

Multi-scale analysis of lime treated sand-bentonite mixtures

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ABSTRACT: Lime treatment of soil induces long term mechanisms of soil stabilization due to pozzolanic reactions between calcium ions brought by lime, clay minerals and water. The goal of the present study is to link the macroscopic evidences of soil stabilization with a progressive understanding of the processes occurring at micro-scale. To achieve this, sand-bentonite mixtures in different proportions have been considered: 10%, 15% and 20% of bentonite and, respectively, 90%, 85% and 80% of sand. First, experimental tests at macro-scale have been used such as Proctor compaction, UCS and lime consumption. Those tests provide clear indications about the evolution of the lime treatment with time. Mixtures with higher percentage of bentonite are found to react faster. At a finer scale, a micro-scale analysis is carried out through X-Ray computed micro-tomography (XR μ CT) followed by automated image segmentation. micro-scale results show that bentonite absorbs all the water and is saturated. Microvoids are filled with water whereas macrovoids are filled with air. Consequently, water content controls bentonite density within the mixture. The ratio of lime consumption by the amount of bentonite shows a fairly constant rate decreasing slightly with increasing bentonite contents possibly due to the bentonite dry density.

Keywords — X-Ray micro-tomography, lime treatment, sand-bentonite mixture

1 INTRODUCTION

Clayey soils can be stabilized by the addition of small percentages, by weight, of lime. The efficiency of this treatment lies in the low quantity of lime added and the potential related ecological advantages obtained by using the soil already in place without requiring soil replacement. Lime treatment influences the soil behaviour on two different time scales. First, lime quickly reacts with clay by modifying its structure, and allowing the clay minerals to merge and form larger aggregates (Little 1964). The second effect is soil stabilization owing to the fact that long term pozzolanic reactions also take place after soil modification (Eades, Nichols, & Grim 1962). CSH (Calcium Silicate Hydrate) and CAH (Calcium Aluminate Hydrate) formations obtained from pozzolanic

reactions improve the soil mechanical properties. In clayey soils treated with lime, the reaction takes place between the calcium of the lime and the silicates and aluminates of the clay minerals. However, the reaction kinetics is slow because it requires the dissolution of clay minerals into silicium and aluminium ions. This dissolution is possible only for high alkaline solutions (pH <10) (Keller 1964). Research on soil stabilization has been active during the last decades. Estéoule & Perret (1979) and De Bel, Gomes, & Verbrugge (2009) observed an increase on the unconfined compressive strength (UCS) as a function of time. Many important parameters influence soil stabilization, such as water content and dry density of soil (Locat, Bérubé, & Choquette 1990). Also, higher temperatures increase the speed of the reaction (Estéoule & Perret 1979), (De Bel, Gomes, & Verbrugge 2009) whereas the presence of organic matter decreases the efficiency of lime (Locat, Bérubé, & Choquette 1990). In addition, the clay mineral type is an important pa-

parameter of soil stabilization (Bell 1996). Montmorillonite, for example, has a better efficiency for lime adsorption than kaolinite. Consequently, the cation exchange capacity (CEC) is an important factor to be considered.

This paper contributes to the understanding of the effect of a small amount of lime in sand-bentonite mixtures. In the present work, different proportions of sand-bentonite mixtures have been studied, for tests carried out at low bentonite contents : 10%, 15% and 20% in weight of bentonite (corresponding respectively to 90%, 85%, 80% in weight of sand).

The first part of the study makes use of macro-scale characterization tools. Samples have been subjected to unconfined compression tests and lime consumption analysis at different curing times up to 112 days. A second set of results relates to micro-scale characterization tools. Samples at three different proportions have been studied using X-Ray Tomography. 3-D images have been analysed based on image segmentation for volume proportion calculation.

