

Research paper

Smectite formation upon lime stabilization of expansive marls

Kerstin Elert^{a,*}, José Miguel Azañón^{b,c}, Fernando Nieto^{a,c}^a Department of Mineralogy and Petrology, University of Granada, Fuentenueva S/N, Granada 18002, Spain^b Department of Geodynamics, University of Granada, Fuentenueva S/N, Granada 18002, Spain^c IACT, CSIC-University of Granada, Spain

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ABSTRACT

Marl was treated with Mg-rich lime to evaluate the effectiveness of lime stabilization for this expansive, clay- and carbonate-rich material. XRD and TEM analyses revealed very limited dissolution of expandable clay minerals and neoformation of Mg-enriched smectites. C-(A)-S-H phases formed and initially cemented clay particles. However, they experienced decalcification and decomposition over time, leading to disaggregation of clay particles and deterioration of some of the marl's geotechnical properties. Results question the effectiveness of lime stabilization for marls, especially using Mg-rich lime. Modifications to commonly used treatment protocols in order to overcome these shortcomings are discussed.

1. Introduction

Lime stabilization has been used to improve the plastic properties of subgrades for almost a century (Bell, 1996). Generally, lime stabilization is performed to reduce the soil's plasticity index and swelling capacity, both being related to the presence of large amounts of smectites in highly plastic soils. In the case of clayey soils, lime treatments often proved successful (Al-Mukhtar et al., 2010; Di Sante et al., 2014). However, doubts have been recently raised regarding the effectiveness of lime stabilization for marls, containing important amounts of carbonates (Sol-Sánchez et al., 2016). In this case, common lime treatment protocols may result in premature stabilization failure. To overcome the shortcomings of lime treatments for marls, alternative stabilization agents (i.e., fly- and biomass-ash, and Ca-rich industrial or mine waste) have been tested (Ureña et al., 2015).

The addition of lime to soils provokes several reactions which include: a) reduction in the soils' moisture content due to the hydration of lime (I. $\text{CaO} + \text{H}_2\text{O} \rightarrow \text{Ca}(\text{OH})_2$); b) reduction of intracrystalline swelling due to cation-exchange in soils containing Na-smectites; c) reduction of osmotic swelling as a result of lower clay particle repulsion in the presence of an electrolyte (flocculation); d) precipitation of calcium carbonate and/or binding of soil particles upon carbonation of portlandite (II. $\text{Ca}(\text{OH})_2 (\text{s}) + \text{CO}_2 (\text{g}) + \text{H}_2\text{O} (\text{aq}) \rightarrow \text{CaCO}_3 (\text{s}) + 2\text{H}_2\text{O} (\text{aq}) + \text{heat} \uparrow (74 \text{ kJ/mol})$ (Moorehead, 1986)); e) dissolution of clay minerals and other mineral phases (i.e., feldspars and quartz) and the formation of pozzolanic materials (i.e., C-(A)-S-H, calcium (aluminum) silicate hydrates) with high cementing capacity and mechanical strength (Bell, 1996).

Mineral dissolution and pozzolanic phase formation are of special importance in order to achieve particle aggregation and improve the soil's geotechnical characteristics. Upon dissolution, aluminosilicate minerals (i.e., clays) provide Al and Si species for the formation of pozzolanic phases (Gaucher and Blanc, 2006). Clay minerals show different dissolution rates in alkaline environments, depending on layer charge, structure, and chemical composition. Generally, illite has been found to dissolve more slowly than kaolinite and smectite (Carroll and Starkey, 1971; Jozefaciuk and Bowanko, 2002). From a geotechnical point of view a reduction of the smectite content due to dissolution would be highly desirable. However, in our previous study only a very minor decrease in the smectite content (i.e., smectite being the dominant clay mineral of the tested marl) was detected and permanent particle aggregation was not achieved in marl treated with calcitic or Mg-rich lime (Elert et al., 2017). Consequently, initially improved geotechnical properties started to deteriorate over time (Ureña et al., 2015).

