



## Research paper

## Lime consumption of different clayey soils

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## ABSTRACT

This study focused on the lime consumption by 10% of lime treatment, of five soils containing different major clay minerals, kaolinite, illite, smectite–kaolinite, smectite–illite and smectite. Assessing the consumption of lime with respect to curing time, using an atomic absorption device, illustrates the lime–clay mineral reaction and the mechanisms involved in curing in the short- and long-term.

X-ray diffraction and thermogravimetric analysis results show that clayey soils have different kinetic reactions depending on to their mineralogical composition, and that all soils consume the amount of lime added after a shorter or longer period of curing time. Two main reactions were assessed: the short-term reaction (cation exchange and flocculation) and the long-term reaction (pozzolanic reaction). These reactions are initiated due to the highly alkaline medium induced by the dissolution of lime in the water contained in the soil. The amount of lime consumed during the short-term reaction varies from nothing for kaolinite and maximum with sodium-smectite. The pozzolanic reaction present in all the clayey soils tested depends on the amount of lime available and is greatly accelerated by increasing the curing temperature from 20° to 50 °C. This reaction induces structural and mineralogical changes in the treated soils due to the newly formed calcium hydrates.

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## 1. Introduction

Lime treatment has a beneficial effect on the properties of clayey soils by enhancing many of their engineering properties: a reduction in water content and swelling potential, an improvement in workability and in-situ compaction, and an increase in mechanical strength (Al-Mukhtar et al., 2012; Lasledj, 2009; Maubec, 2010; Stoltz et al., 2012). Lime treatment leads to structural and textural changes in clayey soils and irreversibly changes their hydro-mechanical behaviour.

The dissolution of lime in the water contained in the treated soil induces a strongly alkaline medium (pH reaching 12.4) and the divalent calcium cations ( $\text{Ca}^{++}$ ) of lime are dissolved. In the first moments of the lime treatment,  $\text{Ca}^{++}$  fixes to the surface of the clay mineral and the calcium ions replace most of the available exchangeable cations (Stumm, 1997). High-valent cations replace lower valent cations and the larger cations are substituted for smaller ones. Instantaneous reactions including cation exchange and flocculation–agglomeration are prompted by the addition of a relatively small amount of lime.

The reaction rate in the long-term depends on only on the crystallographic nature of the clay minerals (smectite, illite and kaolinite react differently to the lime effect) but also on the curing temperature (Little, 1996; Rao and Shivananda, 2005; Rojas and Cabrera, 2002). When triggered, these long-term reactions enable a further

improvement in the properties of the treated clayey soil. However this reaction depends on the presence of a quantity of lime greater than that used in instantaneous reactions (Eades and Grim, 1960; Hill and Davidson, 1960; Sivapullaiah et al., 2000). Calcium hydrates (Ca-hydrates) formed during the pozzolanic reaction with the silica and alumina released from the clay mineral and the lime (calcium and hydroxyl ions), are similar to those of hydrated cement (portland): calcium silicate hydrate (CSH) and/or calcium silicate aluminate hydrate (CSAH) and/or calcium aluminate hydrate (CAH) (Al-Mukhtar et al., 2010a; Al-Rawas and Goosen, 2006; Arabi and Wild, 1986; Bell, 1996; Boardman et al., 2001; Diamond and Kinter, 1965; Khattab, 2002). This crystallisation, although slow at the beginning, will continue as long as residual calcium is available and the pH is high enough. The kinetics of the pozzolanic reaction depend on several factors, including the physical and chemical characteristics of the treated clay mineral, curing temperature, moisture and freeze–thaw cycles (Al-Mukhtar et al., 2010b; Khattab et al., 2007; Liu et al., 2010; Rao and Shivananda, 2005).

