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Binder-soil interaction in Cement Deep Mixing through SEM analysis

Interaction sol-ciment dans le processus de malaxage en profondeur, décrite par l'analyse microscopique (SEM)

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ABSTRACT
This paper deals with the SEM (Scanning electron microscope) analysis of improved soil. A natural dredged material from the Antwerp harbor was mixed with different binders (e.g. CEM I, CEM III). The soil and binders were mixed by means of the SSI (Soft Soil Improvement) technique in the field as well as with a dough mixer in the laboratory. An extensive SEM study was made on improved samples and natural soil to explain the strength behavior, looking at the microstructure of specimens.

RÉSUMÉ
Cette contribution traite les observations par microscope électronique de comportement des échantillons de sol de dragage, améliores aux différentiels types du ciment. Le malaxage au ciment du sol en place se faisait par la méthode SSI; au labo cela se faisait par simple malaxage mécanique. On a essaye a clarifier l'amélioration des échantillons traites par l'analyse de la structure microscopique.

1 INTRODUCTION

A natural dredged soil material was mixed with different types of cement binder for ground improvement purposes. The SSI (Soft Soil Improvement) technique was implemented in the field and 100 kg of the same soil was mixed in laboratory with different types of cement binders. The specific character of this SSI method has been described in another paper to this conference (Van Impe et al. 2005)

The unconfined compression strength (UCS) and other relevant parameters were measured on the improved dredged material as well as on the natural sludge. The large scatter found for each binder, as well as among the groups of samples from the field and laboratory, motivated the researches to look at the microstructure of improved samples and natural soil samples as well.

The paper deals with SEM (Scanning electron microscope) investigation of the natural dredged material, the dredged material improved by means of SSI method and the in laboratory prepared improved specimens.

2 SCANNING ELECTRON MICROSCOPE

The scanning electron microscope principle goes by scanning the surface of the specimen with an electron beam. In the interaction with the material, electrons are emitted from the specimen and are collected. This signal contains a variety of information about a single point on the specimen's surface and eventually produces the 3D image of the surface.

Whenever the electron beam interacts, X-rays are produced as well.

The result of X-ray analysis serves at the same time as chemical identification of the material by measuring the mass amount of different elements in the scanned area. This is the way the cement hydration products were identified, as well as the clayey, silty and organic particles of the soil.

3 PROPERTIES OF THE NATURAL SLUDGE

For purposes of ground improvement project, the natural dredged sludge was taken from the Doeldok site in the harbour of Antwerp. The physical and mechanical properties of the natural dredged material were measured and the unconfined compression strength varies from c_u = 2 to 4 kPa.

The fig. 1,2,3 are SEM pictures of untreated natural sludge specimen on the dried fracture surface with amplification x500, x1100 and x7500. The sets of pictures were taken on the sample surface.

The X-ray technique gave important additional data on recognizing clay minerals, silty particles, and organic content.

