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Scanning Electron Microscope (SEM) investigations in soil microstructure description

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***Abstract* – Composed of discrete particles and particle groups, a soil mass or a reconstituted soil mass should be considered as a continuum for analysis and design. Even with the development of numerous techniques of investigations, the link between the macroscopic behavior and the particle arrangement in soils is still under debate owing the complexity of the system, the different levels of investigations. However the shape of the particles, the particle arrangements, the strength of the particles or the water content of a soil govern the soil microstructure or fabric and the macroscopic properties as the permeability, the compressibility and strength. Some examples presented in this paper illustrate how the use of conventional scanning electron microscope (SEM) can be useful in the description of soil fabric and bring information in the understanding of the soil properties.**

Introduction

The analysis of elementary components of a soil and their arrangement is useful in the understanding of its macroscopic properties. In many geotechnical applications, the soil fabric or microstructure is assessed in order to understand the state and the evolution of a soil under various stresses and chemical environment.

In the context of sustainable development, there is an increasing need of retrieving and re-employing the materials encountered in the vicinity of the earthwork projects, whatever their nature or water content. Soil stabilization with lime and/or cement is a widely used technique, under continuous development, allowing fitting with this objective. The use of lime can improve macroscopic properties of numerous soils but depending of the soil, of the water content and the amount of lime used, the benefit of the lime addition and the macroscopic response can be different. The description of the microstructure and a multi-scale approach are now largely used to understand in detail the reactivity of the system and the macroscopic differences recorded. This paper tends to show how and why the use of SEM can be useful in the understanding of the soil microstructure by focusing 1) on the role of the compaction system and the water content on the soil microstructure, 2) the modification of the microstructure after the lime addition and the development of the pozzolanic reaction.

1 - Influence of the mode of compaction and water on the silt microstructure

The first example concerns the description of the influence of compaction procedures and of the water content on the microstructure properties of a natural soil, sampled in the vicinity of Paris, near the city of St Quentin. After sampling, the silt was homogenized, its water content after preparation being about 14.3%. About 90% of the particles were smaller than 80µm and 25% were lower than 2µm. The liquid limit was equal to 32.5% and its plastic index to 12.7%. The specific weight of the particles was equal to 2.68 $Mg \cdot m^{-3}$. The X-ray diffraction analysis showed that it contained mainly quartz, while the clay fraction was predominantly composed of kaolinite. According to French soil classification [1], that soil is a low to medium plasticity silt. It was classified A-6 in the AASHTO soil classification system and ML in the Unified Soil Classification System.

Two compaction procedures were selected in this study since it has been recognized as a key parameter of the hydraulic conductivity of compacted soil. The first procedure was the standard Proctor compaction procedure outlined in ASTM D698. The second one was the kneading compaction procedure proposed by Kouassi et al. [2]. That reproduces in the laboratory the on-site effects of a kneading compactor. In this procedure, the compaction mold size is the same as in the standard dynamic compaction procedure (California bearing ratio molds). The compaction was carried out with a custom-made steel tool under which three steel kneading tampers (50 mm in diameter and 25 mm in height) are fixed. These dimensions were set so as to have two parameters

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identical in the field and in the laboratory: the surface layer percentage covered by the surface of the feet and the ratio between the thickness of field compactor and the foot length. The compactor used as a reference for the design of the laboratory compacting tool is the tamping roller Caterpillar 825 C. For the compaction, the kneading tool was fixed on the loading frame. The kneading tool was then applied to the soil up to a target load. Then, the soil was unloaded and the kneading tool was rotated for 45°. Eight load applications by layer were done. For the untreated St Quentin silt, several parameters of the kneading compaction procedure were adjusted, in order to obtain a similar compaction curve to that obtained with standard Proctor compaction. Repeatability was maximized when the soil was compacted in three layers, each kneading tool application performed under a load of 1.25MPa and a speed of the tool of 48 $mm \cdot s^{-1}$.

