



Fundamental mechanism of cement in stabilizing Champlain Sea clay

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ABSTRACT

Champlain Sea clay is prominent along the St. Lawrence valley in Eastern Canada. It has an open fabric structure formed during its deposition in seawater and tends to lose its strength when disturbed. There are not many ground improvement techniques readily available to address geotechnical challenges in sensitive Champlain Sea clay. The fundamental mechanisms of cement in stabilizing Champlain Sea clay were investigated in this study using Scanning Electron Microscopy (SEM) method. Specimen were mixed with different types of cement binders under different dosages. A total of five binders were used in this study and all dosages are per cubic metre of treated soil: 1) General Use (GU) Portland cement with three dosages of 50, 100 and 200 kg/m³, 2) 50 kg/m³ of cement blended with 8% of silica fume (HSF), 3) 67 kg/m³ of cement blended with Cement Kiln Dust (CKD3:1) at a cement-to-CKD ratio of 3:1, 4) 100 kg/m³ of cement blended with CKD with a cement-to-CKD ratio of 1:1 and 5) 67 kg/m³ cement blended with slag with a cement-to-slag ratio of 3:1. A series of SEM were conducted to investigate the microstructural change and phase composition of the stabilized soil. The morphology changes and growth of reaction products such as calcium silicate hydrate (CSH) and ettringite were evident based on the SEM observations. Improvement in soil strength is due to the reduction of pore spaces resulting from the growing and binding of reaction products with soil particles. Among the five different binders, the most efficient one is found to be GU in term of strength increase based on unconfined compressive strength test results. This phenomenon becomes even more prominent with an increasing curing time.

RÉSUMÉ

L'argile de la mer de Champlain est éminent dans l'est du Canada. Il a une structure de tissu ouverte formée lors de son dépôt dans l'eau de mer et a tendance à perdre de sa résistance lorsqu'il est perturbé. Il n'existe pas beaucoup de techniques d'amélioration des sols permettant de relever les défis géotechniques de l'argile sensible de la mer de Champlain. Les mécanismes fondamentaux du ciment dans la stabilisation de l'argile de la mer Champlain à l'aide de la méthode de microscopie électronique à balayage (SEM) ont été étudiés. Les échantillons ont été mélangés avec différents liants de ciment sous différentes doses. Cinq liants au total ont été utilisés dans cette étude et tous les dosages sont calculés par mètre cube de sol: 1) Ciment Portland à usage général (GU) avec trois dosages: 50, 100 et 200 kg, 2) 50 kg de ciment Portland mélangé à 8% de fumée de silice (HSF), 3) 67 kg de poussière de four à ciment (CKD3:1) avec un rapport ciment à CKD de 3:1, 4) 100 kg de poussière de four à ciment (CKD1:1) avec un rapport ciment CKD de 1:1 et 5) 67 kg de ciment mélangé à du laitier (CS) avec un rapport ciment-laitier de 3:1. Une série de SEM ont été menées pour étudier le changement microstructural et la composition en phases du sol stabilisé. Les changements de morphologie et la croissance de produits de réaction tels que l'hydrate de silicate de calcium (CSH) et l'ettringite étaient évidents d'après les observations au SEM. L'amélioration de la résistance du sol est due à la réduction des espaces poreux résultant de la croissance et de la liaison des produits de la réaction avec les particules du sol. Parmi les cinq liants différents, le plus efficace semble être le GU. Ce phénomène devient encore plus important avec le temps de la cure.

1 INTRODUCTION

Champlain Sea clay, also known as Leda clay, spreads along the St. Lawrence valley in Ontario and Quebec in eastern Canada. In North America, the last glaciation covered most of Canada, parts of the conterminous northern United States, and part of Alaska. The largest deposits of postglacial marine clays were formed in the Champlain Sea, which occupied the St. Lawrence lowlands from the Gulf of St. Lawrence to the region around the city of Ottawa during the period approximately 8000-12000 years ago. The thickness of the clay layer is variable and exceeds 60m in some places (Gillot, 1970). It is thought that clays deposited in marine environment tend to have an open fabric structure and tend to result in a high sensitivity

in the soils (Gillot, 1970). Sensitivity is defined as the ratio of the shear strength of the undisturbed soil to the shear strength of the remolded soil and is expressed as a dimensionless number (Locat et al, 1985). Soil with such behavior are classified from quick clays to sensitive clays in geotechnical engineering, they can be converted from a relatively strong brittle material to viscous liquid (Gillott, 1970).

