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BEHAVIOR OF BIOCEMENTED SANDS UNDER CYCLIC LOADING

by
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ABSTRACT
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In the past ten years, intensive research efforts were undertaken to study a newly emerging soil improvement technique that uses calcite-inducing reactions to produce natural cementation in soils. Given the fact that current ground improvement techniques are energy consuming, it is imperative that research in the soil improvement field be directed towards more sustainable eco-friendly techniques. Microbial induced calcite precipitation is a sustainable technique that allows for solid sandstone-like material to be produced from naturally loose sand. The technique uses bacterial species to produce urease enzymes, which catalyze the chemical reaction and induce calcite precipitation. Another way for inducing calcite precipitation is to directly use the urease enzyme, leading to enzyme-induced calcite precipitation. Researchers have studied the behavior of biocemented sand using conventional triaxial tests and compressive strength tests. However, the cyclic behavior of these samples is not yet significantly explored and/or understood.

Thus, the objective of this study was to investigate the behavior of biocemented sand subjected to cyclic loading. The study focused on: (1) Measuring the improvement obtained on sand samples due to calcite precipitation by using “continuous” shear velocity measurements from embedded bender elements. (2) Studying the effect of increasing the calcite precipitation on the sand resistance to cyclic loading. The sand type and properties, percentage of cementation, curing time, and vertical confinement were also investigated.

The study concluded with a significant improvement in treated samples which was noticed through the increase in the shear wave velocity, compared to the non-treated samples, and depending on the quantity of cementation solution. In addition, the samples showed resistance to cyclic loading as we increase the cementation, which implies resistance to liquefaction. Finally, the improvement measured using the cone penetration test was in compliance with the shear wave velocity readings.

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CHAPTER 1

INTRODUCTION AND BACKGROUND REVIEW

In the past decades, a wide range of ground improvement techniques have been introduced to enhance the mechanical properties of “weak” and/or marginal soil formations. Most of these techniques require mechanical power or the introduction of man-made binders that consume substantial energy in the material production and installation (Arab, 2019). These include long-established methods such as deep dynamic compaction, stone columns, vibro-compaction, and chemical (cement or resin) grouting.

Recently, due to the increased demand for sustainable solutions for ground improvement, efforts have been done to find new techniques that can replace the available energy-heavy and environmentally questionable methods. Among them, those involving cement grouting and injection are of particular concern. Calcite precipitation is one of the most effective newly implemented techniques that has been intensively investigated as a sustainable alternative for soil improvement and natural soil cementation.

Several methods can be used to induce calcite precipitation, including urea hydrolysis, microbial denitrification, and sulfate reduction (DeJong et al., 2013). Urea hydrolysis is the most advanced mechanism to induce calcite precipitation in terms of its development, and the most common due to its simplicity (Van Paassen, 2009). This method takes advantages of natural bioactivities, technically termed as microbial induced calcite precipitation (MICP), to produce calcite in a soil matrix (Lee, 2014). The chemical reactions governing the process are given by equation 1.

$$CO\,(NH_2)_2 + CaCl_2 + 2H_2O \rightarrow CaCO_3 + 2NH_4Cl \qquad (1)$$

In this reaction, a urease enzyme, produced by microorganisms, or introduced as a free enzyme, catalyzes the hydrolysis of urea which produces carbonate ions in an aqueous solution. In the presence of a calcium source, generally from calcium chloride ($CaCl_2$), calcium carbonate ($CaCO_3$) starts to precipitate. The precipitation of calcite induced by the above reaction will fill the pores and/or bind the sand particles at their contacts, thus creating a “solid” biocemented sand sample (Biosandstone). Figure (1) presents a successful transformation of natural sand into biosandstone in the laboratory (Mujah et al., 2017).

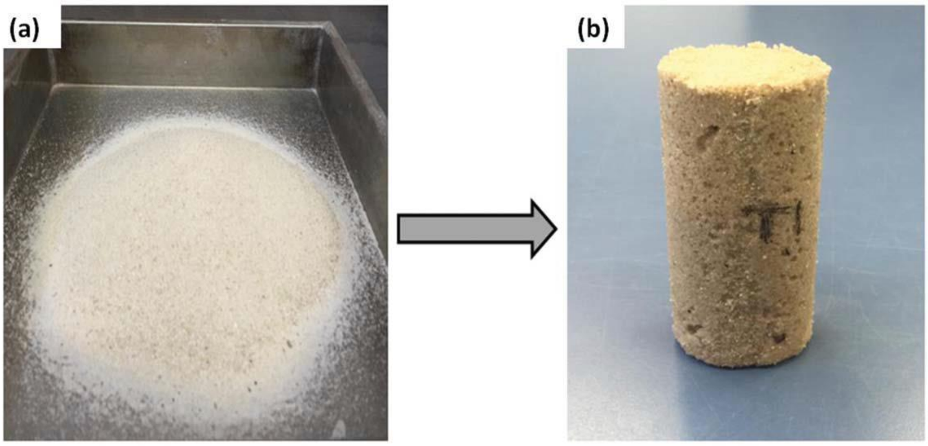

Figure 1. Sand metamorphosis: (a) natural sand and (b) biocemented sand (biosandstone)

EICP (Enzyme Induced Calcite Precipitation) defers from MICP by using free urease enzyme rather than inducing the secretion of urease enzyme from microorganisms. The free urease enzyme can be extracted from plants and is known as plant-derived urease. The use of plant-derived urease to induce urea hydrolysis (EICP) presents several advantages when compared to MICP since the size of the enzyme is on the order of 12 nm, which favors the permeation of the bio-grout in finer soils and/or into smaller pore spaces/necks. Secondly, there is no concern about supplying the microorganisms with nutrients, and about oxygen availability when applying such treatment at significant depths. Finally, EICP bypasses some concerns that lay in the fact that microorganisms may cause, in the long-term, further blocking in the pores and leave the ground heavily contaminated with bacterial cells once the biocementation reactions are complete. However, MICP has one possible advantage over ECIP and that is that it provides nucleation sites for the precipitation of calcite (Kavazanjian & Hamdan, 2015).

Given the above, we have selected for our study to adopt an EICP technique based on a readily available and easy to produce free urease enzyme extracted from the jack-bean plant (Canavalia ensiformis).

In the context of ground improvement for seismic hazard mitigation, the EICP ground technique was established to be particularly suitable for the mitigation of soil liquefaction, which is a phenomenon associated with saturated mostly fine sands under earthquake loading. Liquefaction refers to a phenomenon where saturated or unconsolidated soils composed of independent granules lose their shear strength upon the application of repeated stress or shaking by earthquake causing, at the extreme, the soil to act like a liquid (Kramer 1996). Infrastructure built on such soils are thus more prone to damage, partially or completely, when earthquakes occur, and it is very important to find safety measures or techniques to prevent liquefaction to protect key civil structures (Towhata, 2008 and Unjoh et al., 2012). Throughout history, soil liquefaction has caused significant damage, injury and fatalities, and spectacular failures of key systems (roads, dams, bridges, water and sewage treatment facilities in addition to residential buildings). One way of mitigating for the potential of liquefaction due to earthquakes is the “liquefaction-resistance” of soils. This could be increased by densifying loose deposits or by induced cementation/strengthening at the contacts within liquefiable soil layers using EICP (Kumari et al., 2019). As such, understanding the effect of biocementation on the cyclic behavior of sands is needed to evaluate the efficiency of this technique in liquefaction mitigation.

In the context of biocementation, it is important to highlight that it is a newly emerging soil improvement technique that has been the subject of interest for many researchers in the past few years. Biocementation is found in nature as bio-deposition of sand over a long period of time. These observations in nature inspired geotechnical engineers to further explore, adapt, and implement this phenomenon in geotechnical applications (Mujah et al., 2016).