The microstructure of the compacted soil was investigated by SEM. For that, small specimens with a volume between 1 and $2cm^3$ were taken from the sample. Freeze-drying was selected for our study as an alternative to oven drying to prevent as much as possible the effects of shrinkage during drying on the sample microstructure. Soil pieces were quickly frozen with liquid nitrogen (temperature of −196°C) and then placed in a freeze-drier for 72h for the sublimation of the water. The samples were coated with gold for study with SEM HITACHI SX50 (LaB6) equipped with an energy dispersive X-ray spectrometer (Quantax 200) coupled with a Bruker detector SDD X-Flash. The microscope was operated at an accelerating voltage of 15 kV. Scanning electron micrographs allowed the visualization of the morphology and spatial arrangement of the studied materials.

Both compaction procedures led to the same compaction curve in the case of the untreated St Quentin silt. SEM examination shows, however, a big difference in the particle organization. In the case of the dynamic compaction at the optimum water content, the microstructure is represented by a granular skeleton composed of quartz and feldspar grains covered by the clay particles (Image 1 & 2). This organization induces two classes of pores, with macropores between the covered grains and micropores at the level of the clay particles. This arrangement of particle is modified by increasing the water content (Image 3). At higher water content, the clayey particles are dispersed between the grains of quartz and feldspar, decreasing the porosity and explaining the decrease of the hydraulic conductivity by two orders of magnitude. The significant reduction of the amount of macropores is evident and observed even with the kneading compaction method at high water amount. These observations allow explaining the MIP data [3] and are in agreement with previous findings on the influence of compaction water content on pore size distribution of

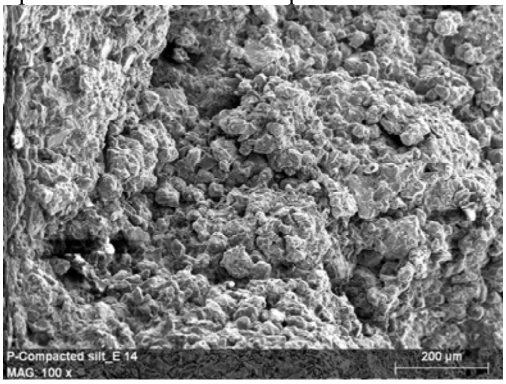

Image 1 – Initial silt, Proctor compaction at optimum water content – low magnification

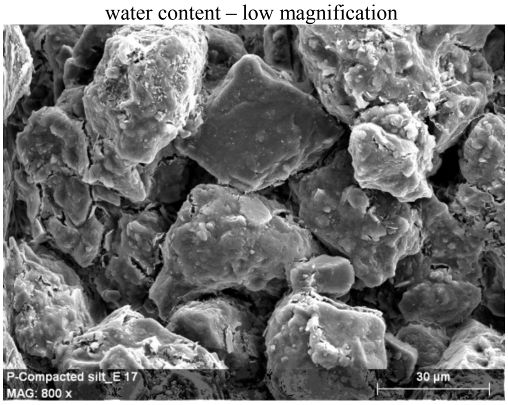

Image 2 – Initial silt Proctor compaction at optimum water content –high magnification

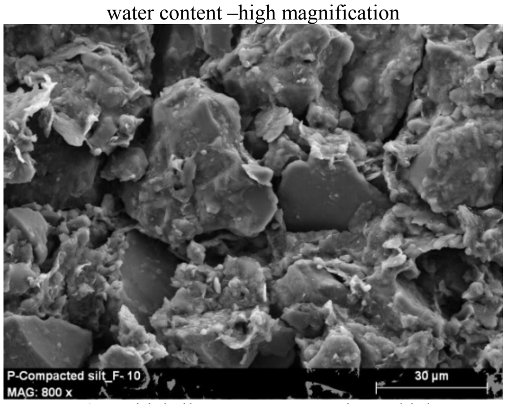

Image 3 – Initial silt Proctor compaction at high water content –high magnification