2 METHODOLOGY

2.1 Soil materials

The sand considered is homometric with a D_{50} of $260\mu m$ (i.e. the particles have all more or less the same size, $D_{60}/D_{10} = 1.5$), with the motivation to keep the sand easiest to consider in subsequent theoretical and numerical modeling. It corresponds to the skeleton of the mixture. The second constituent of the mixture is bentonite, which provides for the clayey cohesive matrix that reacts with lime. Bentonite is selected because of its high reactivity with lime (principally montmorillonite) and its availability in the market. Since sodium bentonite is known to have a very high swelling index, calcium bentonite is chosen to avoid any excessive swelling upon wetting ($7ml/2g$). It has 65% of fine particles ($< 2\mu m$), 28% silt ($2\mu m > D > 67\mu m$) and 7% sand ($> 67\mu m$). All properties of the sand and bentonite are shown in Table 1. The lime used is a dry quicklime named Proviacal[©] from Lhoist R&D S.A.

A preliminary step before sample preparation is the determination of the Optimum Proctor Curve (OPC). The soil is mixed dry by hand with a precise lime quantity. The lime quantity can be calculated according to the Eades & Grim procedure (ASTM D-6276) (Eades and Grim, 1966). It has been decided to add 1% lime for the mixtures. Distilled water is then poured at different water contents corresponding to the OPC of each mixture and the soil is mixed by hand. Finally, the wet soil is put in a plastic bag to mellow for 24 hours at 20°C. After 24h, compaction takes place. The results give the Normal Proctor Optimum density and the Optimum water content to use for the samples preparation. Table 2 shows the summary of the contents for the three mixtures prepara-

Sand:	
Sibelco [©] Mol M32	
D_{50} (μm)	260
$C_u = D_{60}/D_{10}$	1.5
ρ_s (g/cm)	2.648
ρ_d (g/cm)	1.5
Bentonite:	
Calziumbentonit Ibeco [©] Deponit CA	
Fine particles ($< 2\mu m$)	65%
Silt ($2\mu m > D > 67\mu m$)	28%
Sand ($> 67\mu m$)	7%
ρ_s (g/cm)	2.720
ρ_d (g/cm)	0.8±0.005
Methylen-blue-adsorption (mg/g)	300±30
Cation exchange capacity (mval/100g)	60±10
Water absorption capacity	≥ 160%
Swelling index (ml/2g)	≥ 7
Liquid limit	115%
Plastic limit	33%
Plasticity index (calculated)	82%

Table 1: Properties of the sand and bentonite

Mixture	10%-90%	15%-85%	20%-80%
Bentonite %	10%	15%	20%
Sand %	90%	85%	80%
Lime %	+1%	+1%	+1%
Water %	+14%	+17%	+20%
ρ_d (g/cm)	1.741	1.702	1.638

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

tion.

2.2 Macro-scale study

Unconfined compression tests allow measuring both the compression resistance and stiffness of the mixtures. Five samples of each composition and for each curing time have been prepared at 98.5% OPN according to the same procedure as Proctor. Their dimensions are of 70mm length and 36mm diameter. In order to avoid any exchange with the environment, the sample is protected by a plastic film, an aluminum film and a layer of paraffin. The samples are then stored at 20°C and stay for curing. Afterwards, unconfined compression tests are performed to determine the force vs. displacement curve and obtain the unconfined compression strength (UCS).

Another test, the Leduc Method followed by the norm NF EN 459-2/2001, has also been carried out on each sample in order to determine the lime consumption of the samples as a function of curing time. Unreacted lime is extracted with this norm by mixing the soil in a highly concentrated solution of saccharose.