Champlain Sea clay has an open fabric structure and when disturbed, it tends to lose its strength. In addition, the high in-situ water content and low shear strength of Champlain Sea clay can cause excess settlements and slope stability issues (Lefebvre, 1996).

Historically, Champlain Sea clay was responsible for spectacular landslides like the Saint-Jean-Vianney in

Quebec and South Nation River in Ontario (Locat & Demers, 1988). Other engineering challenges such as excess settlement over time or stability issues of structures built on foundation Champlain Sea clay.

Many ground improvement techniques have been developed during last few decades to address the problem of stability and compressibility of soft clay foundations. Deep soil mixing (DSM) is one of the techniques which was first used in Japan in early 1970s. Recently a few studies were made at Ryerson University to investigate the feasibility of treating Champlain Sea clay with DSM. The DSM studies on Champlain Sea clay samples from two sites near Ottawa have reported an encouraging improvement in shear strength. Samples from Arnprior location treated with a cement factor of 12.5% reached an improvement ratio of 8. While the ratio of improvement reached 25 when samples from the Kanata location were treated with 10% cement (Li et al 2016).

Different types of binders may have varying degrees of soil improvement results. In addition, the fundamental reinforcing mechanism of DSM in Champlain Sea clay is missing. This paper attempts to investigate microstructural changes and strength development in cement and other binders-stabilized Champlain Sea clay from the Ottawa region. The unconfined compressive strength (UCS) was used to investigate strength development with time. Scanning electron microscope (SEM) was used to investigate the microstructure changes. The main influential factors for strength development and microstructure changes such as binder content, and curing time were analyzed.

2 SEM STUDIES OF CLAY STRUCTURES

Soil structure is an important aspect in the understanding of fundamental mechanisms of clay structures and its behaviour. Many researchers have investigated the microstructures of clay materials using SEM (Delage & Lefebvre, 1984; Vichan et al, 2013;). SEM is suited for studying clay because of its ability to provide a magnified, three dimensional (3D) view with a great depth of focus (Bohor & Hughes, 1971). SEM method has been used to investigate the nature of clay particles in the soil. Extensive studies have been conducted by Keller (1985) on kaolinite, montmorillonite and illite and presented the fabric details of the soil particles.

Lambe (1953) in his very early model of the microstructure of fine-grained soils considered clay particles to be single platelets. Since then, there have been significant advances in microstructure observation techniques, leading to a better understanding of fine-grained soils with respect to their engineering behavior as reported by Quigley, Graham, and Teller (1983).

Understanding the microstructure of the cement-treated Champlain Sea clay of Eastern Canada is of paramount importance to relate its engineering behavior with its microstructure change.

3 LABORATORY INVESTIGATION

3.1 Soil samples collection and properties

The clay samples used in this investigation were originated from foundation clay for a dam in the city of Arnprior, approximately 60 km west from the city of Ottawa, Ontario. The soil samples were Laval samples with dimensions of 200 mm in diameter and 220 mm in height and delivered to the Geotechnical laboratory at Ryerson University. The samples were mounted on sealed plywood, wrapped in cellophane paper, and coated with paraffin wax to restrict air contact, remoulding or natural water content alteration. Sealed samples were packed in a custom-made wooden box padded with material all around and under to prevent any disturbance during transportation. The samples used in this study were taken at depths of 20.6 m to 21.11 m measured from about of 9 m deep berm. More details about the physical and geotechnical properties of this Champlain Sea clay can be found in Liu et al. (2017). The main physical properties are summarized in Table 1.

Table 1: Physical properties of clay samples

Soil Type (USCS)	CH
Moisture Content (%)	64 to 74
Liquid Limit (%)	62 and 76
Plastic Limit (%)	30 and 38
Specific Gravity	2.65 to 2.70
Sensitivity	4 to 8

3.2 Sample preparation procedures

Two different sample preparation procedures were used: One was used to prepare specimens for the UCS tests and the other was used to prepare specimens for microstructure changes analysis using SEM.

