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## Research paper Influence of the clay type on the strength evolution of lime treated material

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#### ABSTRACT

This paper deals with the effects of lime addition on clay materials properties, in terms of mechanical and physico-chemical evolution. A coupling between these two aspects was undertaken in order to explain the mechanical behaviors of lime-treated clay materials. The study was performed on two different clays, a kaolin and a  $Ca^{2+}$ -bentonite. Lime addition leads to an increase of mechanical properties of these clays. These improvements are faster with  $Ca^{2+}$ -bentonite, even if at long term the performance reached for kaolinite is higher. The coupling between macroscopic and physico-chemical aspects shows that these improvements are linked to the development of secondary compounds such as calcium silicate hydrates, hydrogarnet and calcium carboaluminate hydrate phases.

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#### 1. Introduction

Sustainable development is marked by an increasing need of using the materials directly located within the land project. However, natural materials do not always match the technical requirements and the use of additive often meets the specifications of some constructions. The use of lime, fly ash or cement as additive to a natural soil produces a high strength material. This addition has great potential since it allows using the natural soil with initial poor mechanical characteristics and turns in favour of the natural resources saving. In earthworks, the addition of lime as a stabilizing agent appears to be a potential way since it can improve workability and mechanical properties of soils presenting low mechanical characteristics and sensitivity to water (Eades and Grim, 1960; Diamond et al., 1963; Rossi et al., 1983; Locat et al., 1990; Little, 1995; Bell, 1996; Rogers and Glendinning, 2000; Boardman et al., 2001). The use of lime represents probably one of oldest technique of stabilization since Greeks and Romans used lime-soil mixtures. The lime addition to a soil first provokes cationic exchange reactions between calcium and exchangeable cations of the clay minerals. The high amount of calcium added also induces the flocculation of the clay particles modifying the soil fabric. Then, improvements are immediate as workability, reduction of plasticity, etc. (Clare and Cruchley, 1957;

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Diamond and Kinter, 1965; Eades and Grim, 1966). The addition of a sufficient lime quantity further induces of pozzolanic reaction between the soil minerals, the calcium brought by the lime addition and the water, producing cementitious compounds with binding properties such as calcium silicate hydrates and calcium aluminate hydrates. These hydrates are also referred to by an abbreviated compositional notation as CH, CAH, CSH and CASH phases, respectively, where C = CaO,  $A = Al_2O_3$ ,  $S = SiO_2$  and  $H = H_2O$ . The occurrence of these compounds depends on the temperature, on the amount of lime added and on mineralogy of the soils and takes place over a long period of time.

The addition of few percent of lime  $(CaO, Ca(OH)_2)$  to a soil can raise the rate of strength development and the final resistance because of the formation of secondary cementitious products (Pomakhina et al., 2012; Dewindt et al., 2014; Deneele et al., 2016). Face to the need to reduce the use of raw material and the consumption of natural resources, an optimized use of the lime in the soil stabilization has to be studied. The promotion of this technique goes through: 1) a better comprehension of the lime reactivity towards different soil minerals, 2) a better knowledge of the influence of the clay mineralogy of the soil, and 3) the manner the strength development occurred in relation with the type of secondary compound. Hence, for example, Hilt and Davidson (1960) showed that the addition of lime to clays, whether it is a montmorillonite, a kaolinite or an illite/chlorite mixed, increases the performances with curing time, at room temperature. In similar conditions, the results obtained by Bell (1996) match with the ones of Hilt and Davidson (1960) in the case of the treatment of kaolinite, but are contradictories with a montmorillonite, for which the performances do





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not evolve significantly. Regarding the secondary phases formed, in lime treated kaolinite, Goldberg and Klein (1952) reported the formation of  $C_3AH_6$ , Glenn and Handy (1963) identified CSH and CAH, Bell (1996) recognized also tobermorite (CSH) and  $C_4AH_{13}$ , CAH<sub>11</sub>, CAH<sub>10</sub>. In lime treated montmorillonite, Hilt and Davidson (1960) identified  $C_4AH_{13}$  and Bell (1996) reported the formation of CAH and CSH. Rajasekaran and Rao (1999) noted the formation of CAH, CSH and CASH in marine clays treated with lime. Al-Mukhtar et al. (2010a) studied the pozzolanic reaction of Ca<sup>2+</sup>-bentonite mainly composed of kaolinite (38%) and smectite (48%). They showed a slow increase of the soil strength treated with 6% of lime with time and the formation of CAH.

In this study X-ray diffraction (XRD), mechanical strength testing and thermogravimetric analysis were used for exploring the effects of lime on two clays. The minerals used here consist of kaolinite which is a 1:1 layer clay and a Ca<sup>2+</sup>-bentonite, essentially composed of montmorillonite, a 2:1 layer type. They were treated with 10% of lime over a period from 1 h to 98 days, at 20 °C and 50 °C and at constant water content. In this experimental investigation, the lime amount (10%) was chosen in order to highlight the pozzolanic reaction between the clay minerals and the lime. The effect of the lime amount on the development of the pozzolanic reaction is described elsewhere (Maubec, 2010).