2.3 Micro-scale study

X-ray computed tomography (X-ray CT) is a powerful noninvasive technique to obtain a three-

dimensional representation of objects. From the radiograms of the objects, the computer treatment of the set of images allows obtaining a full three-dimensional gray-level picture representing the X-ray attenuation of the material at the considered location. X-Ray CT made its way to geosciences for which a wide range of issues can be treated. A review of such applications of X-Ray CT can be found in (Mees, Swennen, Van Geet, & Jacobs 2003), (Ketcham & Carlson 2011), (Desrues, Viggiani, & Besuelle 2006). Tomography carries the main advantage that the micro-fabric of the observed materials is not disturbed by the observation technique resulting in the method being non-invasive. The tomography applications for porous media are mainly focused on (i) the analysis of porosity and fluid flow (Peth 2010), (ii) the evaluation of densities, water contents and volume fractions in general (Anderson, Gantzer, & Boone 1988), (Taud, Martinez-Angeles, Parrot, & Hernandez-Escobedo 2005), (Riedel, Ando, Salager, Bésuelle, & Viggiani 2012) and (iii) the characterization of asphalt and concrete microstructures (Shashidar 1999), (Wang, Paul, Harman, & D'Angelo 2004). X-Ray Tomography is particularly interesting in order to better understand the macroscopic behaviour of the sand-bentonite mixtures in the microscopic level (Saba, Barnichon, Cui, Tang, & Delage 2014)

In the present case, micro-scale characterization of sand-bentonite mixtures is important mainly for three characterizing properties :

- the sand-bentonite interactions,
- the proportion of macrovoids and microvoids and
- the density of the bentonite phase.

The first property remains a perspective in view of quantification and will be left out of the scope of this paper. Only the second and third properties are therefore presented in the results section of the paper. In order to quantify those properties from tomographic images, image segmentation is required.

The main challenge of image segmentation consists of treating 3D tomographic data to distinguish the different phases in the heterogeneous material, based on the voxels gray values. With the naked eye, it seems possible to distinguish the regions occupied by each phase. However, due to the large number of particles in granular materials, visual intuition is practically not sufficient. The phase segmentation must therefore be automated using computational methods to obtain quantitative results. Numerical criteria must be defined to extract phases boundaries in a fast, easy and systematic procedure. This treatment aims at the partition of the image into a limited number of entities. A complete overview of digital image processing is presented in (Pratt 2007). However, the image segmentation used here has been developed in the course

of our research and interested readers are referred to (Hashemi, Khaddour, François, Massart, & Salager 2013).

3 MACRO-SCALE RESULTS

3.1 Unconfined compression tests

Figure 1 shows the evolution of the UCS of the mixtures. The composition with the highest UCS depends on the curing time. At the beginning (i.e. 7 to 14 days), the mixture with 20% of bentonite exhibits the highest UCS. Then, at 28 and 56 days, the mixture at 15% presents the maximum value. Finally, after 112 days, the mixture of 10% overtakes all the previous mixtures and has the maximum UCS. All the mixtures have been treated equally at 1% of lime as specified in the Eades & Grim test. The UCS of mixtures of 15% and 20% bentonite stopped increasing after 56 days their maximal UCS because all lime was consumed, whereas with the mixture at 10%, the reaction is slower and the UCS keeps increasing after 56 days.

3.2 Lime consumption

First, the lime consumption of the highest bentonite content (i.e. 20%) decreases more rapidly than the two others (i.e. 10% & 15%). At 7 days, the amount of unreacted lime is higher for the 10% mixture than for the 20% mixture. Second, the UCS of the mixture at 10% keeps increasing at long curing time whereas the other mixtures have already reached a constant value after 56 days, see Figure 1. This is consistent with the lime consumption evolution reported in Figure 2. The decrease of lime content is important for the 10% bentonite mixture between 28 and 112 days. On the other hand, for the two other mixtures, the lime consumption seems to have stabilized after 56 days. This quantity of lime was not sufficient for mixtures at 15% and 20% bentonite.