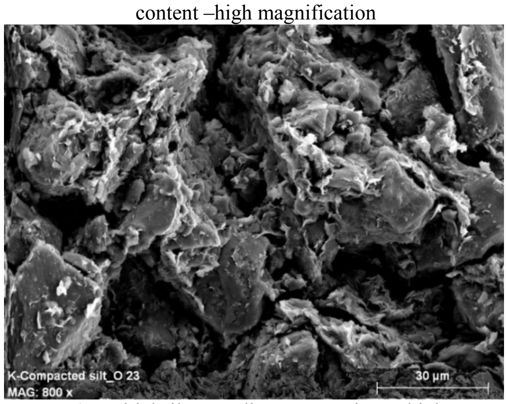

Image 4 – Initial silt Kneading compaction at high water content –high magnification compacted silt [4]. The SEM observation confirm that the maximum pore diameter was lower in the case of the sample compacted by kneading at high water content.

If the water content modifies the distribution of clay fraction within the granular skeleton and the microstructural properties, the investigation was completed by the study of the microstructure after the lime addition.

2 – Influence of the lime addition on soil microstructure

SEM examination was done in order to understand how the microstructure of the soil is modified after the lime addition. The lime amount tested in this study was 3% of quicklime. The selected quicklime contained more than 90.9% of available CaO on a dry-weight basis (measured using EN 459–2).

The lime addition deeply modified the soil microstructure after 28 days of curing, as observed from MIP analysis [3], showing a trimodal pore size distribution, with the formation of a new small class, with a diameter lower than 3×10^3 Å in the compacted soil. This class was mentioned as responsible for the difference in dry density between the treated and the untreated sample after compaction. This class of pores exist whatever the compaction device and the lime used. The image 5 and 6, at low magnification show that the soil exhibits a closed structure after the lime addition, by comparing with the no-treated soil. At higher magnification (image 7), the microstructure is affected by the development of cement which links the soil particles. This cement was developed from the pozzolanic reaction. The pozzolanic reaction is induced by the high pH environment brought by the lime addition which dissolves the soil minerals, mainly the clayey minerals [5] and the calcium of the lime. That leads to the formation of secondary hydrates with characteristics and properties equivalent to the phases formed during the cement hydration: C-(A)-S-H and C-A-H.

The lime addition leads initially to the dispersion of lime nodules in the soil (Image 8), these nodules being progressively dissolved and converted into secondary phases with time. The calcium dispersed within the soil and the high pH of the water also contributes to the formation of pozzolanic compounds within the soil. These new phases are responsible of the increase of the soil resistance [6] and modify the soil microstructure. Observations at higher magnification (Image 10) confirm the amorphous character of these secondary phases with the development of a gel. Increasing the magnification, it appears that the gel presents some pores ranging between 250 and 400 nm (Image 12).

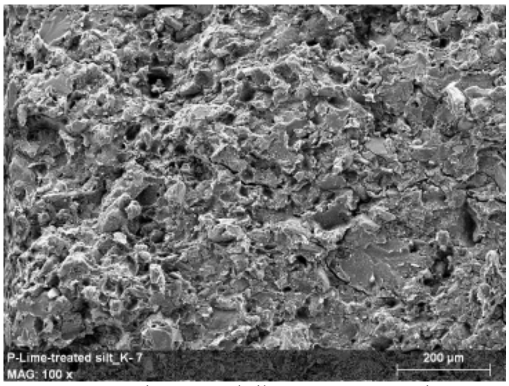

Image 5 – Lime-treated silt, Proctor compaction at optimum water content – low magnification