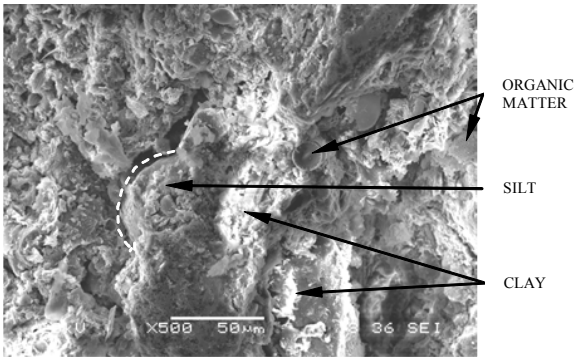


Figure 1. Untreated dredged material specimen – amplification factor x500

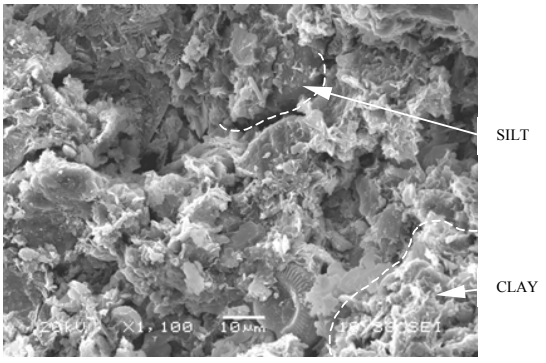


Figure 2. The natural sludge specimen – amplification factor x1100

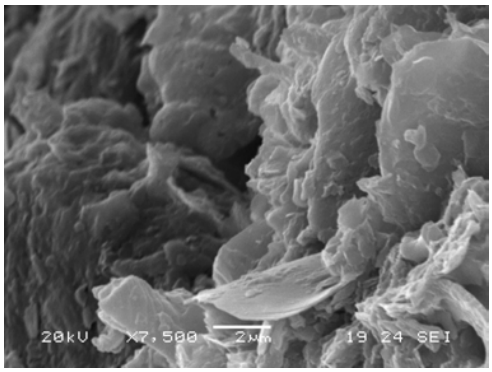


Figure 3. The natural sludge specimen – amplification factor x7500 – illite clay particles are shown here

In Figure 1 and 2 there are solid particles of silt, the smaller clay particles in brighter colour, and some organic matter apparently one mixed together. The silt is mostly covered with platy clay particles. The identification of clay and silt was obtained with the chemical analysis (X-ray) of SEM apparatus, showing the areas of high SiO content (silt SiO_2), or C (organic content), or mixture of Al-O-Mg and other elements characteristic for clay.

The Figure 3 shows a detailed structure of the platy illite clay particles with amplification factor x7500.

The natural dredged material apparently has a very heterogeneous microstructure, with visible voids and empty spaces, at all amplification levels, causing very low compressive strength of these specimens.

4 CEMENT TYPE USED FOR THE DREDGED MATERIAL IMPROVEMENT AND SPECIMEN PREPARATION

The dredged material improvement was done by the SSI method in the field and the mechanical mixing in laboratory, using different type of cement binders.

The analysed SSI field specimen all do reflect the CEM III/A type of blast furnace slag cement, with the slag content in a range 36-65% (binder B).

Table 2. Cements employed in the dredged material improvement

Binder designation	Classification
A	CEM III/A 32.5 LA
B	CEM III/A 42.5 N LA
C	Georoc
D	Blitzdaummer
E	CEM I 42.5 R HES
F	CEM I 52.5 N
G	CEM III/B 42.5 HSR LA

The samples were taken from the SSI columns in the field, brought to the laboratory and cured under water and in a conditioned room at 10^0C. The same curing procedure was done for the samples mixed in the laboratory with a concrete mixer. The amount of soil and cement mixed remained unchanged at 275 kg/m^3 in both procedures, at a W/C = 0,8 factor.

5 SEM INVESTIGATION OF THE SOIL MATERIAL, TREATED WITH DIFFERENT CEMENT TYPE

In Figure 4 some specimen from the field and from the lab treated with binder B were prepared for the SEM. The specimen from the field aged for 300 days, and the lab specimen were 270 days old.

The fractured surfaces were observed with low vacuum SEM, and remained neither coated with any film nor polished.

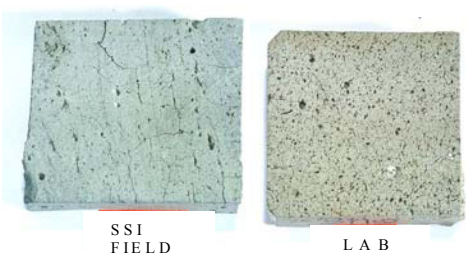


Figure 4. Specimen prepared for SEM investigation from the SSI column and the laboratory treated with **binder B**

Even the normal photo of specimen (x550) shows that the specimen prepared in the laboratory has lot of large pores (>5µm in diameter) and macro pores (>0.05µm in diameter). This is far less pronounced in the SSI improved field specimen, where a much more compact and more homogeneous texture can be seen. The mechanical cement mixing in the laboratory could have caused the incorporation of air bubbles, later on becoming large pores. Pores of smaller diameter were observed in both specimens; they could have been produced during the cement hydration process.

