



Research paper

Short-term lime solution-kaolinite interfacial chemistry and its effect on long-term pozzolanic activity

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ABSTRACT

Soils stabilization with lime addition is a widespread technique in geotechnical field. Better understanding of interaction mechanism is central for optimization of the technique. In the present work, the short-term kaolinite-lime solution interfacial chemistry and the effect on the dissolution and long-term reaction has been investigated. Calcium adsorption from saturated lime solution (i.e. simulated alkaline pore solution ([Ca] = 22 mmol/l and pH = 12.63)) characterized by initial fast uptake followed by slower adsorption rate curves, whereas only initial fast uptake is identified from chloride solution ([Ca] = 22 mmol/l and pH = 7). Species of calcium available for adsorption are strongly dependent on the pH conditions (i.e. Ca²⁺ at pH 7 and CaOH⁺ at pH 12.63). The adsorption increased with increasing initial concentration of calcium. On the other hand, the more the quantity of calcium adsorbed, the less the measured concentration of silicon and aluminum in the supernatant. The adsorbed calcium distribution has been mapped using μ -XRF. Regardless of the quantity of calcium adsorbed, homogeneous distribution has been identified suggesting coating of kaolinite particle by adsorbed calcium. The coating in turn prevented the kaolinite particles from alkaline attack and retards long-term pozzolanic reaction.

1. Introduction

For both environmental and economic reasons and in a context of circular economy, it is necessary to use local material for construction works. However, the soft geo-materials that commonly encounter in construction sites often do not meet the geotechnical requirement and are the main challenges to geotechnical engineers. Particularly, clay mineral rich soils possess low strength, high compressibility, poor workability and exhibits volume change in response to seasonal moisture variation. These properties commonly pose damage on light-weight structures, such as pavements, residential buildings, etc. A chemical treatment, such as lime addition, is widely recognized alternative to stabilize and reuse problematic soils (Diamond and Kinter, 1965; Verhasselt, 1990; Bell, 1996).

Extensive research on soil stabilization with lime has been done by various researchers. The focus of most the studies has been either to understand the effect of lime addition on properties of soil from macroscopic point of view (gain in strength, modification of the plasticity and swelling behavior, etc) (Choquette et al., 1987; Al-Rawas et al., 2005; Al-Mukhtar et al., 2010; Eisazadeh et al., 2012; Pomakhina et al.,

2012; Deneele et al., 2016; Maubec et al., 2017; Guidobaldi et al., 2017; Vitale et al., 2017) or the long-term pozzolanic reactions that arise between the clay mineral and alkaline lime solution (He et al., 1995; Lemaire et al., 2013). The reaction is generally divided in to modification and stabilization. Modification is related to exchange of surface cations by calcium, while stabilization is related to precipitation of hydrated gels (i.e. calcium silicate hydrate (CSH), calcium aluminates hydrate (CAH) and calcium aluminates silicate hydrates (CASH)) and is responsible for solidification and an increase in mechanical properties of lime treated soils in long-term.

In contrast, physico-chemical mechanisms of the stabilization reaction, particularly in the short term, were not sufficiently explored yet. Moreover, they are usually ignored in literature. The effect in the early stage is usually explained by flocculation of clay mineral particles due to compression of diffused double layer and linkage in the presence of calcium (Chemed et al., 2015; Vitale et al., 2016). However, no detailed information is available on mechanism and species of calcium involved in the exchange process. The reaction at this stage (i.e. calcium adsorption, surface charge modification and particle interaction) is crucial in soil stabilization as it can affects the mixing ability of the

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soil with lime, which could in turn influences the physico-mechanical behavior of the treated soil in long term. The better the mixing, the better the dispersion and the diffusion of lime and consequently, the effective stabilization process (He et al., 1995). Therefore, it seems necessary to understand in detail the interfacial phenomenon at lime solution clay minerals interface for predicting the reaction and optimization of the treatment technique.

In fact, it is the dissolved lime solution rather than lime grains that interact with soil constituent minerals. Thus, working with saturated lime solution ($[Ca] = 22 \text{ mmol/l}$, $\text{pH} = 12.63$) is more ideal to simulate the practical condition (i.e. calcium rich alkaline pore solution). The present paper, thus, tries to explore in more detail the interface reaction between saturated lime solution and kaolinite (Kaol) (i.e. the mechanism and chemical nature of calcium adsorbed). In further study, the effect of the amount of calcium adsorption on dissolution reaction will be addressed. Elemental mapping with $\mu\text{-XRF}$ after treatment with different concentration of lime allows to show the relative distribution of adsorbed calcium.

2. Materials and methods

Five Kaol samples of different origins and properties were used. Kaol KGa-1b (low-defect) and KGa-2 (high-defect) were purchased from the Source Clay Repository of the Clay Minerals Society. Kaol Pwt (low-defect), SPwt (low-defect) and B24 (high-defect) were obtained from Brittany, France, Cornwall, UK and Charente basin, France, respectively. Their physical properties are summarized in Table 1. Hydrated lime, $\text{Ca}(\text{OH})_2$ (Merck, analytical grade, purity above 97%) was used for preparation of saturated lime solution.