3.2.1 Sample preparation for UCS

The wet mixing method was used for this study. The following procedure was adopted for sample preparation:

- 1) The slurry was prepared with a water to binder ratio of 1:1. Table 2 summarizes the dosage and binder type used in this study. The cement and water were mixed for 5 minutes until the slurry was consistent and homogeneous.
- 2) The soils samples were homogenized for 3 minutes in a stand mixer.
- 3) The required amount of slurry was added to the soil and mixed manually with a spatula for 1 min and for 9 min in the mixer. After every 2 min the mixer was stopped, and the bottom of the mixing bowl was manually scraped to homogenize the mixture.
- 4) After mixing, the cement soil mixture was compacted in three layers in a 50 mm diameter by 100 mm long reusable polyvinylchloride (PVC) mold. Each layer was tapped 25 times to remove any trapped air. The density of each specimen with the same mixing condition was monitored and kept constant.
- 5) The cylinders were then placed into a humid chamber for curing. The curing chamber had a relative humidity of

95-100 percent and a temperature of 22-25°C. The curing conditions allowed cement to fully react with soil to trigger the Pozzolanic reaction (Kitazume & Terashi 2103).
 6) After the specified curing time, samples were carefully removed from the mold by applying an air shock to the end of the mold (Liu et al. 2017).

Table 2: Binder types and dosages

Binder ID	Binder Type	Dosage (kg/m ³)
GU200	General Use cement	200
GU100	General Use cement	100
GU50	General Use cement	50
HSF50	Cement with 8% silica fume	50
CS3:1	Cement-slag with 3:1 ratio	67
CKD3:1	Cement-Cement Kiln Dust with 3:1 ratio	67
CKD1:1	Cement-Cement Kiln Dust with 1:1 ratio	100

3.2.2 Sample preparation for Scanning Electron Microscopy (SEM)

From the same mix as UCS samples, specimens for SEM tests were prepared in 12 mm diameter by 10 mm high rings. The mixture was pressed using the thumb and carefully made to a flat surface. The specimen was then kept in a sealed plastic bag submerged in water in the same curing conditions as UCS specimen. After the specific curing times, the sample was oven-dried for 24 hours Figure 1 (L). The sample was then gold coated in a vacuum evaporator Figure 1(R), to prevent a build-up of the electrons on the surfaces by conducting away static electricity. The sample was then mounted using a double stick tape and attached to the mounting stub. Two specimens can be mounted at a time, because of the small size of the samples.



Figure 1: Photographs of two gold-coated samples (L) and gold laboratory Vacuum Thin film Thermal evaporator (R).

3.3 Testing Methods

3.3.1 Unconfined Compressive Strength (UCS) Test

UCS tests were conducted in accordance with ASTM D2166 standard. The test was performed to investigate the efficiency of each binder with strength gains after 7, 14 and 28 days.

It is noted that at early age of curing (7 days and some cases after 14 days) the failure mode was conical. However, for 28 days curing most of the failure mode was planar. The photographs in Figure 2 shows typical failure modes observed during UCS testing.

