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Research paper

Pore scale characterization of lime-treated sand–bentonite mixtures

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ABSTRACT

Lime treatment of soils is a complex process which combines chemical and mechanical aspects of the soil behavior. The investigation presented here aims at understanding the effect of lime treatment of clayey soils by characterizing their microstructure evolution, along curing time, using X-Ray Micro-Computed Tomography (XRμCT) and Mercury Intrusion Porosimetry (MIP). Binary sand–bentonite mixtures are considered as a model material to simplify the soil microstructure and the diversity of phenomena involved in lime treatment. Samples containing 10%, 15% and 20% of bentonite and, respectively 90%, 85% and 80% of sand have been treated with 1% lime and compacted. Results in XRμCT show first that porosity is present at two scales: micropores within the bentonite aggregates and macropores between sand particles and bentonite aggregates. Micropores are shown to be exclusively saturated with water, while macropores are only full of air. Second, XRμCT images on the same sample at different curing times show the migration of lime enriched aggregates diffusing into bentonite during the first weeks of curing. Third, bentonite is shown to shrink progressively and to form clusters around the sand grains. Consequently, the fraction of macropores increases while the micropore size decreases. On the other hand, through MIP, three pore size categories have been determined: micropores, mesopores and macropores. The evolution in time of the three pore size categories seen in MIP confirms the behavior observed by XRμCT.

1. Introduction

Lime treatment of soils is widely used in civil engineering in order to increase the soil mechanical properties such as improved cohesion levels and load bearing capacities. Lime, calcium oxide or hydroxide, is an industrial mineral coming from the decarbonation process of calcium carbonate rocks by heating. Silty and clayey soils can be improved by the addition of small percentages of lime (Little, 1964). The advantage of this treatment lies in the low quantity of lime added and the potential related ecological advantages obtained by improving the properties of the soil already in place without requiring replacement. Lime treatment influences the soil behavior on two different time scales. First, lime quickly reacts with clay by modifying its structure, and allowing the clay minerals to merge to form larger aggregates (Little, 1964). Lime modification improves the soil towards a higher load-bearing capacity, a lower plasticity and a shift towards higher grain size distributions. The second effect is soil stabilization owing to the fact that long term pozzolanic reactions also take place after soil modification (Eades et al., 1962). Mineral formations obtained from pozzolanic reactions indeed confer relevant soil mechanical properties such as a higher cohesion level (Thompson, 1965), higher compressive/tensile strengths and frost resistance (Arabi et al., 1989). In lime-treated clayey soils, such reactions take place between the calcium of the lime and the silicates and aluminates of the clay minerals; and CSH (calcium silicate hydrate), CAH (calcium aluminate hydrate) and CASH (calcium aluminium silicate hydrate) are formed (Diamond and Kinter, 1965). However, the reaction kinetics is slow because it requires the dissolution of clay minerals into silicate and aluminate species and this dissolution is only possible for highly alkaline solutions ($pH > 10$) (Keller, 1964). Research on soil stabilization has been active during the last decades. Bell (1996), De Bel et al. (2009), Diamond and Kinter (1965) and many others observed an increase of the unconfined compressive strength (UCS) in lime-treated soils as a function of time. Many important parameters influence soil stabilization, such as the water content and the dry density of soil (Locat et al., 1990). Also, higher temperatures increase the speed of the reaction (De Bel et al., 2009), whereas the presence of organic matter could decrease the efficiency of lime (Locat et al., 1990). In addition, the clay mineral type is an important parameter of soil stabilization. Montmorillonite, for example, has a better efficiency for lime adsorption than kaolinite (Carroll, 1959), illustrating the importance to consider the cation exchange capacity (CEC) in the assessment of lime treatment.

In order to build a progressive understanding of lime treatment, this study aims at characterizing its influence on the microstructure of soils. This contribution combines MIP and XRμCT techniques in order to investigate the time dependent microstructural evolutions in lime-treated sand–bentonite controlled mixtures. The combination of these

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tools provides a visual interpretation with XRμCT as well as the quantitative MIP results on the micro-scale, and thereby sheds light on the microstructure development linked to lime treatment of soils. This study also clarifies the reorganization of a soil composed of a lime-reactive fraction (bentonite) and a non-reactive fraction (sand), as well as the influence of their proportion on the kinetics of the evolution and in the formation of a more or less cohesive matrix after lime-treatment. Let us mention that other methods can also be suggested to study microscale changes in lime-treated soils such as X-Ray Diffraction, Thermogravimetric Analysis and Transmission and Scanning Electron Microscopy, see Al-Mukhtar et al. (2012) for a review.