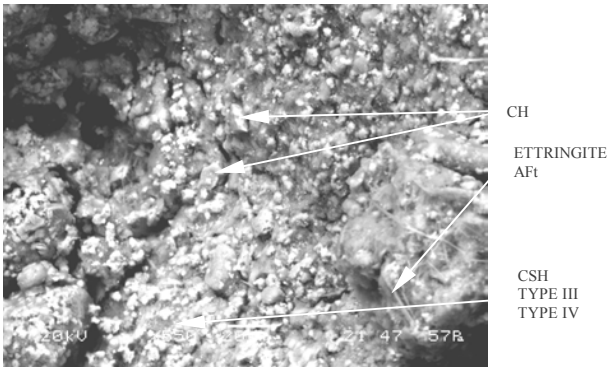


Figure 5. Field specimen with binder B with amplification factor x550

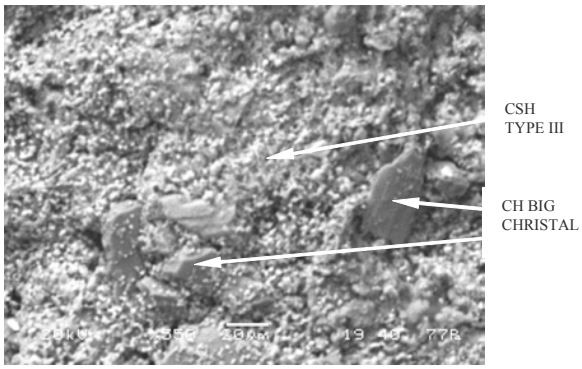


Figure 6. Lab specimen with binder B with amplification factor x550

The specimen cored out of the SSI column in the field, shows more C-S-H gel (calcium silicate hydrate) product, well interconnected, with more C-S-H III and IV; two sub-type characteristic for the late hydration phase. In the specimen prepared in the lab, there are only some C-S-H type I and II present; reflecting the early hydration stage of the cement paste.

The structure of the lab specimen is very heterogeneous as compared to the SSI specimen micro level,; showing also huge CH (calcium hydroxide) crystals that are characteristic for the early hydration of blast furnace slag cements in initially high water content field area.

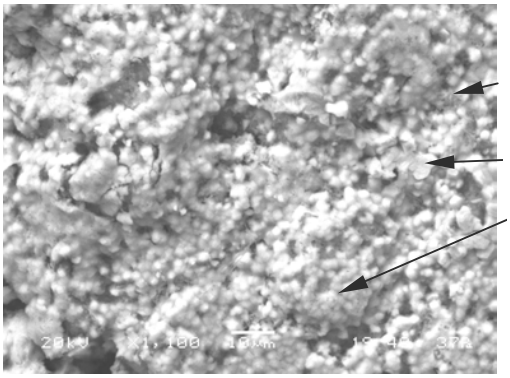


Figure 7. Field specimen with binder B with amplification factor x1100

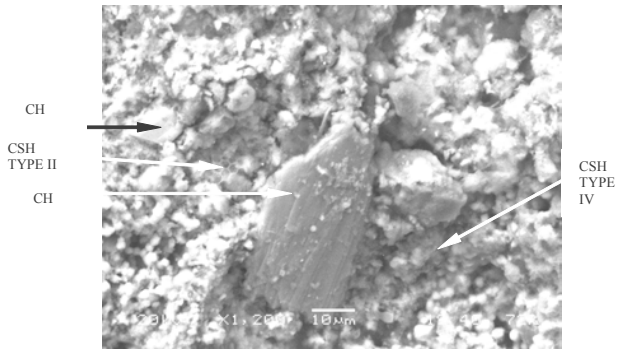


Figure 8. Lab specimens with binder B with amplification factor x1200

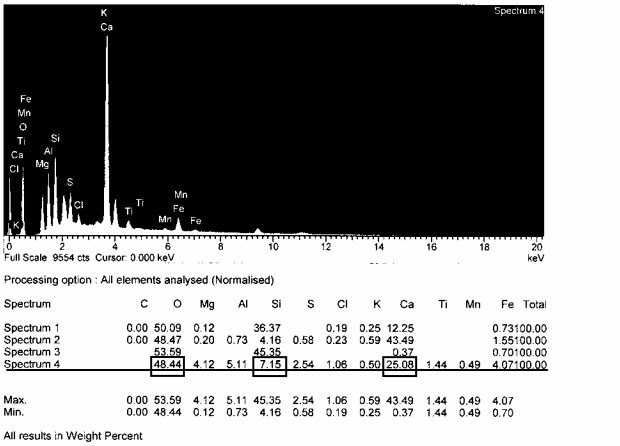
It all suggests the specimen with more voids and micro pores, and so less C-S-H of type III and IV, would have lower unconfined strength.