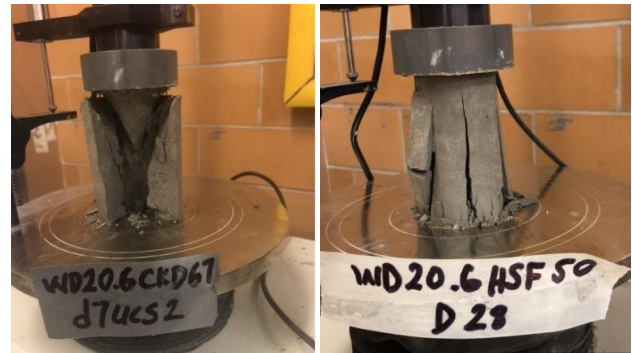


Figure 2: failure modes of CKD3:1 and HSF50 samples

3.3.2 Scanning Electron Microscopy

A JEOL JSM-6380 LV SEM with a probe current of 20 mA and 20 kV as accelerating voltage was used in this study. The 20 kV accelerating voltage was suitable for this study because it causes the least sample charging effect. The samples were dried and freed from any oils or grease before gold coating was performed in a sputter coater Figure 1(R). After coating, the specimen was mounted on a metal stub using double side tape and a metallic strip with a silver-containing glue to increase conductivity. The specimen was then entered the SEM chamber and the data were collected over the selected area of the sample surface to generate a two-dimensional image that displays spatial variations in properties including chemical characterization, texture and orientation of materials. The SEM can determine quantitatively the chemical compositions, crystalline structure, and crystal orientations.

The Energy Dispersive Spectrum (EDS) detector make use of the X-rays spectrum emitted by different element and separates them into an energy spectrum. An EDS software determine the abundance of specific elements by analyzing their energy spectrum. The energy peaks correspond to the element in the sample (Goldstein, 1981).

3.4 Test Results and Discussions

3.4.1 Unconfined compressive strength (UCS) results

Two samples were tested to ensure reliability of the results for each test condition; if the first two did not show agreement a third one would be used. It can be seen from Figure 3 that the samples' UCS and its elastic modulus are directly proportional. The higher the UCS the higher its modulus of elasticity. Untreated sample UCS peak strength reached 99.4 kPa.

Samples treated with 100 and 200 Kg/m³ (GU200) and cured for 28 days reach an average UCS of 2400 and 2844 kPa, respectively, as shown in Figure 3. It is noted that the strength increase ratio of the sample treated with 200 kg/m³ does not differ much from the sample treated with 100kg/m³.

Samples treated with 50 Kg/m³ with an improvement ratio of 8 shows significant improvement compared to other binders with similar dosages (CS3:1, CKD3:1, CKD1:1 and HSF50).

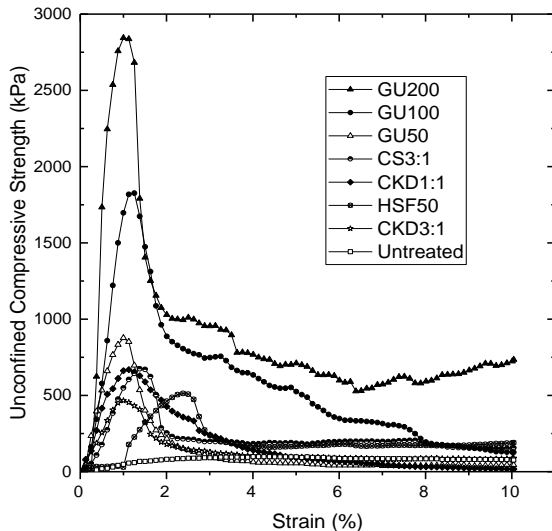


Figure 3: Stress-strain curves of clay treated with different binders cured for 28 days.

Figure 4 illustrates the development of strength of the different binders used in this study against the curing time. It was found that the GU cement is the most effective in treating Champlain Sea clay in term of strength improvement. It can be observed that the strength developed by using 50 kg/m³ Cement with 8% of silica fume results in a lower strength than the same dosage of GU cement at the 28 days of curing, however, the trend of the HSF strength is almost linearly increasing and could reach a significant strength at a longer period of curing. This may be explained by the fact that pozzolanic reactions are slow and more time is needed for the reaction to complete.

