentonite have been investigated in order to provide a better understanding of the kinetic of the ongoing reactions. The effects of lime addition on the clay particle arrangements have been inferred from dynamic light scattering measurements, whereas the mineralogical changes have been monitored at increasing curing time by X-Ray diffraction and thermogravimetric analysis. The bentonite showed a high initial reactivity characterized by a rapid and total consumption of lime, which promoted the formation of new hydrated phases since the very short term (0 days of curing). Oedometer tests on not treated and lime stabilised saturated samples have been performed at increasing curing times. The results showed the combined effect of clay particles arrangement and bonding on the mechanical behaviour of treated samples.

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Peer-review under the responsibility of the organizing and scientific committees of CNRIG2016

*Keywords:* Lime treatment; microstructure; pozzolanic reactions; hydro-mechanical behaviour

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### 1. Introduction

The addition of lime to a clayey soils has a strong impact on the geotechnical properties of the soils as results of

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the chemical reactions which take place after the treatment. In the short-term, the exchange of surface cation by calcium leads to flocculation of clay particles. In addition, the high alkaline environment induced by lime promotes the dissolution of siliceous and aluminous compounds from clay mineral lattice with the precipitation of secondary phases as a result of the development of the time-dependent pozzolanic reactions [1-5]. In order to provide a better understanding of the link between the ongoing of the reactions induced by lime and the macroscopic evolution of soil properties, a multi-scale investigation on the short and long term effects of a lime treated bentonite has been performed.

The effects of lime addition on the mineralogical changes have been monitored at increasing curing time by X-Ray diffraction and thermogravimetric analysis, whereas the clay particle arrangements have been inferred from dynamic light scattering measurements. The experimental evidences at particle level have been complemented by investigations at macroscopic level. The compressibility of not treated and lime stabilised samples have been investigated as a function of lime content and curing time by means of 1D compression tests.

## 2. Materials

Natural bentonite clay mainly composed of Ca-montmorillonite was supplied by SSB Srl, Sardinia, Italy. The specific gravity is  $G_s = 2.54$ , and surface area determined by nitrogen adsorption (BET) is  $99.38 \text{ m}^2/\text{g}$ . The pH value of the soil is about 7.55. The liquid and plastic limits are respectively 143% and 69%, with a plasticity index IP equal to 74%. The soil is mainly formed by Ca-montmorillonite, with minor amount of quartz and calcite. The chemical composition of bentonite is reported in Table 1.

Table 1. Chemical composition of bentonite

Constituent	Percentage (%)
SiO <sub>2</sub>	64.85
Al <sub>2</sub> O <sub>3</sub>	24.33
CaO	2.14
K <sub>2</sub> O	0.21
MgO	3.18
FeO	4.21

## 3. Experimental procedures

### 3.1. X-Ray diffraction analysis (XRD)

X-Ray diffraction analyses have been carried out on not treated and 3%CaO treated samples at increasing curing times, namely 0, 3, 7, 14, 28, 60, 90, 120 days. Samples were freeze-dried before testing [6]. XRD patterns were performed on randomly oriented powder using a Bruker AXS D8 Advance Diffractometer with  $\text{CuK}\alpha$  ( $\lambda=0.154 \text{ nm}$ ) radiation and a step size of 0.021 degrees.

### 3.2. Thermo-gravimetric analysis (TGA)

The mineralogical changes induced by lime addition have been studied by means of thermo-gravimetric analysis (TGA). This technique provides a measure of the mass loss of the sample under a controlled atmosphere as a function of increasing temperature at constant heating rate. The change in mass is determined by the dehydration or decomposition of a mineralogical phase.

Thermo-gravimetric analyses were performed on not treated and 3% CaO and 5% CaO treated samples at increasing curing times using a Netzsch STA 449F3 Jupiter apparatus. Samples were first dried by freeze-drying technique. Approximately 100 mg of finely grounded material was subsequently heated at a rate of  $10^\circ\text{C min}^{-1}$ , under argon atmosphere, from ambient temperature to  $1000^\circ\text{C}$ . The Netzsch Proteus software was used to process the results.

### 3.3. Dynamic Light Scattering (DLS) measurements

The evolution of the average dimension of particles aggregates over the time has been investigated by means of dynamic light scattering (DLS) measurements. The measurements were performed on bentonite suspension (100mg/l) in deionised water and in presence of  $\text{Ca}(\text{OH})_2$  at  $T=25^\circ\text{C}$  using a Malvern Nano Zetasizer apparatus with a scattering angle of  $173^\circ$ .

### 3.4. Oedometer tests

Oedometer tests were performed on not treated and lime stabilised samples cured for increasing time intervals. Hand remoulded samples were prepared by mixing the soil with distilled water at very high water content (slurry) and then placed in the oedometer mould without compaction. The treated samples were sealed in plastic bags and cured at increasing curing times of 0, 7, 28, 60, 120 days. The tests were performed on saturated samples in standard oedometer cells, where vertical stress was conventionally applied by successive steps ( $\Delta\sigma_v/\sigma_v=1$ ).