The X-ray identification of the chemical constituents of the hardened cement paste is shown below. Different type of cement hydration products lead also to a typical 3D pattern as found in SEM pictures of hardened cement paste.

There are four important morphological type of C-S-H (calcium silicate hydrate) gel, depending on the age, starting with the Type I as the fibrous material; the fibers being up to about 2μm long. Type II, usually described as forming honeycombs or reticular networks, is also a common early product. Type III, prominently present in "older" pastes, was denser and stage appears to consist of tightly packed grains up to 300 nm across. Type IV, still more featureless and massive, was the inner product, and was observed in the final older paste constitution.

CH (calcium hydroxide), grows within typical water filled pores where it forms isolated masses. Normally it leads to hexagonal plates (of Figure 14), but as hydration advances the main deposits of CH become massive and of indeterminate shape, though a good cleavage persists. Such cryptocristaline CH (seen in Figure 9) has been found in cement pastes and in pastes prepared from calcium oxide and silica of high w/c ratio and of blast furnace slag activated by CH.

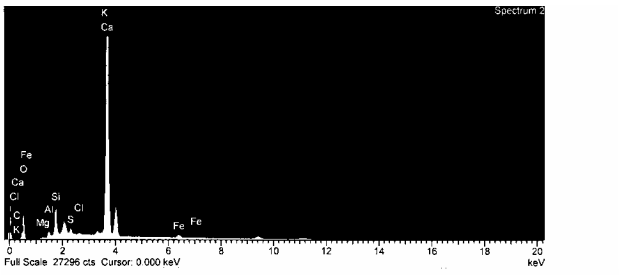
The ettringite – $Ca_3Al(OH)_6 12H_2O)*(SO_4)_3*2H_2O$ or AFt phase, is locally formed as well as in the pore voids. It is crystallized from the solution in needle like forms, which can be up to 10μm long (as seen in Figure 14). Ettringite peaks are detectable already within few hours and increase their intensity to a maximum at about 1 day old cement paste. The ettringite needles were even detected here in the sludge soil improved with binder E (CEM I) prepared in the laboratory, even though the sample was more than 240 days old. The ettringite indeed is supposed to break down again after a couple of days.



Spectrum	C	O	Mg	Al	Si	S	Cl	K	Ca	Ti	Mn	Fe	Total
Spectrum 1	0.00	50.09	0.12		36.37		0.19	0.25	12.25				0.73100.00
Spectrum 2	0.00	48.47	0.20	0.73	4.16	0.58	0.23	0.59	43.49				1.55100.00
Spectrum 3		53.59			45.35				0.37				0.70100.00
Spectrum 4		48.44	4.12	5.11	7.15	2.54	1.06	0.50	25.08	1.44	0.49	4.07	100.00
Max.	0.00	53.59	4.12	5.11	45.35	2.54	1.06	0.59	43.49	1.44	0.49	4.07	
Min.	0.00	48.44	0.12	0.73	4.16	0.58	0.19	0.25	0.37	1.44	0.49	0.70	

All results in Weight Percent

Figure 9. X-ray analysis of CSH gel



Spectrum	C	O	Mg	Al	Si	S	Cl	K	Ca	Fe	Total
Spectrum 1	0.00	50.09	0.12		36.37		0.19	0.25	12.25	0.73	100.00
Spectrum 2	0.00	48.47	0.20	0.73	4.16	0.58	0.23	0.59	43.49	1.55	100.00
Max.	0.00	50.09	0.20	0.73	36.37	0.58	0.23	0.59	43.49	1.55	
Min.	0.00	48.47	0.12	0.73	4.16	0.58	0.19	0.25	12.25	0.73	

All results in Weight Percent

Figure 10. X-ray analysis of CH

Figures 9 and 10 do show the X-ray based chemical composition of CSH gel and CH crystals. The CSH gel consists of high weight percent ages of calcium, silica and oxygen atoms. Some content of magnesium and aluminum atoms are also present in CSH gel suggesting that some clay particles do also participate to the cement hydration process. The hydrogen atoms cannot be identified in this procedure.