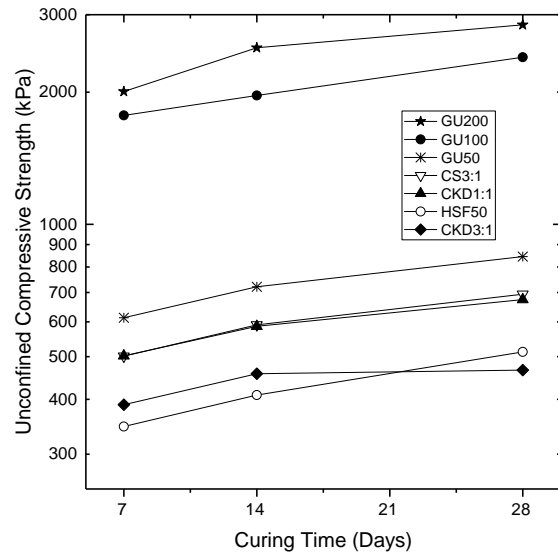


Figure 4: UCS strength of clay treated with different binders vs. curing time

The 28-day UCS strengths for CS3:1 and CKD 1:1 do not differ much, their strengths reached around 680 kPa, however the UCS strength increase in CS3:1 samples seems to be more significant after the 14 days of curing than the one from CKD1:1. CKD3:1 has the lower strength among all other binders, and exhibits a slow strength development after 14 days of curing. The slag to cement ratio seems to be not enough to allow cement to produce enough calcium hydroxide to trigger the pozzolanic reaction by the slag. Further investigation of different cement-to-slag ratio is needed to be carried out to confirm this observation.

3.5 Scanning Electron Microscopy (SEM) Results

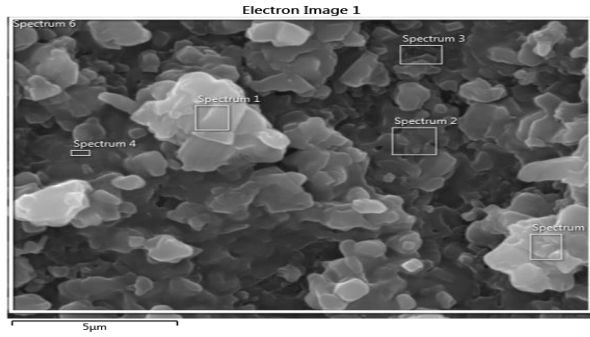
The use of SEM to investigate the changes in microstructures and growth of hydration products between different cementitious material and the soil particles has been useful.

3.5.1 Untreated samples

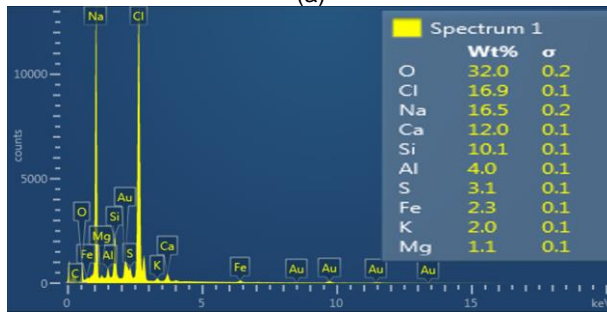
From Figure 6a and 6b, the observed irregular shape of the thin platy particles seems to be an illitic structure. Soils having this structure exhibit high compressibility and low strength (Sridharan & Jayadeva, 1982). SEM micrographs as illustrated in Figure 6- (a) (x 7000 magnification) shows the untreated clay from a depth of 21.6m with an average particle size of 1 μm. 6-(b) (x10000) shows the open fabric of clay sample from same depth. the micrograph also shows the existence of some kaolinite and silt minerals.

Some sodium chloride particles were also found within the soil as shown by the EDS analysis, as shown in Figure 5a, along with the locations of the spots for spectrum analyses. Spectrum 1 of Figure 5b shows that the salt particle in this clay is sodium chloride. Most likely, this open

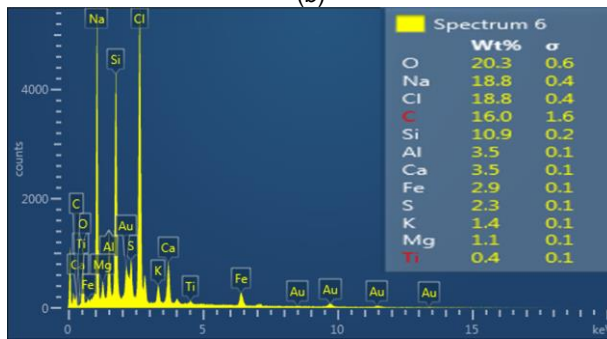
structure is due to the significant pore water quantity that allows the development of a complete double layer causing interparticle repulsion. Clay with this open type fabric is expected to be deposited in sea water. The dispersed structure of the clay sample depends greatly on the cation valence and the electrolyte concentration of the pore fluid (Mitchel and Soga, 2005)