The recent and extensive researcher efforts produced significant progress in our understanding of the occurrence and conditions necessary for the biocementation process. For instance, several experiments were conducted by Rohy et al., 2019 using a “mix and compact” method, where a graded silica sand was mixed with the cementation solution then placed/compacted into the mold. The cylindrical mold had dimensions of D=50mm and H=100mm. The ratio of urea to calcium chloride was fixed at 1:0.67. Three different concentrations were studied in their trials: 1M, 2M and 3M. In addition, the researchers varied the urease (activity = 1500 U/g) and non-fat dry milk (used as an organic stabilizer) quantities proportionally. The table below presents the different quantities used in the preparation of the cementation solutions.

EICP solution	Urea (M)	Urea : $CaCl_2$	Enzyme (g/l)	Organic Stabilizer(g/l)
1M	1	1:0.67	3	4
2M	2	1:0.67	6	8
3M	3	1:0.67	9	12

Table 1. Cementation Solution concentrations as presented by Rohy et al. 2019

The samples were left to cure for 3, 7, and 14 days at room temperature. The compressive strength and the quantity of precipitation were the main objective of their study. In order to quantify these targets, they used UCS to determine the strength, and SEM and acid digestion to determine the carbonate content.

Their results showed that at low concentrations (1M, 2M), the strength gained was lost in a brittle fashion right after the peak, while some ductility was achieved at treating solutions of concentrations of 3M. The highest strength after 3 days of curing was obtained for the 3M concentration (504 kPa) (1M = 219 kPa – Brittle, 2M = 314 kPa – Brittle, 3M = 504 kPa – Ductile).

The highest UCS value was reached with 3M cementation after 14 days of curing (3000 kPa), as compared to 504 kPa after only 3 days of curing. This demonstrated the importance of curing time on the strength development. Moreover, SEM imaging was used to show/qualitatively the concentration of precipitation at the contacts between the soil particles.

The use of dry milk as an organic stabilizer was investigated by Almajed et al., 2019. Almajed and co-workers studied the effect of the dry milk additive on the morphology and efficiency of calcite precipitation. The working hypothesis assumed that adding dry milk will provide nucleation cite for the precipitation and slowing the precipitation rate. The urea to calcium chloride ratio of 1:0.67 with 3g/L urease and 4g/L dry milk was used. The samples were prepared at relative density 76% using Ottawa 20/30. The method which was followed in preparing the samples was “mix and compact”. Acid digestion was used to determine the quantity of deposited calcite cementation. UCS, SEM, XRD and EDX were used in the analysis and verification of the hypothesis.

The results of this set of experiments showed that adding non-fat dry milk yielded higher compressive strengths (1.6 to 1.8 MPa) when compared to the conventional solution with no stabilizing agent (Urea, calcium chloride and urease). The SEM imaging revealed that the size of the calcium carbonate precipitate when using non-fat milk was larger than those formed using the conventional solution. This study proved that relatively high strength can be achieved at low carbonate content with the right pattern and morphology of the precipitate which was favored in presence of non-dry milk.

Another recent study (Almajed et al., 2019) used free urease enzyme of reported activity of 1500 U/g and different concentrations of the cementation solution and investigated their effect on the strength of the biocemented sand. The different concentrations followed the ratio 1:0.67 based on the recommendations of Almajed et al., 2018. The table below show the concentrations used:

Urea (M)	**Calcium Chloride (M)**	**Urea : $CaCl_2$**	**Enzyme (g/l)**	**Organic Stabilizer (g/l)**
0.5	0.335	1:0.67	1.5	4
1	0.67	1:0.67	3	4
2	1.34	1:0.67	6	4

Table 2. Cementation Solution concentrations as presented by Almajed et al., 2019

The samples prepared were 50.9 mm in diameter and 102 mm in height. Samples were left to cure in moist conditions for 7 days and then left to dry at room temperature for 7 days before conducting the UCS test. SEM was used to visualize the particle size and shape and to detect the presence of calcite bonds between the particles. EDX was used to map the chemical distribution of the EICP reaction.

Ottawa sand was found to yield the highest strength since it has rounded shape particles which favors the precipitation of calcite at the contact. It was concluded that the best cementation concentration is 1M urea, 0.67M $CaCl_2$. The results showed that as we increase the cementation solution, higher compressive strengths can be achieved.

The previous studies explored the quantities and strength of the biocemented sand samples by using UCS and acid digestion. Shear wave velocity measurement can be as if not more helpful to non-destructively assess the gradual degree of $CaCl_2$ deposition with time. It may also be used to quantify/validate the amount of precipitation (Qabany et al., 2011). Qabany and his co-workers conducted several tests and determined a relationship between S-wave and calcite precipitation. The samples prepared were D=50 mm and H=50 mm. The setup was equipped with bender elements of parallel type which were placed at the sides of the sample. A linear correlation was found between the shear wave velocity and the quantity of calcite precipitation.

In this study, we aim to investigate the behavior of biocemented sand samples under cyclic loading by using a modified oedometer setup. We will use bender elements to measure wave propagation through the sample as the chemical reactions get under way and rely on these non-destructive measurements to analyze and quantify the calcite content and the development of calcite in the pore spaces of the sample with time.

CHAPTER 2

MATERIALS AND METHODS

2.1. Materials Used

In order to better understand the behavior of biocemeneted sands under cyclic loading, a special setup is built which allows us to subject the sample to cyclic and static loading. This setup is equipped with bender elements to measure Shear wave velocity through the sample. The samples prepared contain Ottawa sand injected with cementation solution in specific quantities.

2.1.1. Ottawa Sand

HM-108 Graded Standard Sand is graded between No. 30 (600µm) and No. 100 (150µm) sieves. The sand meets requirements for ASTM C109 and C778, as well as AASHTO T 106. The naturally rounded silica sand particles of nearly pure quartz are mined from the Ottawa, Illinois area. This sand will be used dry and has a minimum density of 1450 kg/m^3 with a void ratio of 0.83, and a maximum density of 1750 kg/m^3 with a void ratio of 0.51, and with specific gravity of 2.65. Figure (4) shows the grain size distribution curve obtained from the sieve analyses conducted on the sample. These results indicate that the soil is poorly graded. The pH of the Ottawa sand used in this study was determined following ASTM D4972-19 and was found to be equal to 8.1