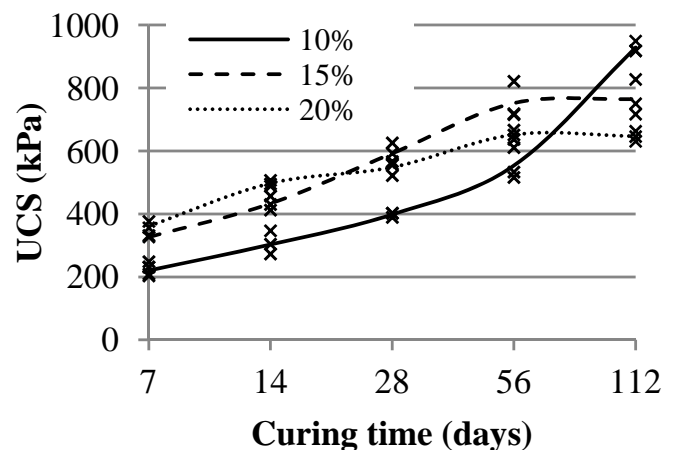


Figure 1: Evolution of UCS for the three mixtures treated with 1% lime as a function of curing time. The reaction stops prematurely after 56 days for the mixtures 15% and 20%, showing that not enough lime was added.

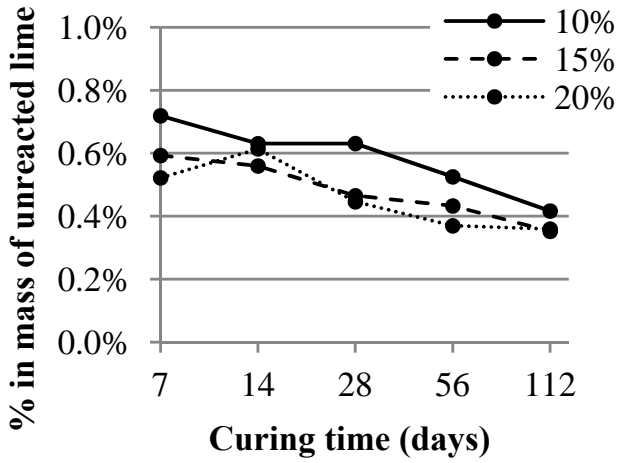


Figure 2: Lime consumption of the three mixtures as a function of curing time. Mixtures of 15% and 20% bentonite react faster than the one with 10%.

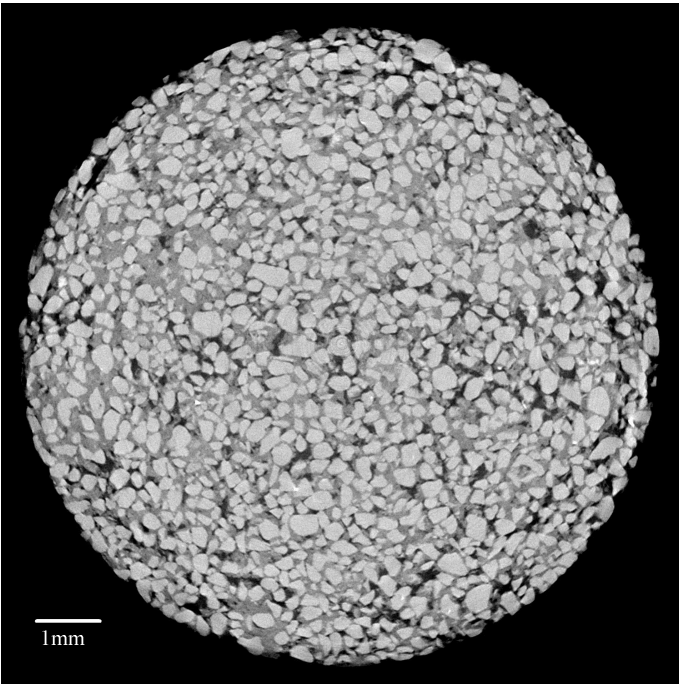


Figure 3: Cut of a tomographic image of a lime treated sand-bentonite mixture

4 MICRO-SCALE RESULTS

4.1 X-Ray tomography

Three samples representing the mixtures at 10%, 15% and 20% of bentonite treated with lime were analysed after 7 days of curing time. The resolution of the images is $7\mu\text{m}/\text{voxel}$. Therefore, the dimensions of the samples used were 10mm height by 10mm diameter, creating images of $1500 \times 1500 \times 1500$ voxels. The mold containing the mixtures was made of Plexiglas, the container volume being measured with mercury. Knowing the density of the mixtures (which is 98.5% of the OPN density), the corresponding mass of soil was added into the container. All mass measurements were made with an analytical balance of 0.1mg readability. The soil was added with a chemical spatula. Therefore the compaction was made statically. Afterwards, the cap of the sample was put and insula-