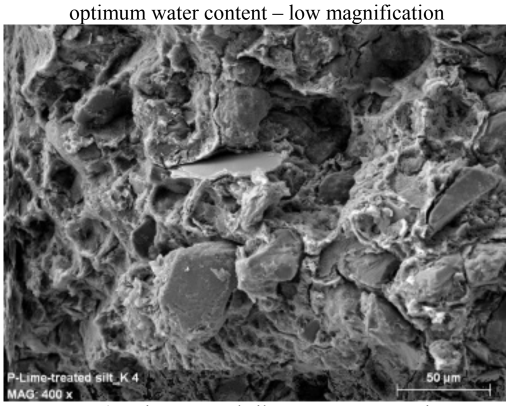

Image 6 – Lime-treated silt, Proctor compaction at optimum water content – low magnification

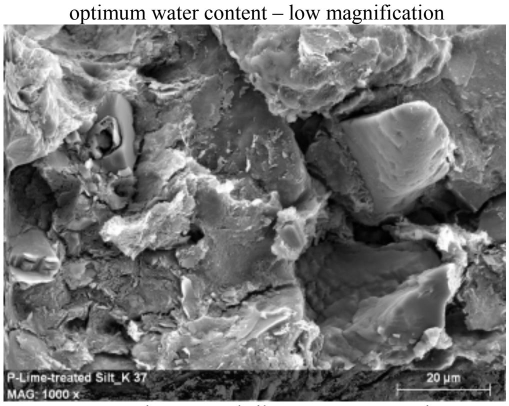

Image 7 – Lime-treated silt, Proctor compaction at optimum water content – high magnification

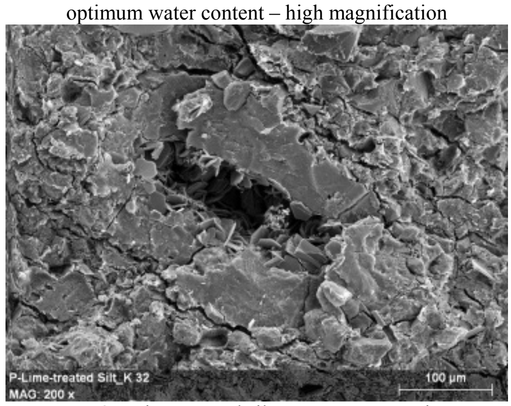

Image 8 – Lime-treated silt, Proctor compaction at optimum water content – Lime nodule and secondary phases (C-A-H, C-A-S-H)

These pores correspond to the new class of pores detected by MIP. Compaction at higher water content than the optimum, showed the whole range of pore radius was altered by lime treatment [3]. The origin of the reorganization under debate when the study was published can be now resolved. As in untreated soils, only the larger pores were modified by the compaction water content and the compaction procedure in the lime treated samples.

SEM observations of the silt compacted with Proctor after the lime addition at high water content, shows a closed structure with no distinction of agglomerates (Image 9). Remembering that the compaction at high water content increases the dispersion of fine particles (clayey particles mainly), the formation of secondary phases sprayed all over the soil contributes to close the porosity and improves the binder development. The amorphous character is evident (Image 10), like the covering character of these secondary phases. It seems that increasing the water content contributes to develop more secondary phases for the same amount of lime used. Depending of the degree of development of the gel, its morphology evolves and the typical honeycomb structure of C-S-H is visible, at the same time that the identification of C-A-H (Image 11). Finally, the surface of the aggregates is completely recovered by these secondary phases, modifying the microstructure and the porosity and also the resistance of the treated soil.

SEM observations allow to understand why and how the large class was absent in the specimens compacted on the wet side of the optimum. That showed the interest to combine MIP and SEM in the description of the microstructure since SEM helps to understand the modification observed by MIP. Moreover, the analysis of the phases with local beam allows to precise the chemical composition of these secondary phases.

The main consequence of lime addition was a significant decrease of the dry density of the St Quentin silt under a given compaction procedure [3]. The SEM observations showed that the modification in dry density due to the lime addition can be mainly attributed to an increase in the amount of small pores volume. It can be observed that the small pores volume was roughly constant for the lime-treated St Quentin silt. The compaction condition modified the macropore class which then can explain the modification of the hydraulic conductivity of the lime treated St Quentin silt and for the untreated silt. The distribution and the volume of the small pores did not depend on the compaction conditions.