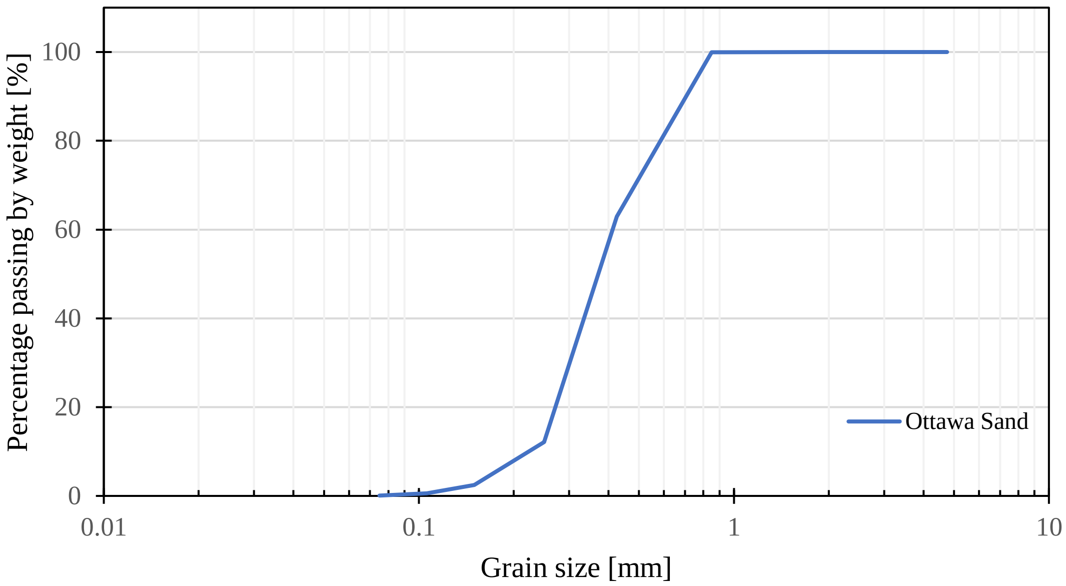

Figure 2. Grain size distribution of the Ottawa Sand

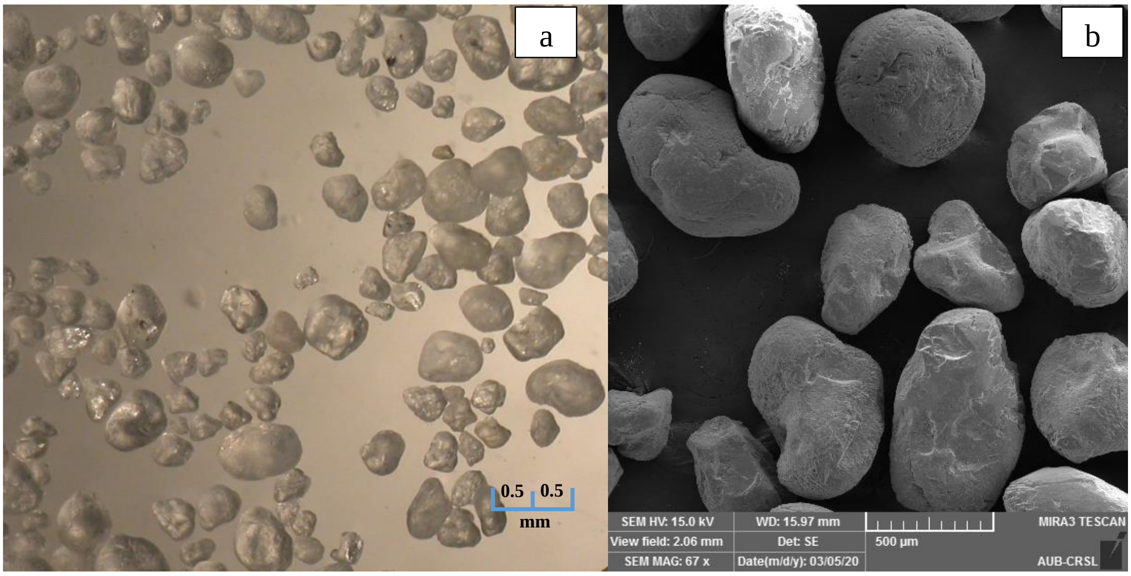

Figure 3. The shape and size of Ottawa sand, (a) under the microscope and (b) using SEM

2.1.2. Cementation Solution

The cementation solution will be prepared in the Environmental Engineering Labs at AUB. As mentioned before, the process of urea hydrolysis will be induced using free urease enzyme extracted from jack-bean plants (Canavalia ensiformis). The free urease enzyme used in this study has a reported activity of 1 U/mg at pH 8.0 and 25°C (Sigma Aldrich). The concentration of the cementation solution to be followed in this study is based on the recommendation of Almajed et al. (2019). The Urea: CaCl2 ratio was maintained at 1:0.67, 3g/L of urease enzyme and 4g/L of protein stabilizer (non-fat dry milk) were used.

The solution is formed by preparing 3 mixtures. The first mixture contains urea and water, the second mixture contains $CaCl_2$ and water, and the last mixture contains urease enzyme, milk stabilizer and water. All mixtures are stirred at 25°C until the solutes dissolve. Then the enzyme with dry milk solution is added to the urea solution. The last step before injection is adding the urea solution to the $CaCl_2$ solution. The injection is done using flow-controlled pump from the top of the sample. Multiple injections are done to attain more cementation in the sand sample.

2.2. Methods

The samples are prepared by the addition of Ottawa sand to the tall oedometer setup by means of pluviation technique. The relative density of the sand samples prepared is directed to 55% which is considered as medium dense. The samples are then introduced to the oedometer device and vertical load is applied. The samples are loaded with a vertical load of 25 kPa, then the cementation solution is injected from the top by means of flow-controlled pump. Shear-wave velocity readings are completed, after the injection, at different time intervals to monitor the development of calcite in the samples.

After 24 hours, which is the curing time, the samples are subjected to cycles of 100 kPa to assess its behavior to repeated loading. The samples are loaded gradually to reach 100kPa vertical load, and then preform several cycles before returning gradually to the 25kPa vertical load. Shear-wave velocity readings are done after each cycle, and at the end after reaching the initial vertical load. Other samples are prepared separately using the same procedure described before to conduct the cone penetration tests. Finally, the SEM imaging and EDX analysis are conducted on portion of the samples to view the structure of the calcite bonds and size at different concentrations.

2.2.1. Shear-wave Velocity Measurements

The use of bender elements is an attractive option to measure the shear wave velocity due to its non-destructive nature. The bender elements can also give the value for the small-strain shear modulus G_{max}. The small-strain shear modulus G_{max} provides valuable soil information, which is relevant to a wide range of engineering tasks including the design of foundations subjected to dynamic loading, process monitoring, liquefaction assessment, and soil improvement control (Lee and Santamarina, 2005). Results from bender elements will yield information about the stiffness, relative density, and state of the untreated sand samples. For bio-cemented samples, results from bender element tests will allow for quantifying the effects of cementation on the strength and stiffness of the improved specimens as they occur over time.

Small-scale laboratory tests were conducted on sand samples to improve our understanding of bio-cementation and its effects on enhancing soil strength properties. The proposed experimental setup was designed as a tall oedometer that allows for instrumenting the specimen with bender elements fitted at the top and base caps. The designed and built modified oedometer has the following dimensions: a height H=70 mm and a diameter ID=70 mm (Figure 2). The bender element is composed of a thin layer of piezoelectric ceramics connected by thin conductive metal plates (Saneiyan, S. 2019). Parallel bender elements are adopted for this setup; the negative terminal is connected to the outer ceramic layers and the positive terminal is connected to the inner metal plate. The parallel connection yields to highest signal to noise ratio of the shear-wave signal (Lee and Santamarina, 2005).