Prior to experiment, Kaol samples were purified and exchanged with sodium at pH 7. Briefly, in order to remove carbonates, 30 g of raw samples were dispersed in 100 ml sodium acetate–acetic acid buffer of $\text{pH} = 4.8$ and stirred overnight while heating at 80°C . The process was repeated until there was no more evolution of carbon dioxide bubbles. In order to remove organic matter, 30 ml of 30% (w/w) hydrogen peroxide solution were added to the slurry after decarbonation and the mixture was stirred for 4 h at 50°C . The slurry was then centrifuged and washed five times with ultrapure water. Clay particle size fractionation was done by dispersing about 10 g of the purified sample in 1 L of ultrapure water and centrifugation method. To obtain the mono-cationic Na-Kaol, the dispersion of clay size fraction ($< 2 \mu\text{m}$) was treated with 1 M NaCl solution. After centrifugation at 8000 rpm, the supernatant solution was discarded and replaced with fresh solution (the procedure was repeated three times), and was followed by several washing and centrifugation cycle until electrical conductivity stayed below $5 \mu\text{S}$.

Table 1

Physical properties, mineralogical and chemical composition (%) of the studied Kaol. Other mineral includes quartz, anatase and goethite. Values in brackets correspond to quantification error for each mineral, estimated by TOPAS 3.0® software. SSA corresponds to the specific surface area measured by BET method and HI to the Hinkley Index (crystallinity Index).

	KGa-1b	KGa-2	Pwt	SPwt	B24
Kaolinite (0.4–1.3)	96	97	94	95	95
Muscovite (0.6–0.8)	–	–	5	4	3
Other (0.1–1.1)	4	3	1	1	2
SiO_2	44.57	45.41	51.33	49.19	49.01
Al_2O_3	39.45	38.08	34.91	34.53	34.86
Fe_2O_3	0.39	2.08	1.63	1.23	1.76
K_2O	0.02	0.06	2.3	1.67	1.09
TiO_2	1.14	1.56	0.06	0.02	0.79
Na_2O	0.03	0.02	0.06	0.08	0.06
CaO	0.01	0.01	0.03	0.02	0.04
MgO	0.01	0.02	0.29	0.15	0.13
SSA (m^2/g)	10.8	21	17.2	19	27.7
HI	1.1	0.38	1.28	1.24	0.28

Bulk mineralogy of the samples was determined from X-ray powder diffraction (XRD), on randomly oriented samples, with a Bruker AXS D8 device, using $\text{CuK}\alpha$ ($\lambda = 0.154 \text{ nm}$) radiation and a step size of 0.021° . The mineral phases were quantified using a Rietveld-based refinement routine using the TOPAS 3.0® software. Bulk chemical analysis was performed by inductively coupled plasma-optical emission spectroscopy (ICP-OES) after sample digestion. The mineralogical and chemical composition of the samples are summarized in Table 1. Kaol is the dominant clay mineral phase in all samples. Pwt, SPwt and B24 also contain $< 5\%$ muscovite. Minor phases present in the samples are quartz, anatase and traces of goethite. Structural order was estimated using the Hinkley index (HI) and given in Table 1.

The adsorption of the calcium ion onto the Kaol samples was studied using batch equilibrium tests with Kaol sample/solution ratio of 1:5. The clay size fraction was put in contact with a freshly prepared saturated lime solution (initial calcium concentration $[\text{Ca}]_0 = 22 \text{ mmol/l}$ and initial pH of 12.63) in centrifuge tubes and stirred periodically for the desired contact time. All preparations (weighting, batch preparation, and sampling) were performed in a nitrogen glove box under argon atmosphere. After each equilibration time, the tubes were ultra-centrifuged (at 8000 rpm for 20 min) and the supernatant was analyzed for concentration of Ca, Na, Al, Si... by using ICP-OES. The pH of the supernatant before and after the adsorption experiment recorded from which the OH-ions amount determined. The amount of calcium and hydroxide ions adsorbed and sodium ion desorbed was determined in mole ions per gram of Kaol sample using mass balance equation as follow:

$$q = \frac{V(C_i - C_f)}{M}$$

where C_i represents initial concentrations of ions in the initial solution, C_f is the concentrations of ions in supernatant after each contact time, V is the initial volume of lime solution (30 ml) and M is the mass of Kaol (6 g).

Calcium ion desorption measurement was carried out on 24 h treated sample to test the exchangeability of adsorbed calcium. First the treated sample was washed once with ultrapure water and dispersed in 30 ml 1 M KCl solution and shaken mechanically for 24 h. The supernatant was separated with a high speed centrifugation (at 8000 rpm for 20 min) and analyzed for calcium concentration by using ICP-OES.

In order to assess the effect of initial calcium concentration on the amount of calcium adsorbed and Kaol dissolution, the concentration of calcium and hydroxide remained in supernatant after a contact of 3 h was measured with increasing the concentration of $\text{Ca}(\text{OH})_2$. $\mu\text{-XRF}$ was used to quantify the chemical nature of the extreme surface of Kaol and the distribution of adsorbed calcium via elemental mapping. The M4 Tornado Bruker system using small-spot Micro-XRay fluorescence ($30 \mu\text{m}$) equipped with Rhodium RX source and an EDS system (i.e. an SDD detector) was used. The analysis were carried out on pressed powder pellet. ^{29}Si NMR and TGA were used to identify secondary phases if formed in the studied time scale. The ^{29}Si NMR spectra were acquired with Bruker Avance III (300 MHz frequency) spectrometer by direct excitation of ^{29}Si nuclei using the magic-angle spinning (MAS) technique of 5 kHz frequency. The spectra were acquired with a recycle delay of 180 s. For all spectra, ^1H decoupling was performed during acquisition. To avoid broadening of the MAS NMR spectrum, Fe-free Kaol (KGa-1b) was selected for this experiment.

3. Results

3.1. Calcium adsorption studies

3.1.1. Adsorption kinetics

The time dependence of calcium adsorption by different Kaol from saturated lime solution is showed in Fig. 1. The result showed that the calcium uptake obeyed multiple first order kinetics as indicated by

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