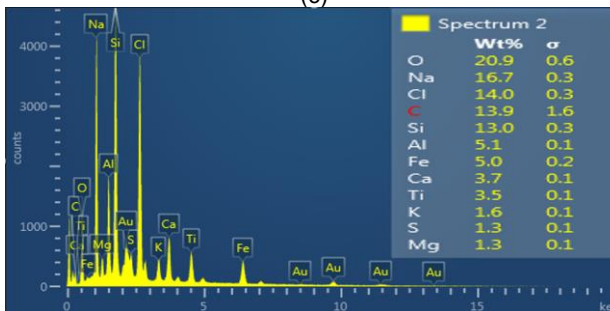
(a)



(b)



(c)



(d)

Figure 5: Photographs of EDS analysis (a) SEM micrograph, (b) spectrum1, (c) spectrum6, (d) spectrum2

Spectrum 2 in Figure 5d shows the spot element composition of the clay and their weight percentages. And Spectrum 6 in Figure 5c shows the average element composition for the entire surface of the micrograph.

3.5.2 Treated samples

Significant changes in soil structure could be observed by SEM micrographs of treated samples as illustrated in Figures 6c to 6i.

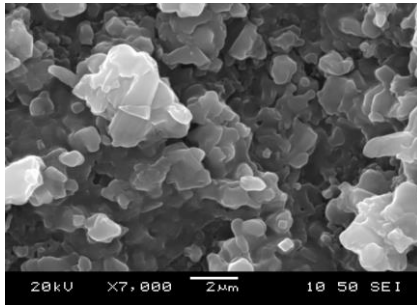
Figure 6c illustrates a sample of clay treated by GU50 cured for 7 days. It can be seen that most of the voids are filled with the hydration products. Figures 6d and 6e represent samples treated by GU100 and GU200 cured also for 7 days, the amount of hydration product in these sample seems to be higher than the one in GU50, the results are also confirmed by the high compressive strength associated with a high cement dosage.

Figures 6f and 4g illustrate the microstructures of the clay samples treated by CKD3:1 and CKD1:1. The observation of their micrographs does not show a big difference. However, a general improvement in the void structure is clear due to the growth of the hydration product. Figure 6h shows the significant changes in mineralogical phases treated by CS3:1 cured for 7 days.

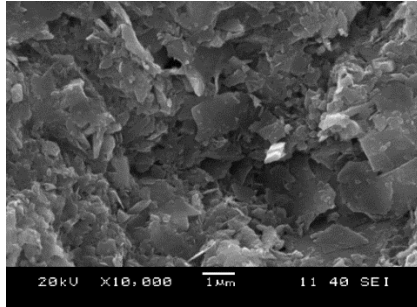
The SEM micrograph in Figure 6i illustrates the early stage of calcium silicate hydrate (CSH) development in a sample treated by HSF50 cured for 7 days. The voids are not significantly filled compared to the mixtures with other binders, which may explain the low compressive strength of HSF50 samples.

Generally, it can be seen the significant changes in the morphology of the treated soil. It also could be observed that the platy structure of the soil sample is coated by CSH and ettringite. It's hard to conclude a correlation between UCS and the observed morphology change, because the selection of the spot area observed under SEM can greatly affect the image produced. However, a general trend of hydration product growth is observed.

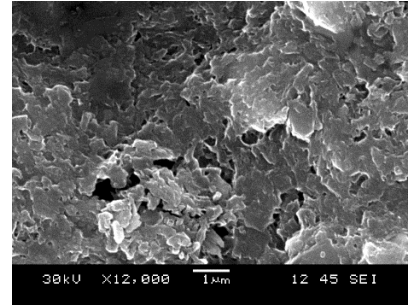
As curing time increased to 28 days, significant changes in the microstructures of the clay samples are observed and the amount of CSH phases increased and developed as a fabric structure as shown in all micrographs in Figure 7. On the other hand, some needle-like ettringite could be seen in Figures 7b and 7c. Micrographs for GU100* and GU200* show the abundance of the hydration products associated with the high cement factor. It is assumed that filling in the void network by the hydration products results in a denser and stronger structure, which leads to an improvement in the compressive strength and also improvement in other engineering properties, such as permeability and compressibility. In summary, the amount of CSH and ettringite is significantly greater in 28-day cured samples than in 7-day cured samples, which lead to cement-soil improvement.