In order to determine possible mineralogical changes other than the limited dissolution of clay minerals and formation of C-(A)-S-H, which could explain the observed stabilization failure, exhaustive additional analyses were performed. In the current study we focused on the effects of Mg-rich lime for marl stabilization. Mg-rich lime is widely used for soil stabilization and is the most common lime exploited in the USA (Boynton, 1984). It is also very abundant in the area of Granada (southern Spain), where problems associated with expansive marl have been reported (Azañón et al., 2010). Furthermore, marl treatment with Mg-rich lime is more likely to produce detectable compositional changes in smectites upon neoformation, because Mg^{2+} is expected to

* Corresponding author.

E-mail address: kelert@ugr.es (K. Elert).

partially replace Al^{3+} in the octahedral layer of smectites, whereas Ca^{2+} from calcitic lime would only enter as an interlayer cation.

The results obtained here point to a transformation, rather than a destruction of smectite clay minerals. This unexpected finding has important implications in the field of civil engineering and might lay the basis for the modification of current treatment protocols, especially in the case of post-stabilization failure.

2. Materials and methods

2.1. Materials

The marl sample was extracted from clay-rich levels at 32 m depth from the base of the Diezma landslide (southern Spain), responsible for important structural damage to the A-92 highway back in 2001 (Azañón et al., 2010). This material belongs to a Flysch-type formation outcropping continuously in the western and central parts of the Betic Cordillera (Bourgeois et al., 1974). According to X-ray diffraction (XRD), the untreated marl contained clay minerals (predominantly smectite and small amounts of kaolinite and mica), quartz, calcite and minor amounts of feldspar and dolomite. Elemental analysis (EA) revealed a carbonate content of 24.4%, and thermogravimetric (TG) analysis of ethylene glycol (EG) solvated samples (Nieto et al., 2008) gave an average smectite content of 30.5 ± 2.0 wt% in the untreated marl (Elert et al., 2017).

The Mg-rich lime was obtained from residual sludge from the local extraction of magnesium-rich limestone and marble. This sludge was calcined and hydrated prior to its use and contained 54.21 wt% CaO and 25.26 wt% MgO as well as trace amounts of SiO_2 , Al_2O_3 , and Fe_2O_3 according to X-ray fluorescence analysis (Elert et al., 2017). Considering a loss on ignition of 18.60 wt%, it was concluded that the hydrated lime only contained an insignificant amount of carbonates and was, therefore, expected to be highly reactive.

2.2. Sample preparation

Hundred fifty grams of dry marl sample was mixed with 15 wt% of dry Mg-rich lime and thoroughly mixed for 10 min. A sufficient amount of water was added until the plastic limit was reached. The sample was kept in the laboratory exposed to air at room T . Water was added when needed in order to keep the sample completely covered and to limit carbonation. Aliquots were taken at 7, 14, 21, 49, 77, 105, and 201 days. The treated marl was stirred prior to the collection of aliquots in order to obtain a homogeneous sample.

2.3. Analytical methods

pH measurements were performed periodically during the lime treatment using a pH-meter Stick Piccolo HI 1280 with ± 0.1 pH accuracy (Hanna Instruments).

A laser particle size analyzer (Mastersizer 2000LF, Malvern Instruments) was used to determine the particle size distribution of untreated and treated marl samples dispersed in alcohol.

Surface area measurements (BET method (Brunauer et al., 1938)) were performed on untreated and treated soil samples using a TriStar 3000 analyzer (Micrometrics). Prior to analysis, samples were degassed at 80°C for 24 h using a sample degas system (VacPrep 061, Micrometrics). Degassing was performed at such a relatively low T in order to avoid changes in the smectite structure.

Mineralogical changes upon lime treatment were studied with a PANanalytical X'Pert Pro X-ray diffractometer using disoriented powder samples. Equipment settings: $\text{CuK}\alpha$ radiation, 45 kV, 40 mA, $5\text{--}70^\circ$ 2θ exploration range, 0.008° 2θ step size, and 10 s/step counting time. Xpoder software (Martín-Ramos, 2004) was used to identify mineral phases.