The Figures 12 and 11 illustrate laboratory mixed specimen of dredged material with binder G (blast furnace slag cement). If we compare those two Figures with the Figures 11 and 12, of the binder E, being pure Portland cement, differences in the fabric became visible. The blast furnace sample obviously has a more compact microstructure with more C-S-H product of later stage. The sample treated with binder E has less C-S-H product, more large CH crystals, and also some ettringite needles that are however not expected to be seen in old hydratated specimens, as well as lot of relatively big voids and empty areas.

In the Figure 14 the voids and empty spaces seem to be bigger and better interconnected than in picture 10. Figure 12 shows a lot of voids, ettringite and monosulfate plates characteristic for early hydration phase, which leads to serious doubts about the hydration process itself in the presence of soil material with binder E (Portland cement).

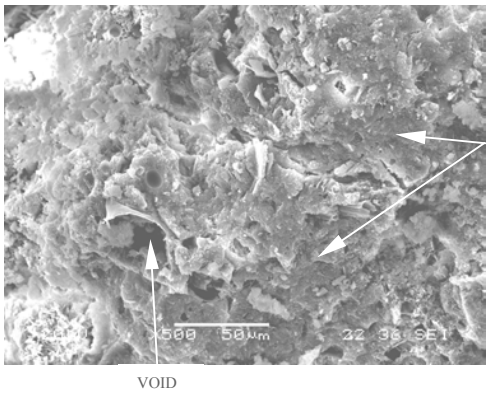


Figure 11. Lab specimens with binder G with amplification factor x550

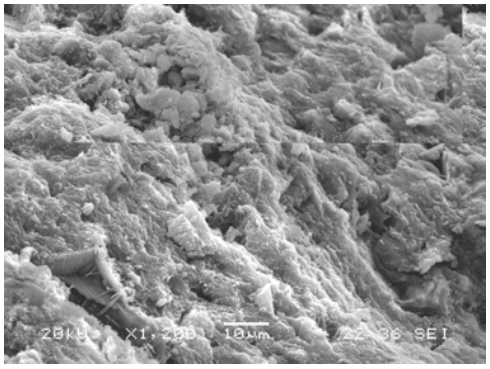


Figure 12. Lab specimen with binder G with amplification factor x1200

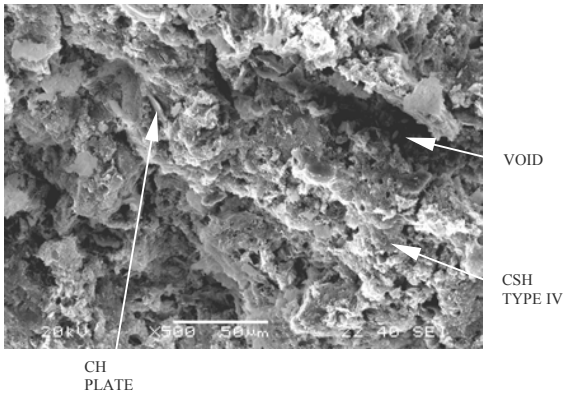


Figure 13. Lab specimen with binder E with amplification factor x550

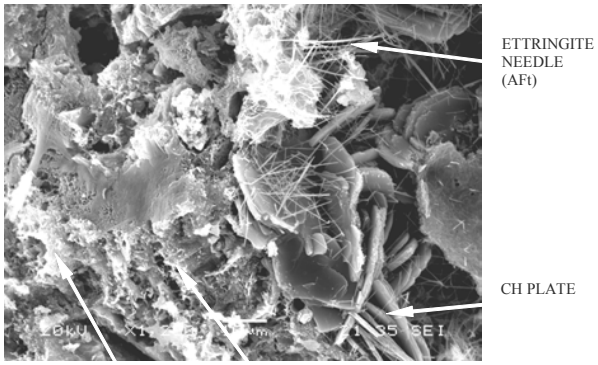


Figure 14. Lab specimen with binder E with amplification factor x1200

6 CONCLUSIONS

This paper deals with SEM investigation of dredged material improved with cement binders, by means of SSI field deep mixing method as well as through mixing the soil in the laboratory, all with cement based binders.