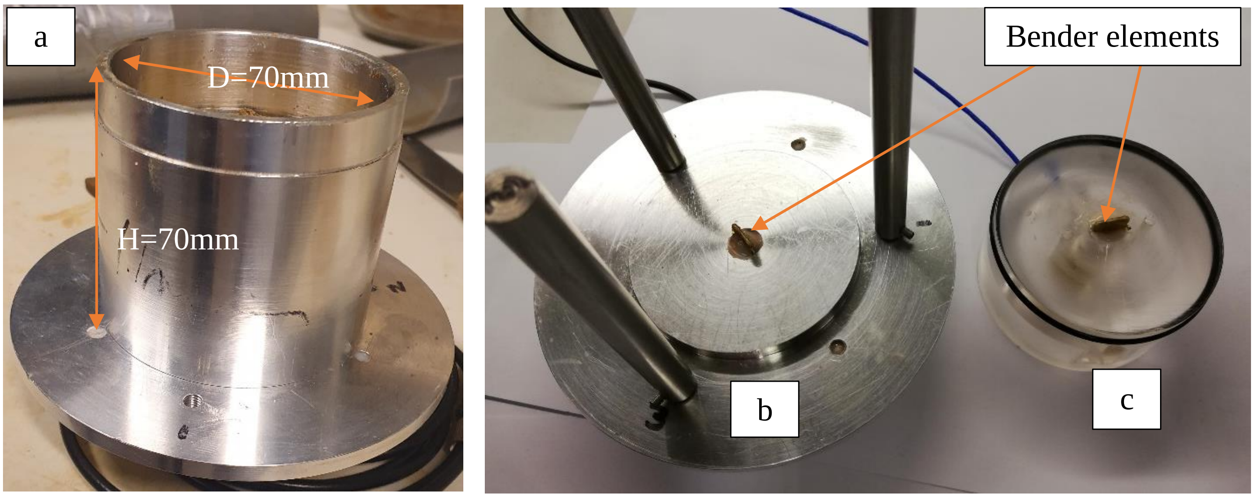

Figure 4. Bender element setup, (a) tall oedometer, (b) base cap contains the excitation and (c) top cap contains the receiver

In bender element testing, the shear wave velocity is used as an indicator of the degree of bio-cementation as it requires a medium with shear stiffness to propagate, and thus, changes in velocity would reflect the change in the specimen's shear stiffness (Qabany et al., 2011). The shear-wave velocity is calculated by having the distance between the two bender elements (d = height of sample – 2×bender element length) and measuring the time (t) required for the arrival of the first peak of the signal propagated through the sand sample by means of an oscilloscope, equation 2.

Shear wave velocity: $Vs = \frac{d}{t}$ (2)

The tall oedometer setup was specifically designed to fit in the standard oedometer device with few modifications. Extension arms are made for the device to allow for the tall oedometer to be placed. The setup is equipped with an LVDT to determine the change in the height of the specimen when the confinement load is added (Figure 3).

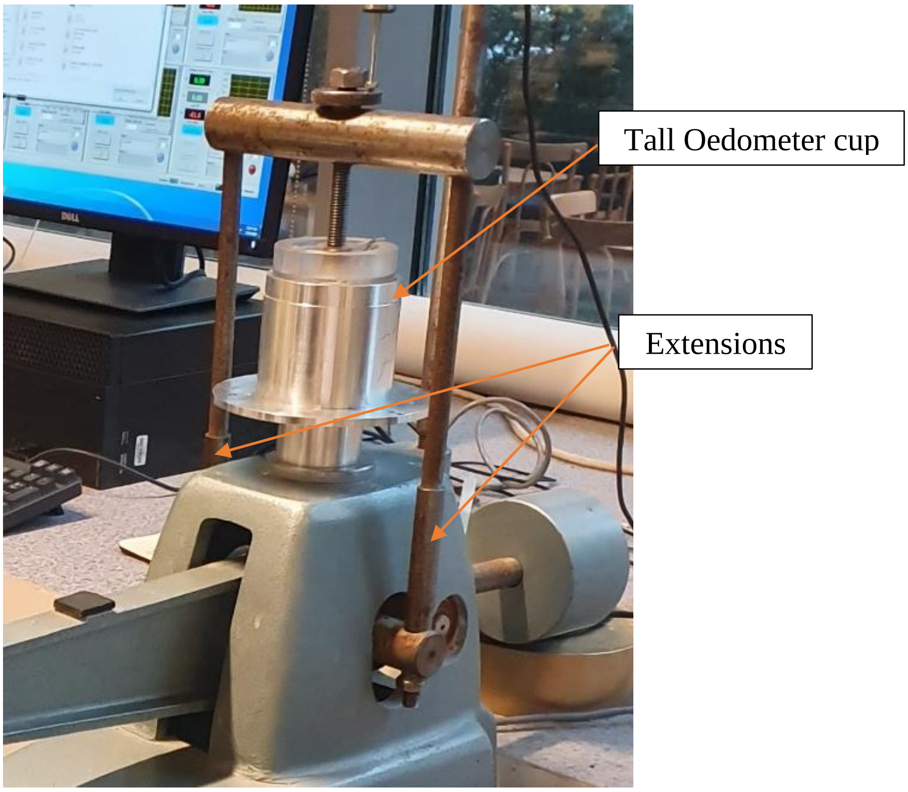

Figure 5. Modified oedometer device

2.2.2. Miniature Cone Penetrometer

It is recommended to perform direct testing alongside with geophysical monitoring, which will help for better understanding of the bonding process and signal interpretation. The use of cone penetration tests is a significant tool in geotechnical engineering to measure the resistance of the soil and correlate with soil properties and behavior (Honardar, S. 2019). The miniature cone penetrometer was designed using a linear actuator, a load cell, and a 6.5 mm diameter cone prob. The whole setup is fixed on an adjustable frame (Figure 6). The speed of the actuator while penetrating the sample is 3mm/s. The readings from the load cell and the displacement of the actuator are done using LabVIEW.

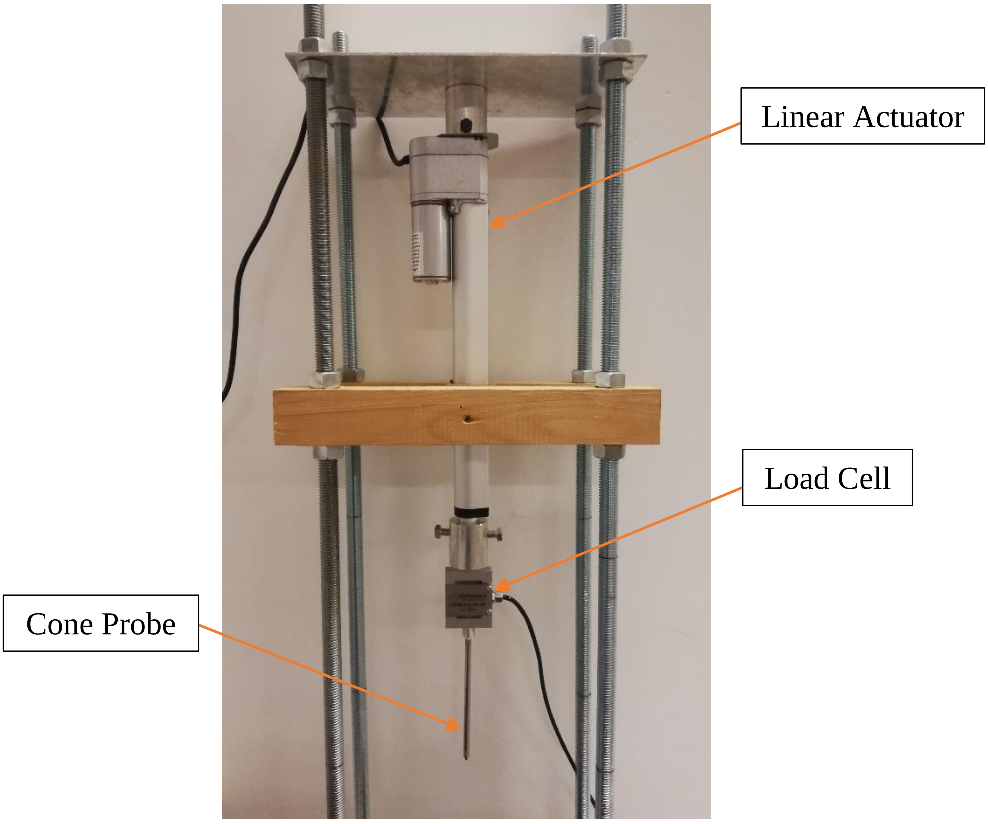

Figure 6. Miniature Cone Penetrometer Setup

2.2.3. SEM Imaging and EDX Analysis

In addition to using the bender elements, scanning electron microscope (SEM) and energy dispersive x-ray (EDX) analysis are conducted to produce and visualize the bonds between the sand particles and the calcium carbonate. SEM will help identify the size and shape of the calcite precipitation on the sand particles, and this provide information about the type of bonds between the sand particles. EDX, on the other hand, will identify the elemental decomposition of the material. We can then verify that the bonds between the sand particles is calcite.

