Research paper

Aggregate size effect on the development of cementitious compounds in a lime-treated soil during curing

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ABSTRACT

This work aims to investigate the aggregate size effect on changes in mineralogical composition and microstructure of lime-treated compacted soils. Three soil powders with different maximum aggregate sizes (D_{max} = 5, 1 and 0.4 mm) were prepared prior to the treatment with 2% of lime. X-ray diffraction (XRD), environmental scanning electron microscope (Env. SEM) coupled with chemical analysis using energy dispersive X-ray spectrometry (EDX) and mercury intrusion porosimetry (MIP) were used to analyse untreated and treated samples at various curing times. Crystallized C-S-H on tobermorite form was identified in the lime-treated soil prepared with large aggregates after one year curing, and an evident increase in nanopores <0.1 μm C-S-H was also observed due to C-S-H creation. By contrast, in the case of smaller aggregates, no obvious C-S-H peaks were observed by XRD technique after the same curing time, even though some evidence of such phases are provided by Env. SEM coupled to EDX analysis. But a large amount of undetectable nanopores <6 nm (considering the MIP technical limitation) was supposed to be formed and could be attributed to the creation of nanocrystallized C-S-H or poorly-crystallized C-S-H (that may fill the pores larger than 2 μm). Such type of C-S-H phases occurred when lime was coated in thin layer on the large surface associated to lime-treated soil prepared with small aggregates.

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1. Introduction

Lime treatment is an effective soil improvement technique widely applied in the field of construction. It largely modifies the soil geotechnical properties through the physicochemical reactions within the lime-soil-water system (Boardman et al., 2001; Russo, 2005; Al-Mukhtar et al., 2010; Tang et al., 2011; Tran et al., 2014). When quicklime (CaO), soil and water are mixed together, hydration and ionization of quicklime immediately take place. Then, the Ca^{2+} ions in the pore water released by calcium hydroxide ($Ca(OH)_2$) are adsorbed by ion-exchange at clay minerals surface. The diffuse hydrous double layer surrounding the clay particles can be modified by the Ca^{2+} ion-exchange process, resulting in the flocculation-agglomeration of clay particles (Bell, 1996). These modifications of clay particles induced by lime addition will largely improve the workability of soil by reducing the plasticity, the swelling and shrinkage (Bell, 1989; Russo, 2005). In the long-term, the main reactions between lime and clay minerals are of pozzolanic nature which contributes significantly to the improvement of soil

mechanical behaviour in terms of shear strength, shear modulus, compression strength and compressibility (Rajasekaran and Narasimha Rao, 2002; Khattab et al., 2007; Consoli et al., 2009; Tang et al., 2011; Dong, 2013). The mechanical improvement is attributed to the cementitious compounds generated by the pozzolanic reaction, coating the soil particles and bonding them together (Bell, 1996; Onitsuka et al., 2001; Nalbantoglu, 2006).

Mineralogical studies of cementitious compounds have been undertaken in recent years. The cementitious compounds can be of various forms due to the different mineralogical composition of soils containing mainly clay minerals such as kaolinite, montmorillonite or illite, and other minerals like quartz and feldspars. Generally, the main cementitious compounds are calcium silicate hydrate (C-S-H), calcium aluminate hydrate (C-A-H) and calcium alumino-silicate hydrate (C-A-S-H) (Khattab, 2002; Ríos et al., 2009; Maubec, 2010; Al-Mukhtar et al., 2010). For lime-kaolinite mixture, the production of C-S-H, C-A-H and C-A-S-H was reported by many researchers (Goldberg and Klein, 1953; Eades and Grim, 1960; Glenn and Handy, 1963; Willoughby et al., 1968; Bell, 1996). C-S-H and C-A-H were also detected in the lime-treated montmorillonite (Bell, 1996; Hilt and Davidson, 1960). Arabi and Wild (1986) noted that C-S-H hydrates were present in the lime-

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Specific gravity G_s	Liquid limit w_L (%)	Plasticity index I_p (%)	Optimum water content w_{opt} (%)	Maximum dry density $\rho_{d, max}$ (Mg/m³)
2.70	51	23	17.9	1.76

treated marls containing illite, quartz and feldspar. Eades et al. (1962) also identified the production of C-S-H in the lime-treated quartz.

Even though many studies were performed on lime-treated soils, most of them focused on the lime-treated soil samples prepared in the laboratory. However, often lower performance of lime-treated soils and poor durability of lime treatment are observed in the field conditions. Puppala et al. (2006) reported about 40% lower for stiffness and 20 to 30% lower for strength in the case of treatment in field. Similar results were reported by other researchers (Horpibulsuk et al., 2006; Kavak and Akyarlı, 2007). Additionally, higher hydraulic conductivity and swelling potential of lime-treated soils in the field conditions were observed (Bozbey and Guler, 2006; Cuisinier and Deneele, 2008). There are several factors that can contribute to this difference between field and laboratory conditions. In addition to the climatic factors, especially the wetting/drying cycles and freezing/thawing cycles (Pardini et al., 1996; Guney et al., 2007; Tang et al., 2011; Stoltz et al., 2012), the aggregate size may play an essential role in the hydro-mechanical behaviour of lime-treated soils (Tang et al., 2011; Wang et al., 2015). The bender elements testing performed on the lime-treated samples prepared with different maximum aggregates sizes (D_{max} = 5, 2, 1 and 0.4 mm) revealed that lime-treated soils prepared with larger maximum aggregate size presented a relatively lower stiffness (Tang et al., 2011). Dong (2013) also indicated that the lime-treated soil prepared with larger aggregates was more sensitive to wetting/drying cycles. Note that aggregates are assemblages of adjacent soil particles in which the cohesive forces are larger than the disrupting force (Kemper and Chepil, 1965). In the field construction, the scarifying/pulverizing process is performed before the lime treatment to control the soil aggregate size. However, the size of soil aggregates in the field can still reach several centimetres, which is much larger than that of soil aggregates prepared in the laboratory before sample reconstruction. In the laboratory, natural tested soils are usually air-dried, ground and sieved into few millimetres. For example, Du et al. (2014) who studied the engineering properties and microstructure of the cement-stabilized contaminated soil, prepared the samples with kaolin clay which had a maximum aggregate size lower than 2 mm; while Cai et al. (2015) used reactive magnesia to treat in the laboratory a silty soil with the