## 4. Chemo-physical evolution

The chemo-physical evolution of clay-lime water system have been investigated by means of X-ray diffractions and thermogravimetric analyses. The X-ray diffraction patterns of not treated and 3% CaO treated samples as a function of curing time are shown in Figure 1. Lime treated samples showed a total consumption of the added lime since the very short term (0 days) as evidenced by the absence of portlandite signals in the diffraction patterns. A broad reflection corresponding to poorly crystalline compounds of C-S-H was detected in all the treated samples starting from 0 days of curing time. These results were also confirmed by the results of the thermal analyses. In Figure 2 the mass loss of the sample as a function of the curing time in the range of  $390^\circ - 460^\circ\text{C}$  (characteristic of portlandite) and  $110^\circ\text{C}-400^\circ\text{C}$  (dehydration of Ca-hydrates) has been represented. No losses in mass were detected in a range of temperature typical of the dehydration of portlandite for both 3%CaO and 5%CaO treated samples, whereas a continuous increase in mass loss was detected between  $110-400^\circ\text{C}$  attributed to the dehydration of Ca-hydrates formed as a result of the development of pozzolanic reactions. The evolution of particles aggregation process as a function of time has been shown in Figure 3. Bentonite suspension in deionised water ( $\text{pH}=7.55$ ) exhibited a dispersed fabric. The average floc size remained unchanged over time, evidencing a dispersed state of montmorillonite particles as a consequence of the repulsion between negative charged particles. A change in the average size of the aggregates was observed after the addition of lime. The presence of calcium ions and higher pH ( $\text{pH}=12.4$ ) increased the overall size of the particles aggregates as a result of the cation exchange and flocculation reactions.

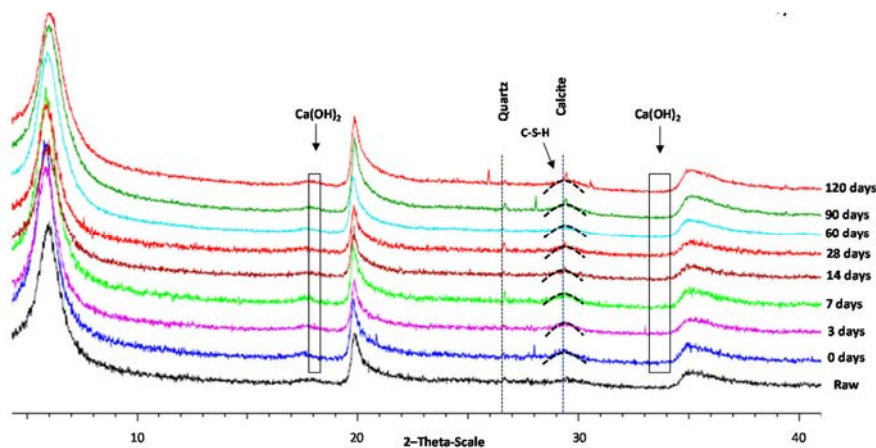


Fig. 1. X-ray diffraction pattern of not treated and 3%CaO treated bentonite at increasing curing times.

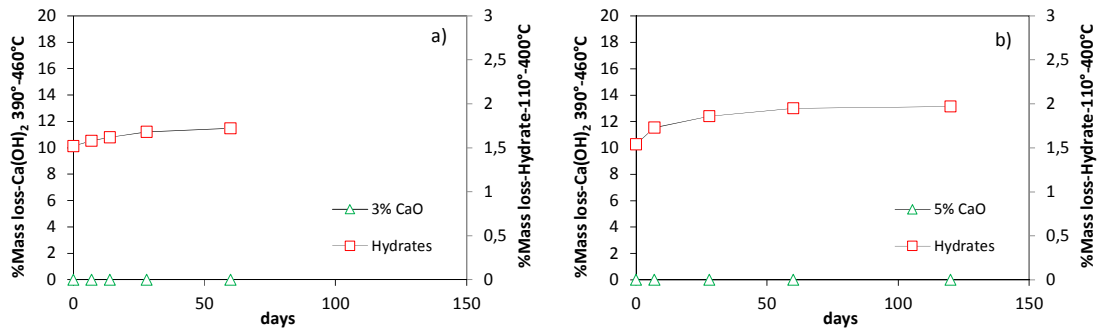


Fig. 2. Mineralogical evolution of lime treated bentonite: Portlandite consumption and precipitation of hydrates as a function of time – a) 3%CaO; b) 5% CaO.