CHAPTER 3

RESULTS

Determining shear wave velocity was done by measuring the time of arrival of the signal to the receiver bender element in the top cap, by means of an oscilloscope. The signal is sent by the bender element in the bottom cap by means of a low frequency generator (LFG), set at 10V and produces a square signal with frequency = 20Hz. This input is followed throughout the whole testing. The signal recorded on the oscilloscope is given in figure (7).

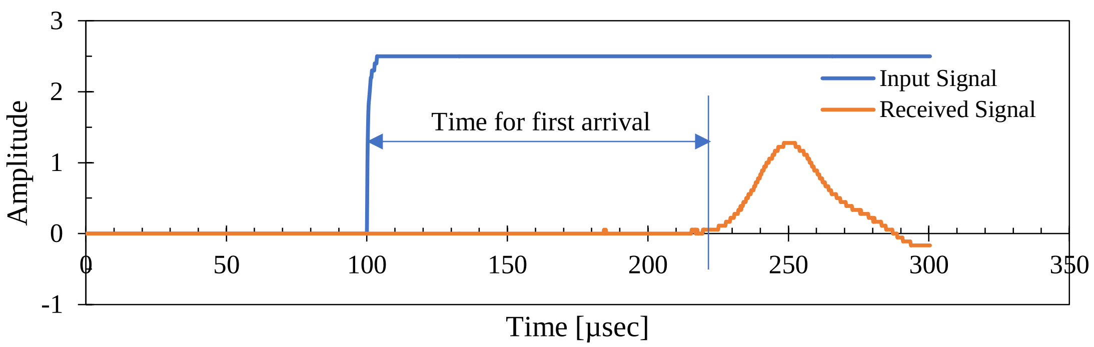

Figure 7. Signal recorded by the oscilloscope showing the input and received signals

The recorded signal on the oscilloscope shows the input signal (blue) and the received signal (orange) with a time delay between the input and received signals. This time represents the time needed for the signal to travel through the sand sample. The time is recorded from the instant of applying the input signal to the beginning of the first arrival of the received signal, as described by Lee and Santamarina, 2005. They mentioned that the first arrival in the received signal is the representation of the shear wave velocity. It is worth noting that the scale for both signals is modified for better presentation, since the input signal is in voltages and the received is in the range of 500µV.

3.1. Non-treated Samples

The tests were first performed on non-treated samples to compare and assess the improvement happening in the sand due to biocementation. A sample of 357g of Ottawa sand was prepared in the tall oedometer (55% relative density), and deionized water is injected in the sand to replicate the same conditions as when the sample is injected with the cementation solution. The readings from the bender elements are recorded and presented in table (3). The values of Vs are not varying with time which indicates that no reaction or improvement is taking place in the sample after adding deionized water.

Time of Signal Arrival (sec)		S-wave Velocity (m/s)		Time of curing (hr.)
t_1	0.0001132	Vs_1	408	0
t_2	0.0001118	Vs_2	414	1
t_3	0.0001088	Vs_3	428	24
t_4	0.0001104	Vs_4	421	48

Table 3. Shear wave velocity measurements on non-treated Ottawa sand for 24 hr.

The next step was to perform cyclic loading on the sample. First, gradual increase in the vertical load to reach 100kPa takes place, then cycles of 100kPa are applied. Vs is recorded after applying the loads to reach 100kPa and at the end of each cycle, and are presented in figure (8).

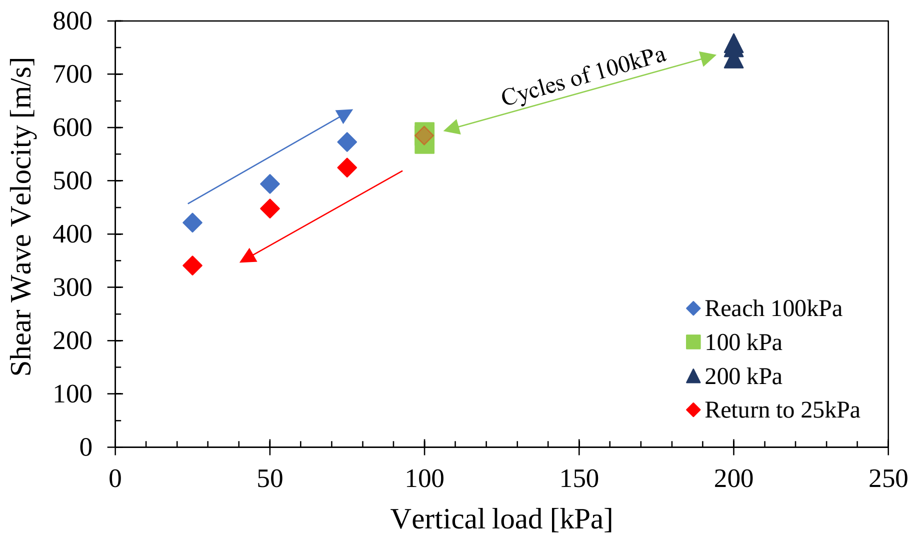

Figure 8. Variations of Vs while varying the vertical load on non-treated Ottawa sand

Vs increased from 420m/s to 586m/s when the vertical load increased from 25kPa to 100kPa. When reaching 100kPa vertical load, cycles of 100kPa are performed by increasing the load 100kPa to reach 200kPa and then return to 100kPa. The graph shows that as the load increases from 100 to 200kPa, Vs increased from 590m/s to 740m/s. The increase in Vs is mainly due to the confinement of the sand particles, better contacts; hence, the emitted signal by the bender elements is transmitted more efficiently through the soil mass.

After performing the cycles on the untreated sand sample, the load was decreased gradually from 100kPa to 25kPa, which is the initial vertical load. It is noticed that Vs returned to 340m/s, which is lower than the initial value (420m/s), having a slope parallel to that of the increasing load.

3.2. Treated Samples with EICP

Mixing the cementation solutions and injecting them in the sand sample triggers the calcite precipitation. According to the quantities prepared, the reaction time is five hours where calcium carbonate formation will stop and crystallization will take place, producing the more stable calcite product. Consequently, the signal readings from the bender elements are done at every one-hour interval. The arrival time of the signal decreases significantly as the reaction time passes; hence, the shear-wave velocity increases.

The target expected precipitation of calcium carbonate varied between 0.5, 1.0 and 1.5% by mass of the sand sample. The target calcium carbonate precipitation is reached by increasing the quantity of solutes (Urea and calcium chloride) while maintaining the ratio Urea:$CaCl_2$ at 1:0.67.

The injection of the cementation solution is done using a flow-controlled pump, and the solution is cooled by using cold water. The use of cold water will delay urea hydrolysis reaction, which ensures that the reaction will start after all the solution is in the sand.

The results from the 3 tests treated with biocementation at different precipitation quantity are presented in table 5 to table 7. The values of Vs increase with time, indicating the formation of calcite in the sand voids, filling the pores and densifying the sample. It is noticed that Vs reaches constant values after 24hr. when the reaction is completed (all the reactants reacted).

The different calcite precipitation quantities yielded proportional Vs values. As the precipitation quantity increases, higher Vs values are reached. To better compare the different cementation quantities tested, figure (9) shows the three tests complied together. The increase in Vs at 0.5% expected cementation precipitation has a mild slope compared to 1.5% which has a steep slope.