The aim of this work was to study the mechanisms involved in the clay mineral–lime reaction in both the short-term and long-term by monitoring the progress of lime consumption during six months, using atomic absorption, for two curing temperatures of 20 °C and 50 °C. Five different clayey soils with different mineralogical compositions of  $\text{Na}^+$ -smectite,  $\text{Ca}^{++}$ - $\text{Mg}^{++}$  smectite, illite and kaolinite were studied. A high amount of lime addition (10%) was used in this study to ensure a complete reaction with the soils and to easily determine the amount of lime consumed by the clayey soils tested. Changes in

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the mineralogy of the five clayey soils due to lime treatment were studied by X-ray diffraction (XRD) and thermogravimetric analysis (TGA). The mineralogical changes and the assessment of lime consumption lead to a better understanding of the main factors that control the progress (kinetics) of lime–clay reactions.

## 2. Materials and methods

### 2.1. Materials

The main clay minerals of the five clayey soils tested are kaolinite, illite, smectite/kaolinite, smectite/illite and smectite. These soils have different pH, electrical conductivities (EC) and cation exchangeable capacities (CEC), and are denoted as Kaol, I, Sm–Kaol, Sm–I and Sm respectively. The chemical and the mineralogical compositions of these soils are given in Tables 1 and 2.

Kaol soil contains 83% kaolinite and the rest is composed of 10% illite and 7% quartz. The major component of I soil is illite (55%). The Sm–Kaol soil is a mix of 48% dioctahedral Na<sup>+</sup>-smectite (with sodium as the main exchangeable cation) and 38% kaolinite. An equivalent percentage of smectite and illite compose the Sm–I soil, with two exchangeable cations: Ca<sup>++</sup> and Mg<sup>++</sup>. In the Sm soil, the percentage of smectite is about 72% with exchange cations Ca<sup>++</sup> and Mg<sup>++</sup>. These soils with different mineralogical compositions and properties (CEC and swelling) were chosen to represent different types of clay minerals: interlayer space with exchangeable cations (smectite), and interlayer space without exchangeable cations (kaolinite, illite); layers with a TO structure, i.e. one tetrahedral (T) sheet and one octahedral (O) sheet, that is repeated (kaolinite) and layers with a TOT structure, i.e. one octahedral (O) sheet between two tetrahedral (T) sheets that is repeated (smectite, illite) (Bergaya and Lagaly, 2006).

A commercial hydrated lime (Calcia) was used for treatment of the studied soils. This commercial lime contains 94% lime (portlandite) – Ca(OH)<sub>2</sub> – and 6% calcite.

### 2.2. Methods

#### 2.2.1. Determination of lime consumption by chemical analysis

The determination of lime which has not reacted with the clay mineral was carried out by atomic absorption spectrometry (AAS) using a Hitachi Z-8100 Polarized Zeeman device. The concentration of calcium was measured in the solutions extracted from mixtures of lime-treated soil, after curing times of 1.5 h, 7 days, 28 days, and 3 months and 6 months and for two curing temperatures of 20 and 50 °C. The lime consumption was deduced from these measurements.

Samples of treated soil were prepared by mixing 0.5 g soil with 2, 4, 6 and 10% lime. The lime–soil mixture was dispersed in a bottle

**Table 1**

Chemical composition, pH, electrical conductivity (EC) and cation exchange capacity (CEC) of the clayey soils tested.

Clayey soils element	Kaol soil	I soil	Sm–Kaol soil	Sm–I soil	Sm soil
SiO <sub>2</sub>	48.0	49.7	46.0	55.0	55.8
Al <sub>2</sub> O <sub>3</sub>	36.5	15.8	23.3	15.5	18.0
Fe <sub>2</sub> O <sub>3</sub>	0.9	6.0	7.6	5.4	4.4
MnO	Trace	0.1	0.1	0.0	0.0
MgO	0.2	2.5	1.4	2.1	5.9
CaO	0.2	8.9	4.4	8.9	3.4
Na <sub>2</sub> O	0.1	0.1	2.5	0.2	0.1
K <sub>2</sub> O	1.4	3.9	0.2	2.9	1.2
TiO <sub>2</sub>	0.2	0.8	1.0	0.8	0.4
P <sub>2</sub> O <sub>5</sub>	0.0	0.1	0.0	0.0	0.0
Loss on ignition	12.5	12.0	12.3	9.2	10.4
pH	6	7.4	10.6	7.9	8.1
EC, mS/cm	-	1.9	2.7	2.3	2.3
CEC, meq/100 g of soil (Bergaya et al., 2006)	3–15	10–40		70–120	

**Table 2**

Mineralogical composition (mass%) of the clayey soils tested.