The study, carried out at 50 °C, should allow observing long-term effects thanks to an increase of the reaction kinetic between lime and clay materials. The increase of the temperature aims to accelerate the rate of reaction between the lime and clay materials. This approach is common and was used by many authors (Al-Mukhtar et al., 2010b; Eisazadeh et al., 2012; Dewindt et al., 2014). The applicability of the Arrhenius activation law was also demonstrated in the kinetics of lime/zeolite pozzolanic reactions (Snellings et al., 2009). Temperature enhanced in a similar manner the pozzolanic reaction of fly ash and silica fume cements (Bach et al., 2012; Deschner et al., 2013).

#### 2. Materials

Two clays were chosen: a kaolin (Kaolinite Polwhite) and a Ca<sup>2+</sup>bentonite (Bentonite Ikobond) supplied by Imerys and Ibeco companies respectively. Both materials are composed of particles of size <315 µm. The main geotechnical and physico-chemical characteristics of the materials are given in Table 1.

Except for calcite which was estimated by calcimetry, the amount of the other phases was evaluated by integration of the <sup>29</sup>Si signals obtained by nuclear magnetic resonance spectroscopy (Pomakhina et al., 2012). The kaolin is essentially composed of kaolinite (~88%) and small amount of muscovite (~10%), calcite (~2%) and quartz (~1%). The mineral assemblage of the Ca<sup>2+</sup>-bentonite is composed of smectite (~83%), feldspars (~10%) cristobalite (~5%) and calcite (~2%). The lime used is industrial quicklime provided by Lhoist Company which contains 94% of free oxide calcium.

Table 1
Main geotechnical and physico-chemical properties of the materials.

	Polwhite kaolinite	Ikobond calcium bentonite
Geotechnical properties		
Plastic limit, Wp (%)	40.2	54.5
Liquid limit, W <sub>L</sub> (%)	58	158
Plasticity index, Ip (%)	17.8	103.5
Specific density (Mg $m^{-3}$ )	2.6	2.8
Physico-chemical properties		
Cation exchange capacity (meq/100 g)	5.9	55.3
Specific surface area $(m^2/g)$ (BET method, N <sub>2</sub> adsorption)	13.3	47.8

#### 3. Experimental procedures

#### 3.1. Mechanical test

The mechanical behaviour was assessed by the measurement of the unconfined compressive strength (UCS). It was performed on specimens of clay materials untreated and treated with 10% of lime prepared and compacted at 98.5% of the optimum Proctor conditions (Table 2).

The clay materials were first mixed with distilled water in order to reach the desired moisture content and were sealed in a plastic bag for homogenisation during 3 and 7 days for kaolin and Ca<sup>2+</sup>-bentonite, respectively. Then the lime, 10% in mass of the dry material, was added to the wet material, before mixing. The compaction was performed 1 h after mixing in cylinder moulds of 100 mm in height and 50 mm in diameter using a hydraulic compaction apparatus to which the load was applied to a rate of 1 mm/min. Then compacted specimens were sealed using both cellophane and aluminium paper to avoid moisture loss and carbonation and were cured at room temperature (20 °C) for different curing times (1 h to 98 days). A second set of specimens were compacted in similar conditions and cured at a temperature of 50 °C, in order to increase the reactivity between lime and clays according the Arrhenius law.

Unconfined compressive tests were carried out on compacted specimens at the end of different curing times. The tests were carried out at a constant displacement rate of  $1 \text{ mm} \cdot \text{min}^{-1}$ . After the tests, samples are freeze dried at -58 °C for 24 h and then stored in a dessicator under vacuum before the physico-chemical characterization. Physico-chemical analyses were performed on samples after mechanical testing.

#### 3.2. Physico-chemical investigation

Before characterization, the specimens were crushed then analysed by X-ray diffraction (XRD) and thermogravimetric analysis (TGA). XRD analysis of untreated and treated materials were performed on a Bruker AXS D8 Advance with Cu-K $\alpha$  radiation ( $\lambda = 1.54056$  Å), from 4° to 40° 20, with a step of 0.017° 20 and a 3 s counting time. The analyses were carried out on randomly oriented powder samples.

TGA analyses were carried out on a Setaram TG-DSC 111 apparatus from 25 °C to 830 °C with 5 °C/min. The analyses were performed under argon atmosphere in order to limit sample carbonation.

#### 4. Results and discussion

#### 4.1. UCS evolution

Table 2

The UCS evolution of compacted  $Ca^{2+}$ -bentonite sample treated with 10% of lime and cured from 1 h to 98 days at 20 °C and 50 °C is presented in Fig. 1. The results are compared to the untreated material which is characterized by an unconfined compressive strength of 300 kPa.

At 20 °C, the treated material exhibits a linear growth of the UCS, from 300 kPa to 1500 kPa. After 98 days of curing, performances are about 5 times more important than the untreated material. At 50 °C it is characterized by a significant gain in strength during 7 days followed by stabilization.

It can be noted that the unconfined compressive strengths values of specimens cured at 50 °C for 1 and 2 days are equivalent to those

Optimum Proctor conditions of samples untreated and treated with 10% of lime.
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Materials	Optimum	0% CaO	10% CaO
Kaolinite Polwhite	Water content (%)	25.50	27.50
	Density (g·cm <sup>-3</sup> )	1.48	1.40
Calcium Bentonite Ikobond	Water content (%)	40	34.80
	Density (g·cm <sup>-3</sup> )	1.22	1.15

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