0.5% Expected Calcium Carbonate Precipitation				
Time of Signal Arrival (sec)		**S-wave Velocity (m/s)**		**Time of curing (hr.)**
t_1	0.0001256	Vs_1	458	0
t_2	0.0000886	Vs_2	649	1
t_3	0.0000736	Vs_3	781	2
t_4	0.0000666	Vs_4	863	3
t_5	0.0000462	Vs_5	1245	13
t_6	0.0000442	Vs_6	1301	22

Table 4. S-wave velocity measurements on treated Ottawa sand with 0.5% expected precipitation for 24 hr.

1.0% Expected Calcium Carbonate Precipitation				
Time of Signal Arrival (sec)		S-wave Velocity (m/s)		Time of curing (hr.)
t_1	0.0001214	Vs_1	474	0
t_2	0.0000742	Vs_2	775	1.1
t_3	0.0000646	Vs_3	890	2.5
t_4	0.0000602	Vs_4	955	3.1
t_5	0.0000562	Vs_5	1023	4.1
t_6	0.0000542	Vs_6	1061	4.9
t_7	0.000039	Vs_7	1474	24

Table 5. S-wave velocity measurements on treated Ottawa sand with 1.0% expected precipitation for 24 hr.

1.5% Expected Calcium Carbonate Precipitation				
Time of Signal Arrival (sec)		S-wave Velocity (m/s)		Time of curing (hr.)
t_1	0.0001166	Vs_1	493	0
t_2	0.0000602	Vs_2	955	1
t_3	0.0000522	Vs_3	1102	2
t_4	0.0000486	Vs_4	1183	3
t_5	0.0000446	Vs_5	1289	4
t_6	0.000033	Vs_6	1742	12.5
t_7	0.000032	Vs_7	1797	24

*T*able 6. S-wave velocity measurements on treated Ottawa sand with 1.5% expected precipitation for 24 hr.

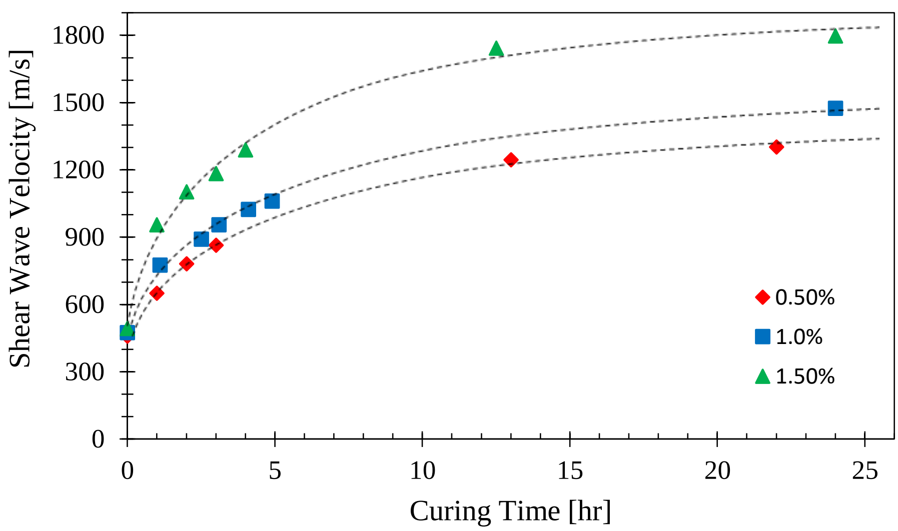

Figure 9. S-wave velocity variations for different calcium carbonate precipitations

It is worth mentioning that the reaction is repeatable and gives the same behavior when adding the same concentrations with the same relative density of the sand. We conducted several tests using bender elements and the results coincided as shown in figure (10).

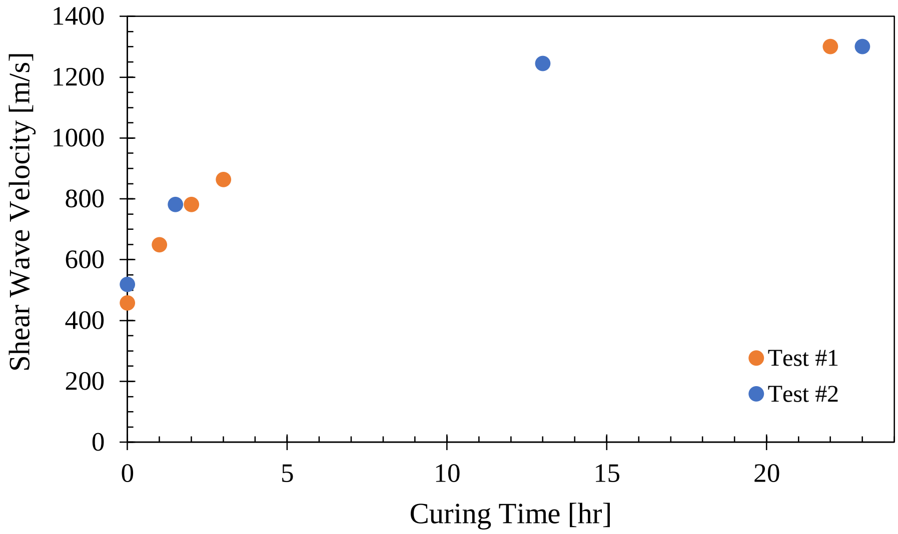

Figure 10. S-wave velocity measurements for two repeated tests with 0.5% target expected precipitation

3.3. SEM Images and EDX Analysis

To further investigate the shape and type of the bonds between the sand particles that are responsible for the increase in Vs, SEM imaging is conducted along with EDX analysis. Samples from the biocemented sand is introduced to the SEM machine after applying a metallic coating (Platinum or Gold). The sample size is 1cm×1cm and was viewed to the scale of 20µm to 100µm. The SEM imaging shows the size and shape of the calcite precipitation. The images show that the size of calcite precipitation ranges between 5µm to 50µm, and had a hexagonal crystal shape in all the tests completed (Figure 11a-11b-11c).

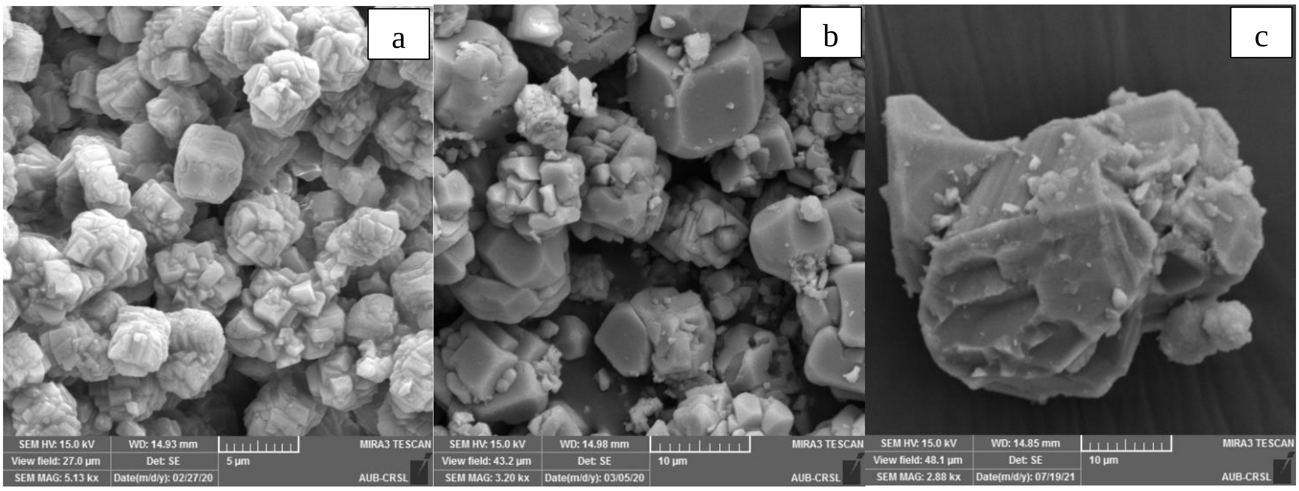

Figure 11. $CaCO_3$ precipitation size at different cementation quantities, (a) 0.5%, (b) 1.0% and (c) 1.5%

As expected, more calcite crystals are viewed in SEM when the target expected precipitation is varied between 0.5, 1.0 and 1.5% (Figure 12a-12b-12c).