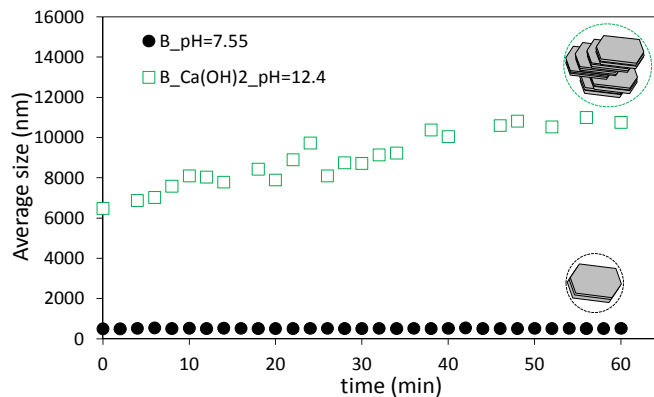


Fig. 3. Evolution of the average floc size of not treated and lime treated bentonite suspensions.

## 5. Macroscopic behaviour

The link between the ongoing reactions induced by lime and the macroscopic behaviour of the treated samples was investigated by means of 1D compression tests. In Figure 4a the compressibility curves of not treated and 3% CaO treated samples at increasing curing times (namely 0, 7, 120 days) have been represented.

The effects of lime addition resulted in a reduction of the volume strain and an increase of the yield stress, although slight changes were detected with increasing curing times. 3% CaO treated samples showed a slight dependence on curing time, as highlighted by the overlapping of compressibility curves for treated samples cured 0, 7, 120 days. A similar evolution of the mechanical behaviour, but quantitatively more relevant, and a more evident curing time dependent behaviour was observed for samples treated with 5%CaO (Fig. 4b). The observed behaviours, for both the 3%CaO and 5%CaO treated samples is consistent with the typical behaviour of a structured soil, and can be explained with the chemo-physical evolution of the system promoting the formation of larger aggregates together with the precipitation of bonding compounds. As confirmed by thermal gravimetric analyses (Fig. 2), the initial amount of hydrates was not increased significantly over curing time for 3% CaO bentonite samples (Fig. 2a), whereas the addition of 5% CaO induced an increase of the hydrated phases with curing time (Fig. 2b). The higher amounts of the hydrated phases, and its formation over time, is consistent with the increase of yield stress and reduction of

compressibility as a function of the curing time (Fig. 4b), highlighting the role of pozzolanic compounds in promoting the typical response of structured soils.

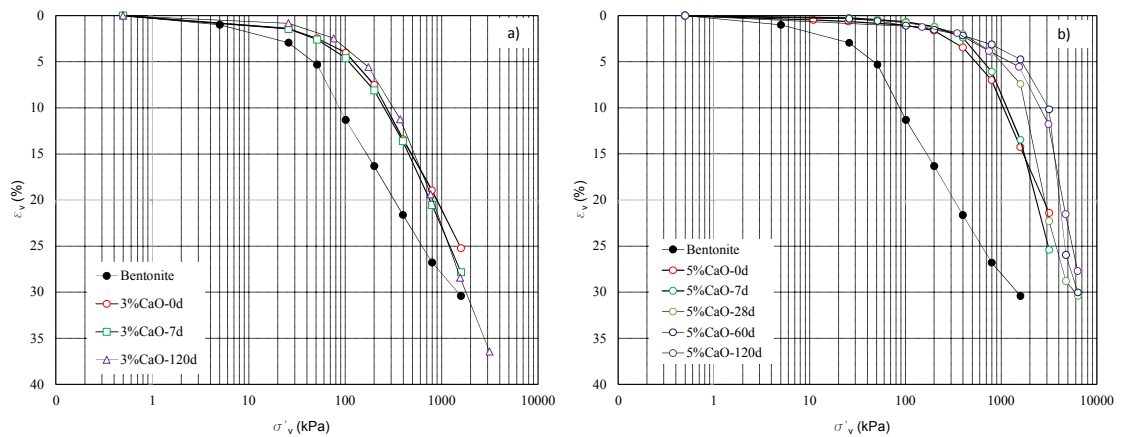


Fig. 4. Compressibility curves of not treated and lime treated bentonite at increasing curing time: a) 3%CaO; b) 5%CaO.

#### 4. Conclusions

The chemo-physical evolution induced by the addition of lime strongly affects the mechanical behaviour of the treated bentonite. Test results highlight the high reactivity of montmorillonite to promote the development of pozzolanic reaction since the very short term. As a result, the mechanical behaviour of the treated bentonite depends on the combined effect of two classes of reactions, which simultaneously affect the microstructural features of the treated soil, namely cation exchange and flocculation and pozzolanic reactions. The formation of larger aggregates together with the precipitation of bonding compounds explain the evolution of the mechanical behaviour towards the behaviour of a structured soil over curing time (i.e., increase of yield stress, reduction of compressibility and clear debonding stage for higher stress levels).

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