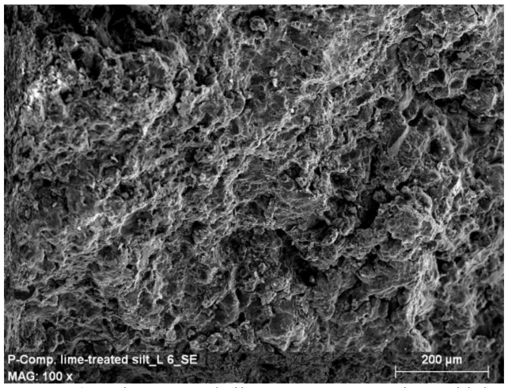

Image 9 – Lime-treated silt, Proctor compaction at high water content – Low magnification

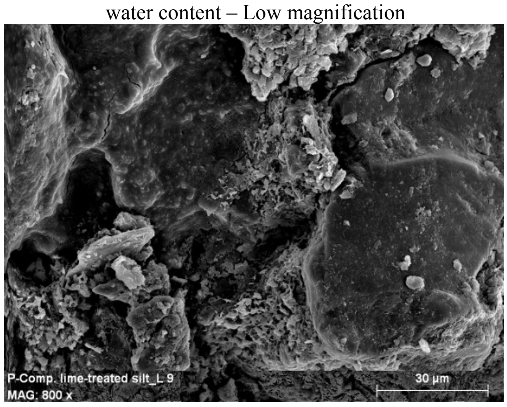

Image 10 – Lime-treated silt, Proctor compaction at high water content – High magnification

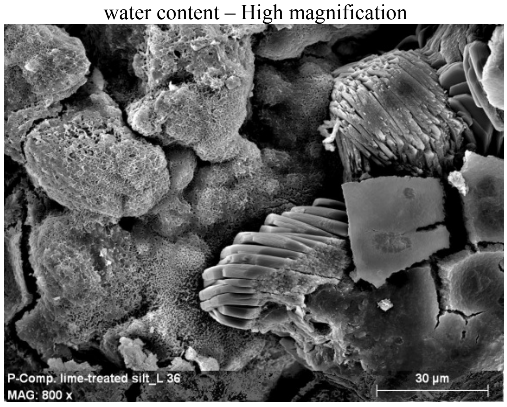

Image 11 – Lime-treated silt, Proctor compaction at high water content – High magnification

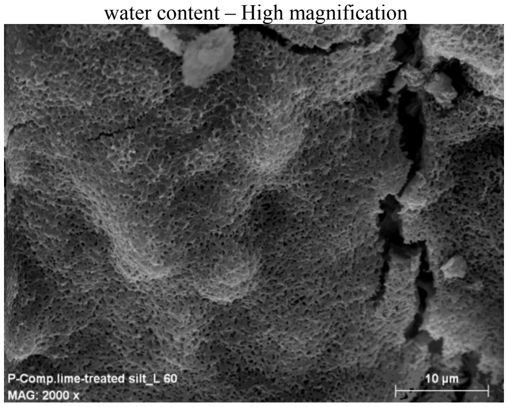

Image 12 – Lime-treated silt, Proctor compaction at high water content – High magnification

I. CONCLUSION

In the present paper, Scanning electron microscope was used to assess the microstructure of a soil for different conditions of compaction and after the lime addition.

The compaction method influences the distribution of particles and modifies the soil skeleton as the water content. The lime addition induced the development of an amorphous gel at the surface of the soil particles explaining the modification of the resistance. The extension of these secondary phases depends also of the initial dispersion of the clayey particles. This dispersion was dependent on the compaction method and the water content used for compaction. SEM observations allowed understanding the modification of the hydraulic conductivity of the treated soil.

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