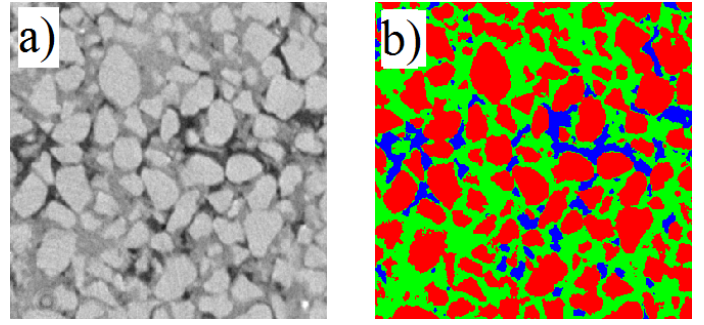


Figure 4: a) Raw image of a cut in the tomography of one of the mixtures in gray levels; b) Image segmentation technique aimed to separate sand, bentonite and macrovoid phases

tion made with scotch tape followed by paraffin coating. The samples were subsequently stored in a room maintained at 20°C for 7 days. The scans were produced in the 3S-R Laboratory of the Université Joseph Fourier in Grenoble, France.

As illustrated in Figure 3, the samples are composed of three phases : the sand, the bentonite and the macrovoids. The volume proportions of each component of the mixture can be calculated by determining its mass in the mixture and its specific density. Measurements of specific density for sand and bentonite have been made using the pycnometer whereas the water was supposed to have a density of 1. The volume of lime was neglected due to the low amount present in the mixture. Specific densities of sand and bentonite are shown in Table 1.

4.2 Image Segmentation

These three phases (sand, bentonite and macrovoids) present distinct gray values, but the image suffers from gaussian noise and partial volume effect and hence a simple threshold technique cannot be used to proceed to image segmentation. An image segmentation technique has therefore been developed in (Hashemi, Khaddour, François, Massart, & Salager 2013) in order to obtain a more accurate segmentation of the phases. Figure 4 shows on the left (a) the original image and on the right (b) the result of the image segmentation.

Based on the segmentation of the three images representing the three mixtures, it is possible to deduce volume proportions of each phase in the samples. Consequently, two properties can be calculated from the result : (i) the proportion between micro and macrovoids and (ii) the density of bentonite between the sand particles. These volume proportions are put with theoretical volume proportions for comparison in Figure 5. Results show that the macropores phase represent the air whereas the bentonite phase represents the bentonite fully saturated with water within its micropores. The micro-macro distribution of the sand-bentonite mixtures is consequently controlled by the water content.