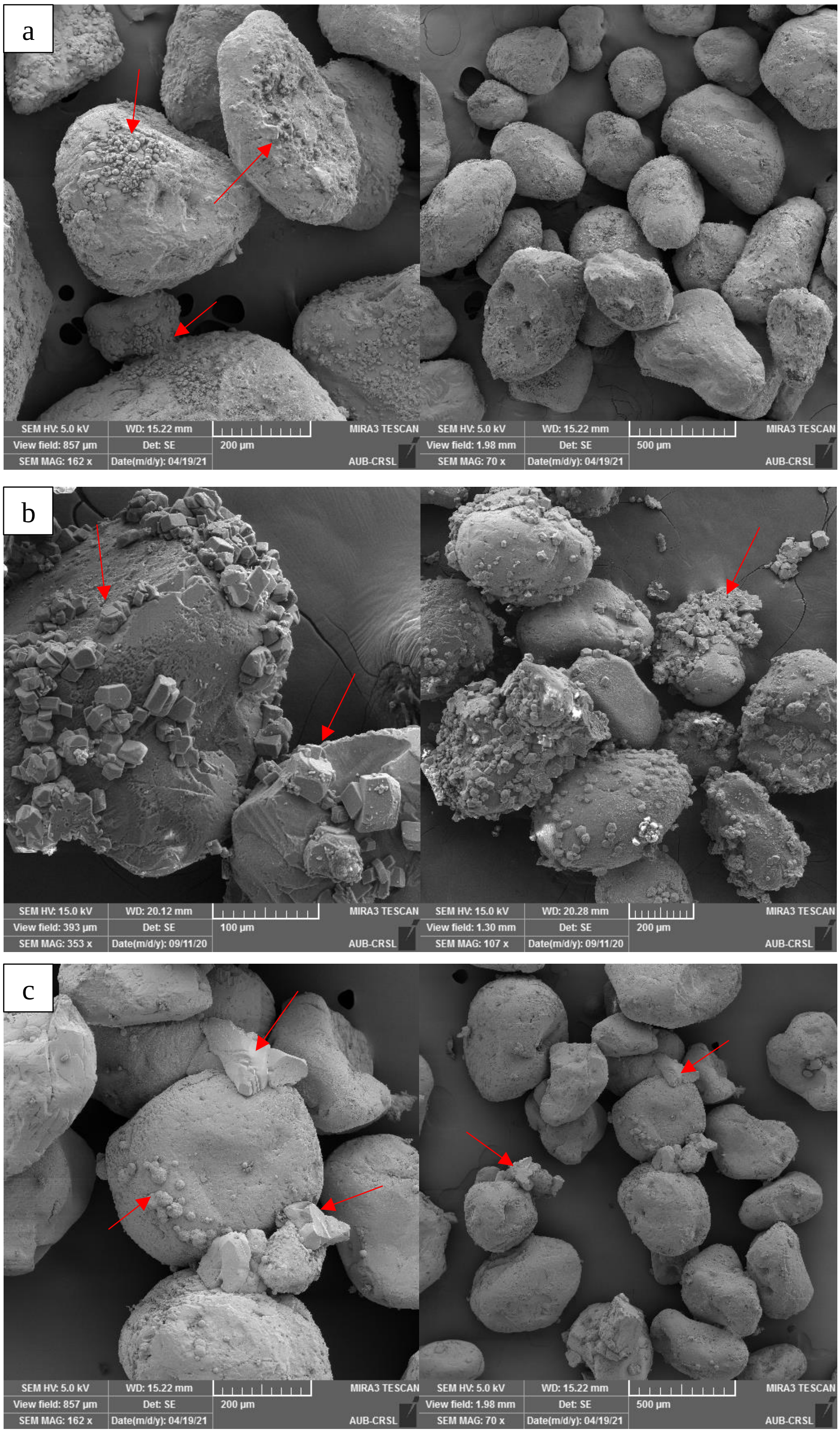

Figure 12. $CaCO_3$ precipitation size at different cementation quantities, (a) 0.5%, (b) 1.0% and (c) 1.5%

EDX analysis was used to verify the nature of these crystals and prove that the improvement taking place is because of calcite crystals. The analysis detected the presence of Carbon (C), Oxygen (O), and Calcium (Ca) representing the calcium carbonate molecules (Figure 13a-13b-13c).