Clayey soils/ components	Kaol soil	I soil	Sm–Kaol soil	Sm–I soil	Sm soil
Smectite	-	-	48.0	38.1	72.0
Illite	10.0	55.0	-	40.0	15.0
Kaolinite	83.0	22.0	38.0	-	10.0
Quartz	7.0	9.0	3.2	10.6	0.8
Calcite	0.0	13.0	3.4	10.3	2.1
Goethite	0.0	0.0	6.4	0.0	0.0
Anatase	0.0	0.1	1.0	0.9	0.1
Exchange cation	-	-	Na <sup>+</sup>	Ca <sup>++</sup> , Mg <sup>++</sup>	Ca <sup>++</sup> , Mg <sup>++</sup>
Main mineral	Kaolinite	Illite	Smectite kaolinite	Smectite illite	Smectite

containing 50 mL (50 g) of demineralised water then the bottle was closed. The mixture was protected from the atmosphere to avoid carbonation of the lime during curing. The mixture was periodically stirred to ensure uniformity until the end of the curing time, at which time the test solution was extracted, filtered to 0.45 µm and analysed.

#### 2.2.2. Determination of mineralogical changes by XRD and TGA

The mineralogical changes were studied by XRD and TGA performed on the samples treated and compacted after a long curing period (6 months) at 20 and 50 °C. At the end of the curing time and before testing the samples were dried for 4 h in an oven at 50 °C, ground, and then sieved at 100 µm.

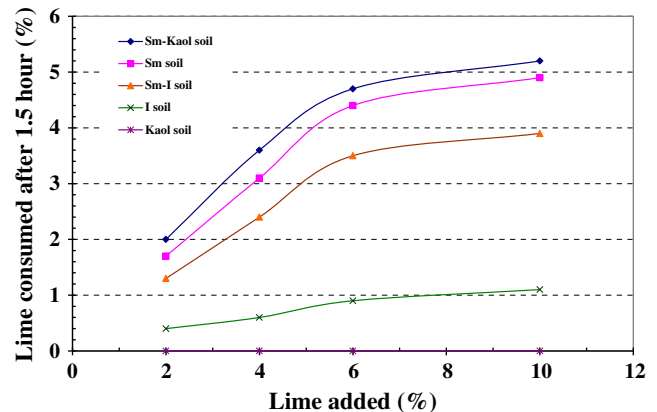
XRD was performed on the powder, using CuKα<sub>1</sub> radiation, obtained with a monochromator. The diagrams were recorded on a Philips PW3020 diffractometer (scanning from 2° to 70° with a step width of 0.1° and time step of about 100 s).

TGA was performed using a Setaram TGA 92 apparatus. Samples were first dried in a desiccator with P<sub>2</sub>O<sub>5</sub> to stabilise the mass of the sample. The heating rate was 1.7 °C/min under an argon flow. The temperature varied from 20 °C to a final temperature of 900 °C, with a plateau (fixed temperature) at 110 °C for 2 h. The stand-by at 110 °C led to a constant mass in time at this temperature, which served as the reference mass.

## 3. Results and analyses

### 3.1. Lime consumption

It must be mentioned here that the maximum amount of lime that can be consumed is 9.4%, since the lime added contained 6% calcite and 94% lime.



**Fig. 1.** Immediate lime consumed (1.5 h) by different clayey soils tested.

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