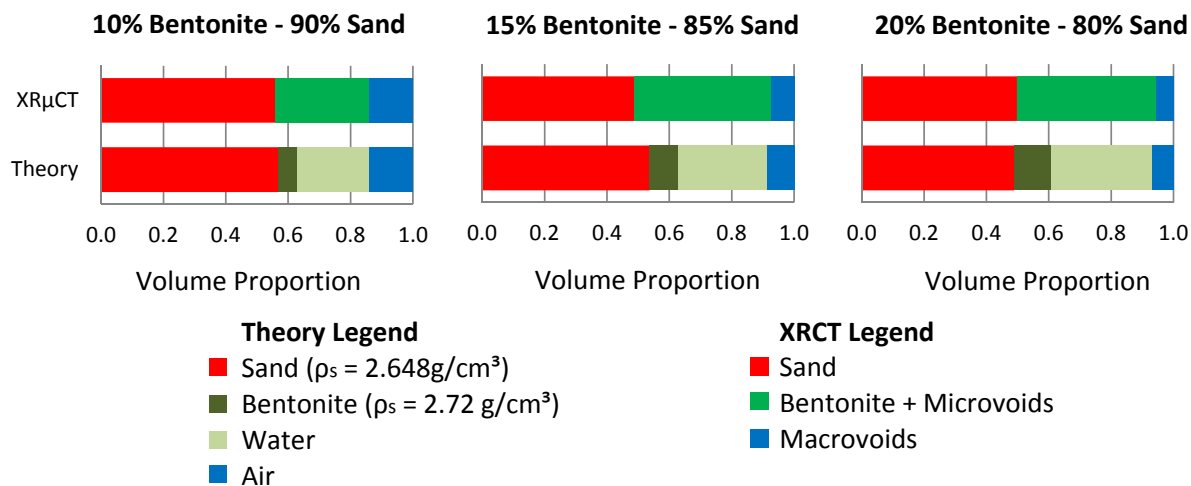


Figure 5: Comparison between theory and XRCT images show that microvoids within the bentonite are saturated with water and macrovoids represent air

Bentonite (%)	10%	15%	20%
Total w (%)	14%	17%	20%
Bentonite w (%)	140%	113%	100%
$\rho_{d,bent}(g/cm^3)$	0.55	0.67	0.73
$\Delta m_c(7d)/m_d$ (%)	0.28%	0.41%	0.48%
$\Delta m_c(7d)/m_{bent}$ (%)	2.8%	2.7%	2.4%

Table 3: Water content and dry density of the bentonite present within the mixtures supposing all water saturates the bentonite. Δm_c represents the mass of lime consumed after 7 days, showing a constant consumption per mass of bentonite with a slight decrease at higher bentonite content.

5 DISCUSSION AND CONCLUSIONS

Since water content is exclusively contained in the microvoids of the bentonite, it also controls its dry density. Table 3 shows the dry density of the bentonite for each mixture. Although the mixture of 20% bentonite has the highest water content per total weight, it has the least per weight bentonite.

Table 3 synthesizes the macroscopical characteristics of the three mixtures. Δm_c represents the amount of lime consumed during the first 7 days of curing time. When compared with the total mass of dry soil, it is shown that the consumption of lime is higher for the mixture at 20% bentonite. However, if the consumption is compared with the mass of bentonite, the ratio is observed to be fairly constant with a slight decrease at higher bentonite contents. The effect may be linked with the density of the bentonite which restricts the accessibility of the available lime by the bentonite platelets.

As a conclusion, in order to understand macroscopic evidences of soil stabilization, this study aims to investigate those effects at the micro-scale using X-Ray computed micro-tomography. Mixtures of sand and bentonite have been tested at three different proportions. Proportions of 10%, 15% and 20% of bentonite and 90%, 85% and 80% of sand with added water content of, respectively 14%, 17% and 20%

of total weight. All mixtures were treated with 1% of lime. First, macro-scale tests have been carried out with Unconfined Compression Strength and Lime Consumption as a function of curing time. Results have shown that the mixture with 20% bentonite reacts faster than the others but stops first (after only 56 days). The mixture with 15% bentonite is reacting slower than the previous. The mixture of 10% initially has a lower UCS, but it finally supersedes the other mixtures because its reaction continues. The mixtures of 15% and 20% stop their increase of UCS because of the lack of lime, some bentonite remaining unreacted in the mixture. Therefore, a necessary stoichiometry should be calculated beforehand in order to optimize soil stabilization with lime. On the other hand, based on micro-scale analysis, volume proportions of the different mixtures show that the micropores are concentrated in the bentonite and are exclusively filled with water, whereas macropores are outside the bentonite in the intergranular area and do not contain water. Consequently, the water content controls the porosity of the bentonite and thus its density. The lime consumption thus depends on two factors: the amount of bentonite in the mixture (giving the lime/bentonite ratio) and the water content (giving the water/bentonite ratio). It is shown that of the two factors, the most influencing is the quantity of bentonite.

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