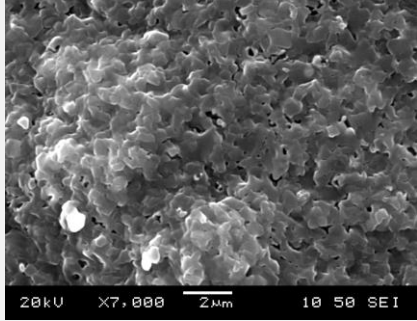
(a) untreated



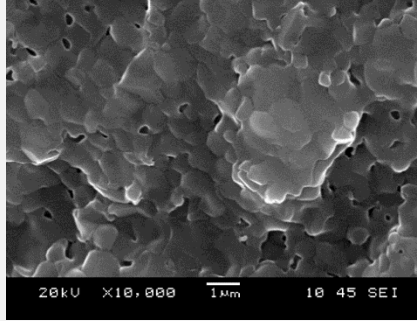
(b) untreated



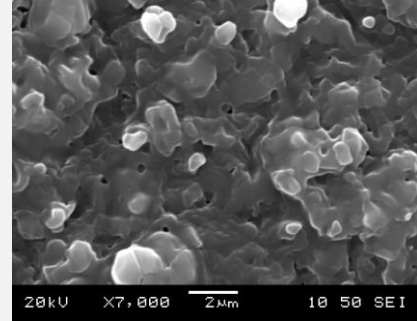
(c) GU50



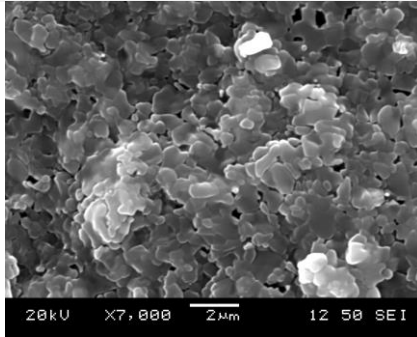
(d) GU100



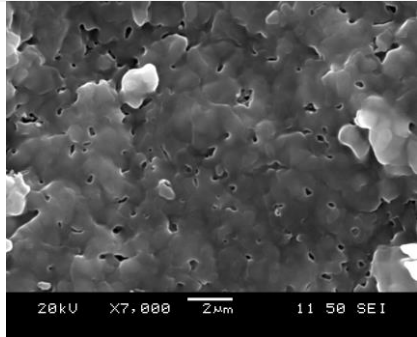
(e) GU200



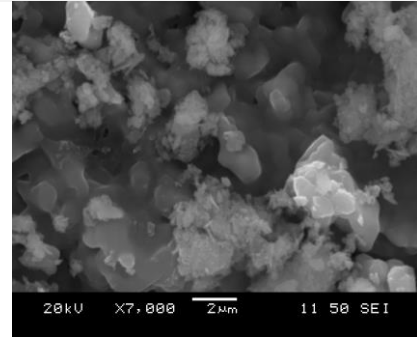
(f) CKD3:1



(g) CKD1:1



(h) CS3:1



(i) HSF50

Figure 6: SEM micrographs of untreated and treated clay cured for 7 days

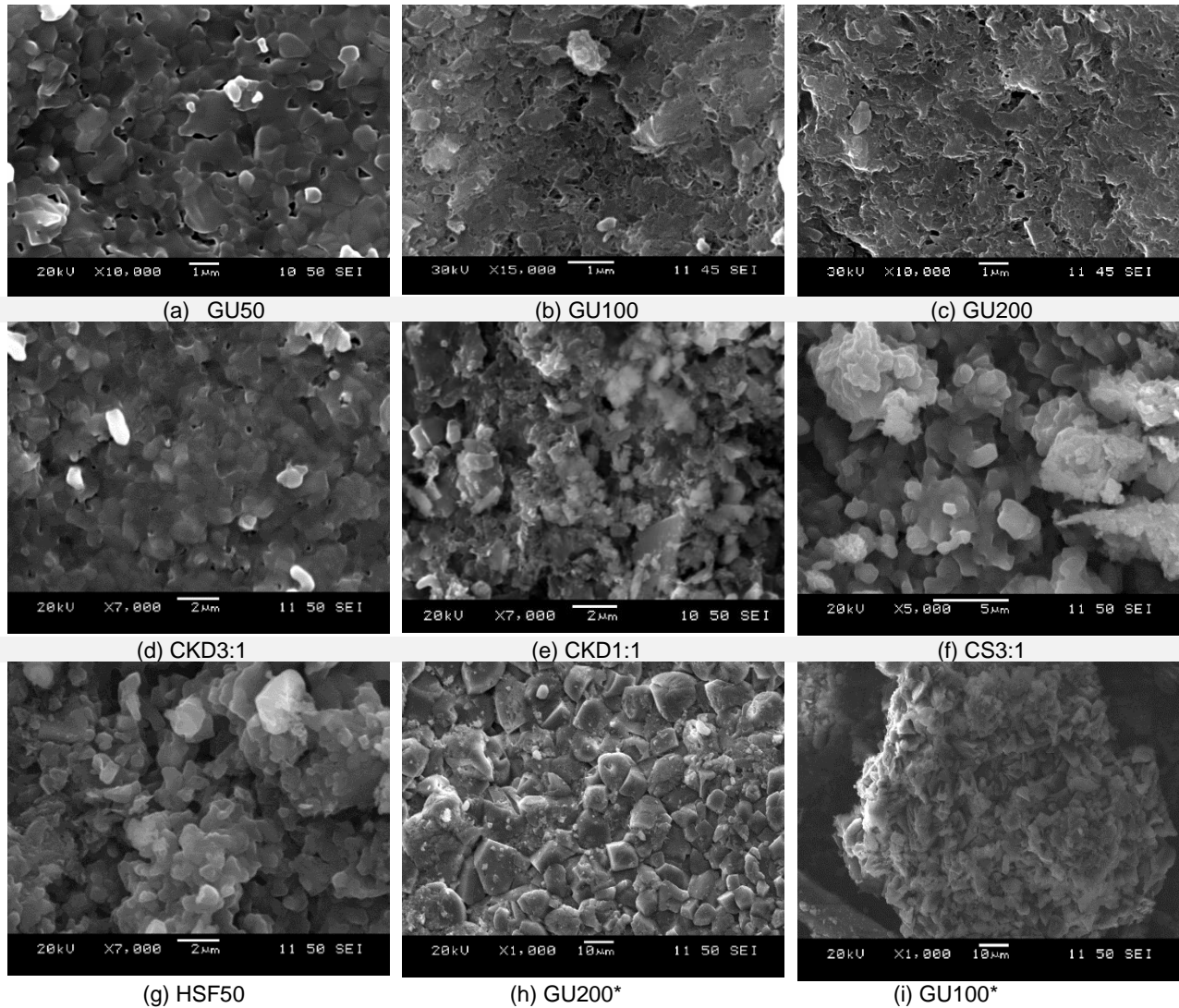


Figure 7: SEM micrographs of treated clay cured for 28 days

4 CONCLUSIONS

The observations of microstructures for both treated and untreated Champlain Sea clay are subjective due to the selection of the tested spots. However, the combination of scanning electron microscopy (SEM) and unconfined compressive strength (UCS) can allow us to verify and interpret certain hypotheses. The principal conclusions of this study on cement-treated Champlain Sea clay are summarized as follow:

- 1)The Champlain Sea clay used in this study exhibits an open fabric structure which explains its low shear strength and high compressibility
- 2)Based on the tests conducted in this study, General Use (type GU) cement is the most effective in treating Champlain Sea clay compared to other binders.
- 3)A significant reduction of void is observed in the treated samples due to the growth of hydration product such as

calcium silicate hydrate and ettringite, which contributes to the strength increase in treated samples.

- 4)More studies are required to confirm the findings with samples for longer curing time and Champlain Sea clay samples from other regions.

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