The smectite content was determined by TG analysis according to

the methodology developed by Nieto et al. (2008), consisting in weight loss (WL) measurements of EG solvated and Mg saturated samples in a T range between 100 and 450°C . The smectite content was calculated according to the following equation: $\text{Sme}\% = 3.96 \text{ WL} - 4.05$.

Field emission scanning electron microscopy (FESEM, Auriga (Carl Zeiss)) was used to determine morphological and microstructural changes upon lime treatment. Carbon coated samples were analyzed at 3 kV beam accelerating voltage in secondary electron imaging mode.

Transmission electron microscopy (TEM) was performed on untreated and treated marl samples using two microscopes. A Philips CM20 was used for quantitative elemental analysis (TEM-AEM) in scanning transmission electron microscopy (STEM) mode coupled to an EDAX solid-state energy dispersive X-ray (EDX) detector. Equipment settings: 200 kV accelerating voltage and 20×100 nm scan window. Images and compositional maps were obtained using a Titan (FEI) with XFEI emission gun, spherical aberration corrector and High Angle Annular Dark Field (HAADF) detector working at 300 kV. Powdered samples were dispersed in ethanol, sonicated, and deposited on C-coated Cu grids. Mineral standards were used to obtain k-factors according to the method by Cliff and Lorimer (1975).

3. Results and discussion

3.1. Physical properties and pH evolution

Particle size distribution analyses revealed that the average particle size of untreated marl was $10 \mu\text{m}$. It increased by 70% immediately after 15 wt% Mg-rich lime was added (Fig. 1). Concomitantly, the surface area of the marl sample decreased from $39 \text{ m}^2/\text{g}$ to $14 \text{ m}^2/\text{g}$. These changes can be attributed to flocculation, which is a pH-independent process. Actually, no drop in pH was observed at this point. The presence of an electrolyte, which includes most salts, bases, and acids, facilitates the flocculation of colloidal clay particles. Flocculation is achieved by reducing the range of electrical double layer repulsion between particles (van Olphen, 1987). These particles aggregate into larger clusters and, consequently, experience a surface area decrease (Mowafy et al., 1985), as was observed in the marl sample treated here.

After 1 week of treatment, the particle size and surface area increased by 124 and 146%, respectively. Both effects can be correlated with the formation of C-(A)-S-H phases (Elert et al., 2017). FESEM images (Fig. 2) confirmed the aggregation of clay particles and the formation of new phases, possibly C-(A)-S-H, in samples treated for 1 week. C-(A)-S-H phases have high cementing capacity and result in the formation of large, relatively stable aggregates of clay particles, leading to the observed particle size increase (Fig. 1). They generally

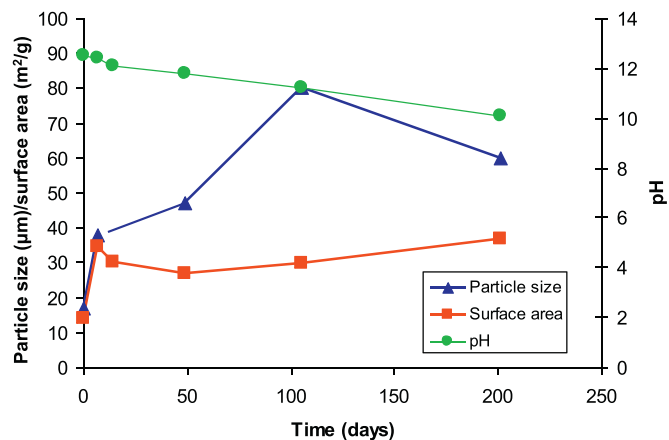


Fig. 1. pH evolution, particle size and surface area of marl treated with 15 wt% Mg-rich lime for 201 days. The surface area and particle size of the untreated marl were $39 \text{ m}^2/\text{g}$ and $10 \mu\text{m}$, respectively.

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