The results of unconfined strength (UC) analysis performed as a part of the extensive laboratory investigation programme showed huge discrepancies of the improved material behaviour(by means of such simple control tests as the UC). The strength development was however continuously followed for more that 500 days. The blast furnace slag cement treated soil (binders B and G) showed the highest values. The Portland cement treated specimen didn't show such positive performance (Van Impe et. al, IS OSAKA 2004). In order to explain such anomalies somewhat more rationally this SEM research was set up.

The SEM investigation results suggest that the hydration process of blast furnace slag cement was indeed quicker and the C-S-H gel had a more advanced degree of hydration and consequently was showing a more regular distribution than in the case of Portland cement.

The Portland cement treated specimen showed less optimistic picture; microstructures with lot of voids, CH and even ettringite.

Compared to the laboratory samples, the SSI column field specimen lead to a much higher strength. The SEM investigation showed indeed lot of voids and pores in the laboratory mixed specimen. The mixing method of SSI, implemented in the field, provided on the contrary a much more uniform distribution of cement particles in the soil causing faster hardening of the cement interaction.

REFERENCES

Griffin J.L.W., Coveney P.V, Whiting A, Davey R. 1999. *Design and synthesis of macrocyclic ligands for specific interaction with crystalline ettringite and demonstration of a viable mechanism for the setting of cement*, J. Chem. Soc, Perkin Trans. 2.

Mitchell J.K. 1993. *Fundamentals of soil behavior, 2nd edition*. J. Willey & Sons

Odler I. 2000. *Special inorganic cements*. E&FN Spon, London.

Taylor H.F.W., *Cement chemistry, 2nd edition*. Univesity of Aberdeen, Thomas Telford

Van Impe W.F., Verastegui F.R.D., Menge P., Van den Broeck M. 2004. *Stabilization of dredged sludge with different types of binder*. Proceedings of the Int. Symposium on Engineering Practice and Performance of soft deposits, IS – Osaka. Japan.

Verástegui, R.D., Van Impe, W.F., Mengé, P., Van den Broeck M. 2003. *Construcción en etapas y mejoramiento de fango para el diseño de un dique sumergido*, Journal of the Spanish Society of Soil Mechanics and Geotechnical engineering. Vol. 123, pp. 53-62.

W.F. Van Impe, R.D. Verástegui F., P.O. Van Impe, J. Van Mieghem 2002. *Underwater sediments for the design of an embankment*, International navigation congress PIANC 2002. Sidney, Australia, pp. 449-460.

Van Impe, W ; Verastegui Flores, R ; Mengé, P; Van Den Broeck, M 2004. *Stabilisation of dredged sludge with different types of binder*. Proc. of the Int. Symposium on Engineering Practice and Performance of Soft Deposits / Ed. Matsui, T ; Tanaka, Y ; Mimura, M. ISBN 4-88644-812-7. p. 197-20

Van Impe, W ; Verastegui Flores, R ; Van Mieghem, J. 2004. *Staged construction and soil improvement applied in near-shore condition*, Proc. of the Int. Symposium on Engineering Practice and Performance of Soft Deposits, IS-OSAKA / Ed. Matsui, T ; Tanaka, Y ; Mimura, M. - 2004 - ISBN 4-88644-812-7. p. 549-559.

Van Impe W. F., Verástegui Flores R.D., Mengé P., Van den Broeck M. (2005) Considerations on laboratory test results of cement stabilised sludge. Submitted to the International Conference on Deep Mixing - Best Practice and Recent Advances, Deep Mixing '05, Sweden.

Van Impe, W.F., R.D. Verástegui Flores, P.O. Van Impe, P. Mengé & M. Van den Broeck 2005. *Deep mixing research results in under water conditions*, Submitted to the ICSMGE, Osaka 2005, Japan.