X-Ray Computed Tomography made its way to geosciences in the past decade for which a wide range of issues can be addressed. Its implementation is based on the computer processing of numerous snapshots of the sample taken at different angles by an X-Ray source. Since X-Rays pass through matter with a level of absorption that depends on the local density and atomic number, the snapshots represent the local X-Ray absorptivities of the sample. Computer processing allows further recombining the snapshots to form the entire 3D reconstruction of the object. A review for the use of XRμCT in geosciences can be found in works by Mees et al. (2003), Ketcham and Carlson (2011) and Desrues et al. (2006). Tomography carries the main advantage that the micro-fabric of materials is not disturbed by the observation technique, and was therefore used for various characterizations in a geomechanical context, in which the evaluation of densities, water content and volume fractions at small scale is of particular interest (Anderson et al., 1988; Taud et al., 2005; Riedel et al., 2012). Also, it is a powerful tool in order to follow local deformations as performed in Lenoir et al. (2007) for rock materials and Desrues et al. (1996) as well as Hall et al. (2010) and Higo et al. (2011) for sandy soils. Investigations on bentonite-based mixtures were also studied recently. In Kawaragi et al. (2009), the microstructure of sand–bentonite mixtures was analyzed through XRμCT for permeability studies for sealing plugs of radioactive waste disposal. In Saba et al. (2014), XRμCT was used to understand the effect of swelling non-homogeneities of a sand–bentonite mixture throughout the sample. Lime treatment investigation with XRμCT was recently carried out by Lemaire et al. (2013). Results show that lime binders aggregate the silt particles during short term soil modification and form a strong shell structure surrounding the aggregates during the long term soil stabilization. This shell structure in silty soils was also studied before by Cabane (2004). Both studies concluded with conceptual models of silty soils forming aggregates covered by strong lime-treated shells. As a complement, the present research aims at investigating the effect of lime on clayey soils and at characterizing the lime treatment behavior on these types of soils with a complementary conceptual model as well.

Table 1
Properties of the sand and bentonite.

Sand:	
Sibelco© Mol M32	
D_{50} (μm)	260
$C_u = D_{60}/D_{10}$	1.5
ρ_s (g/cm^3)	2.65
Bentonite:	
Ibeco© Deponit CA	
Fine particles (<2 μm)	65%
Silt (2 μm > D > 67 μm)	28%
Sand (>67 μm)	7%
ρ_s (g/cm^3)	2.72
Methylene-blue value (mg/g)	300 ± 30
CEC (meq/100 g)	60 ± 10
Water absorption capacity	≥160%
Free swelling index (ml/2 g)	≥7
Liquid limit	115%
Plastic limit	33%
Plasticity index (calculated)	82%

Table 2
Ingredients for the three different mixtures: the lime quantity is the same and the water content and density follows the MOP curve.

Mixture	10b	15b	20b
Bentonite %	10%	15%	20%
Sand %	90%	85%	80%
Lime %	+1%	+1%	+1%
Water %	+14%	+17%	+20%
ρ_d (g/cm^3)	1.74	1.70	1.64

Mercury Intrusion Porosimetry (MIP) is the second tool selected here to provide a quantitative characterization of the soil microstructure. It was recently applied for lime treatment research. For instance, Cuisinier et al. (2008) used MIP to investigate the effect of alkaline fluid circulation into a sand–bentonite mixture. Le Runigo et al. (2009) studied the microstructure of a lime treated compacted silt submitted to long-term leaching. Combined with suction control, MIP studies were also used in Stoltz et al. (2012) to investigate the effect of swelling and shrinkage on micropores and macropores in lime-treated expansive clayey soils. More recently, Tran et al. (2014) tracked the evolution of the pore size distribution of a lime-treated expansive clay during the first seven days of curing. They observed that macropores (i.e. inter-aggregate pores) increase in size while the intra-aggregate pore-size remains constant. They attribute this change to the hydration of lime that induces a macroscopic swelling. On the contrary, Cuisinier et al. (2013) observed the formation of a small pore population due to lime treatment. Similarly, Russo and Modoni (2013) and Cecconi and Russo (2013) noticed an increase of micropores frequency during curing time on compacted silty soils and pyroclastic soils, respectively, due to the formation of stable bonding compounds that splits large pores into a series of smaller pores. This is consistent with the MIP observations from Metelková et al. (2012) on a compacted and stabilized loess. To study the evolution of the microstructure of lime-treated soils upon wetting–drying cycles, Aldaood et al. (2014) and Khattab et al. (2007), performed MIP tests along curing times after various wetting–drying cycles. They concluded that those hydraulic cycles may have detrimental effects on the soil microstructure but they did not focus on the effect of curing time itself.

In the present study, mixtures of sand and bentonite have been investigated for three different proportions through XRμCT scanning and MIP at different curing times. XRμCT allows obtaining information about the proportion between interparticular macropores and micropores in the bentonite and the local evolution of bentonite due to the presence of lime. The evolution of micro–macro-pores through curing time is also investigated using MIP. Moreover, MIP was used to compare micro–macro-pore proportions with XRμCT measurements.

The paper is structured as follows. Section 2 presents the methodology used for both XRμCT and MIP analyses. The technologies of XRμCT and MIP, the samples preparation, the curing times and the data

Table 3
Summary of the 10 samples scanned under XRμCT. The x markers show the curing times at which the samples were scanned. (*) Accelerated curing at 45 °C after the first 7 days.

	Curing time (days)					
	7	14	17	21	56	105
10b7	x			x*		
15b/7	x		x*			
20b7	x	x*				
10b56					x	
15b56					x	
20b56					x	
10b105						x
15b105						x
20b105						x
15b3t	x	x		x		