same maximum aggregate size. Jiang et al. (2016) also reported that the used soil was first passed through the sieve with 0.5 mm size prior to treatment.

As the improvement in the mechanical behaviour of soils by lime treatment is proven to be primarily controlled by the cementitious compounds from the pozzolanic reactions, it is expected that the different behaviours of treated soils with various aggregate sizes can be also interpreted from mineralogical analysis. However, no studies have been conducted on this aspect. This constitutes the main objective of the present work. In this study, three different maximum aggregates sizes (D_{max} = 5, 1 and 0.4 mm) of soil powders were prepared before lime treatment. The creation of cementitious compounds was investigated by X-ray diffraction (XRD). In addition, environmental scanning electron microscope coupled with energy dispersive X-ray spectrometry (Env. SEM-EDX) and mercury intrusion porosimetry (MIP) were applied to investigate the microstructure and chemical composition of the treated soils.

2. Materials and methods

The tested soil was taken in Héricourt (France). It is a plastic silt with a clay-size fraction of 27%. The basic geotechnical properties of this silt given by Wang et al. (2016) are listed in Table 1. This soil corresponds to a silt of high plasticity (MH) following the Unified Soil Classification System (USCS). To prepare soil powders with different maximum aggregate sizes, natural soil was first air-dried, gently ground to crush the block of soil and passed through three target sieves (their maximum diameters, D_{max}, are 5, 1 and 0.4 mm, respectively). The large aggregates which could not pass through the sieve were ground manually until all particles passed through, ensuring no changes in mineralogical compositions during sieving (Tang et al., 2011). Afterwards, soil powders S5, S1 and S0.4 are obtained, with D_{max} equal to 5 mm for S5, 1 mm for S1, and 0.4 mm for S0.4. Fig. 1 shows the aggregate size distributions of the three soil powders, determined by dry sieving. Quicklime was used in this study and it has a high purity with a CaO content as high as 97.3%. Particle size analysis shows that 82.7%, 95.2% and 100% of this lime could pass through 80 μm, 200 μm and 2 mm sieves, respectively (Dong, 2013). A lime content of 2% by mass was selected as binder dosage.

The dry soils were firstly mixed thoroughly with 2% quicklime. Then, distilled water was added by spray into the dry soil-lime mixture to obtain the target water content (w = 17%, dry side of the optimum according to the proctor curve). Static compaction was performed after a mellowing period of 1 h, to prepare soil samples at the target dry density (ρ_d = 1.65 Mg/m³). The samples were carefully wrapped by plastic membrane and scotch tape immediately after compaction. The well-covered sample was stocked in a hermetic box for curing in a chamber at a temperature of $20 \pm 2\,°C$. Prior to mineralogical and microstructural analyses, the samples were freeze-dried following the procedure proposed by Delage and Pellerin (1984).

X-ray diffraction (XRD) analysis was performed on both untreated and treated samples. To prepare soil powder for this analysis, freeze-dried and oven-dried samples were crushed and ground to pass through a 32 μm sieve. After sieving, soil powders were mixed well manually in agate mortar and sprinkled gently in XRD sample holder using a 65 μm sieve. The top layer was removed carefully by cutting the surface with a thin razor blade leading to a smooth surface without compaction (such preparation allows to decrease the preferential orientation of clay particles). XRD patterns were obtained using a D8 Advance diffractometer from Bruker (θ-θ configuration, Cobalt anode, E = 35 kV, I = 40 mA, no monochromator, LynxEye detector). A continuous scan mode, between 3 and 80° 2 theta, at a rate of 1 s per 0.01° 2 theta was selected. Diffractograms were exploited with EVA program coupled with the ICPdf2 mineralogical database.

Environmental scanning electron microscope (Env. SEM, Quanta 400 from FEI company) coupled with energy dispersive X-ray spectrometry
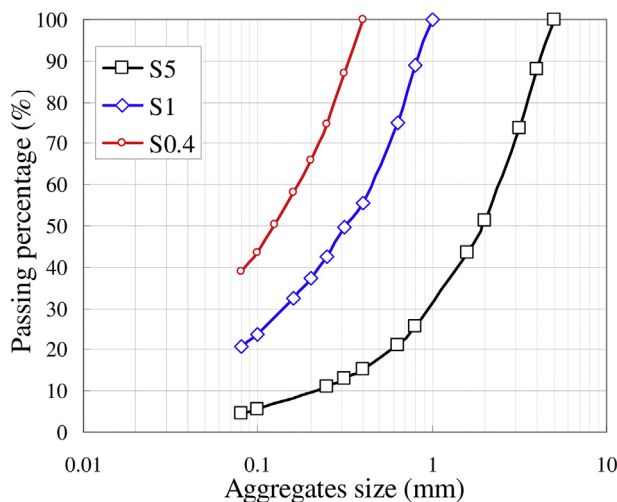
Figure 1. Aggregate size distributions of soil S5, S1 and S0.4.