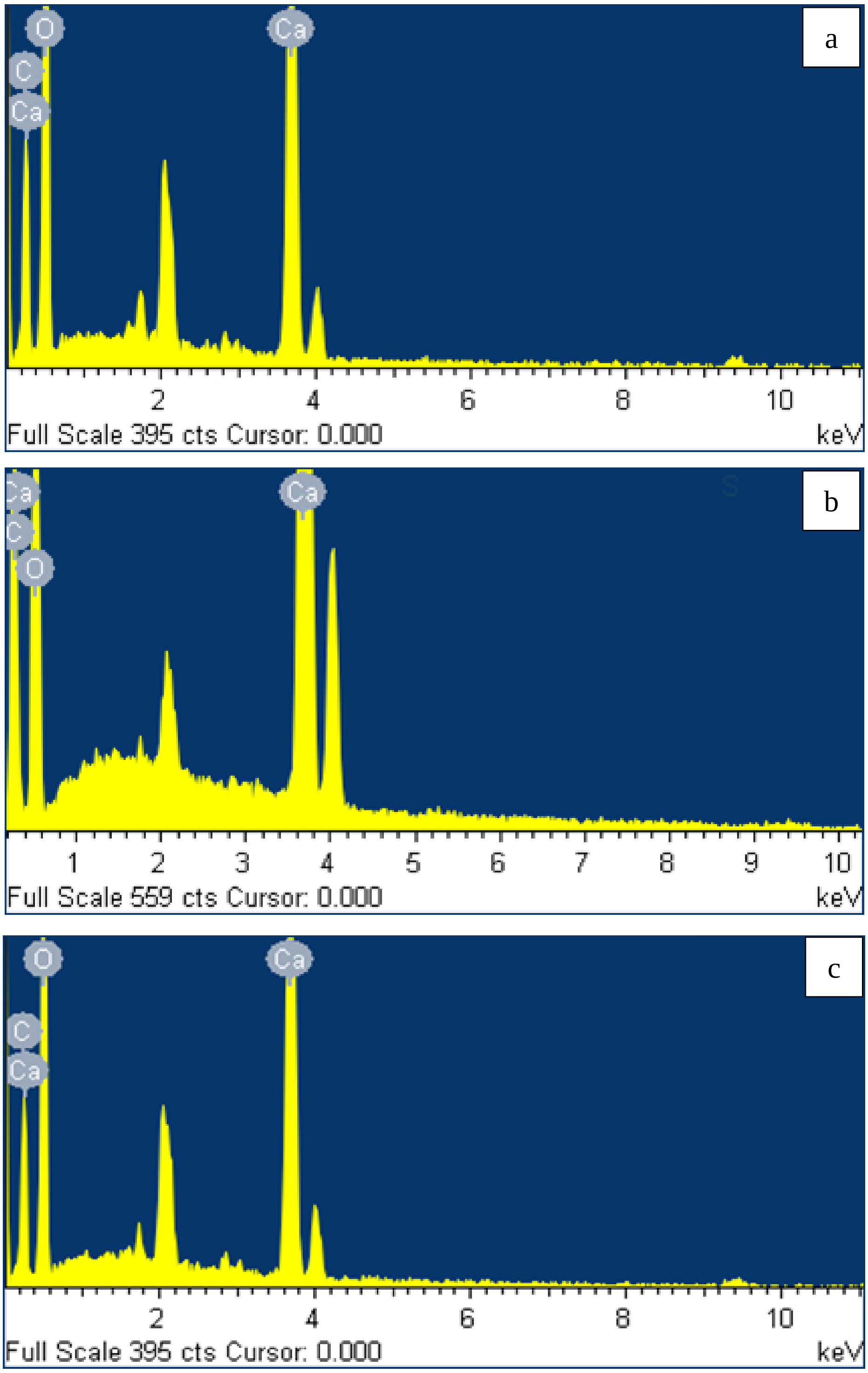

Figure 13. EDX analysis showing the constituents of the precipitations, (a) 0.5%, (b) 1.0% and (c) 1.5%

EDX analysis on sand samples detected the presence of Silica (Si) and Oxygen (O), which complies with the composition of the Ottawa sand used in the study as seen in figure (14).

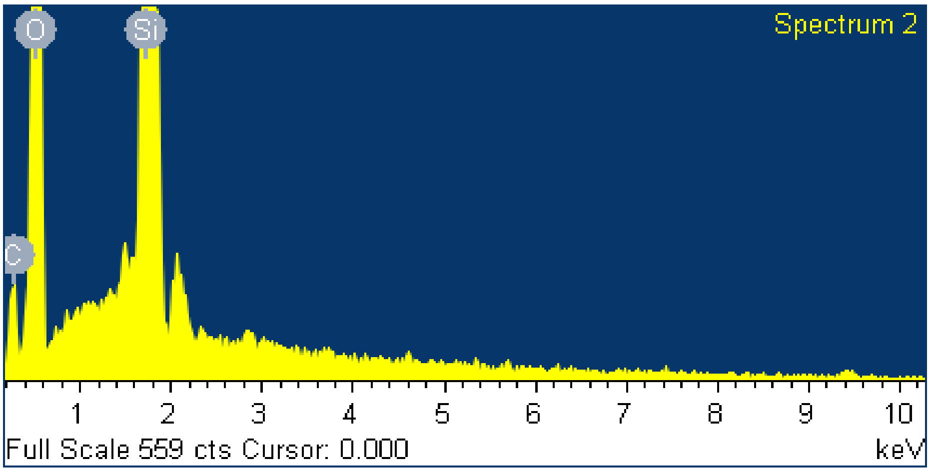

Figure 14. EDX analysis for Sand sample

3.4. Cyclic Loading on Treated Samples

At the end of the 24-hour curing time, gradual increase in the vertical load from 25kPa to 100kPa while performing signal readings. The cycles are then applied on the treated samples as done previously on the non-treated ones.

The cycles on the sample with 0.5% expected calcite precipitation are presented in figure (15). The cycles started with Vs of 1400m/s. After several cycles, Vs decreased from 1400m/s to reach 1200m/s at the end of the cycles. When applying each load, Vs reached values between 1350-1400m/s.

At the end of the cycles, gradual decrease in the load is done and the signals for Vs are recorded. It is noticeable that this sample lost part of it strength after performing the cycles since it reached Vs values (Point (2) = 1000m/s) below what we started the testing with (Point (1) = 1300m/s) as shown in figure (16).

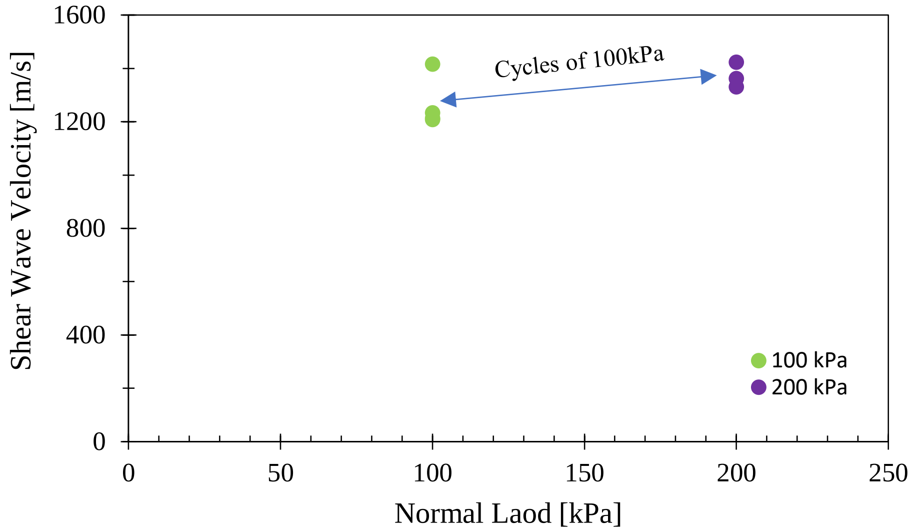

Figure 15. Vs values of samples with 0.5% expected precipitation under cyclic loading

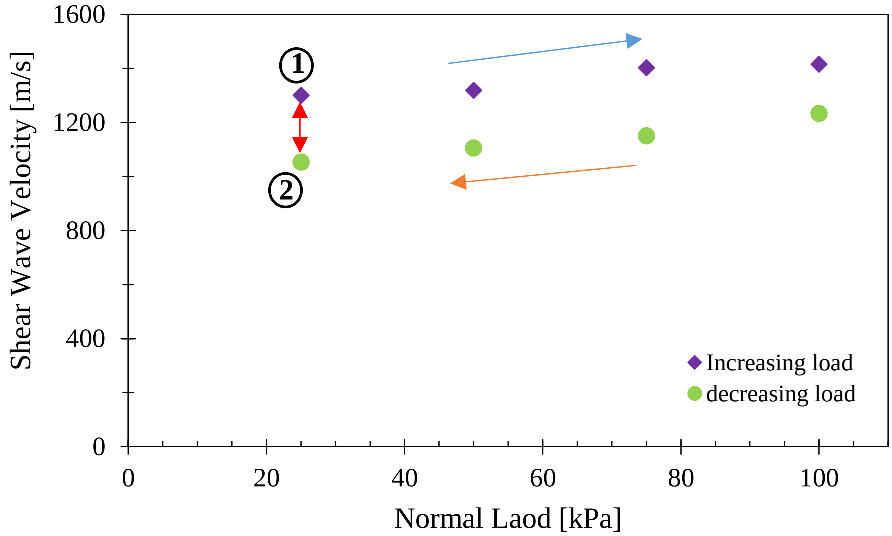

Figure 16. Comparison between Vs values at the beginning and end of the testing for samples with 0.5% expected precipitation

The same procedure was repeated on samples with 1.0% and 1.5% expected calcite precipitation. The sample with 1.0% showed similar behavior to the sample with 0.5%, as shown in figure (17). Vs reached constant values of 1550m/s when applying the loads. The same drop as in the previous sample (0.5% expected precipitation) in Vs value happens at the end of the testing (Point (2) = 1100m/s), compared to the values at the beginning (Point (1) = 1500m/s) as shown in figure (18).

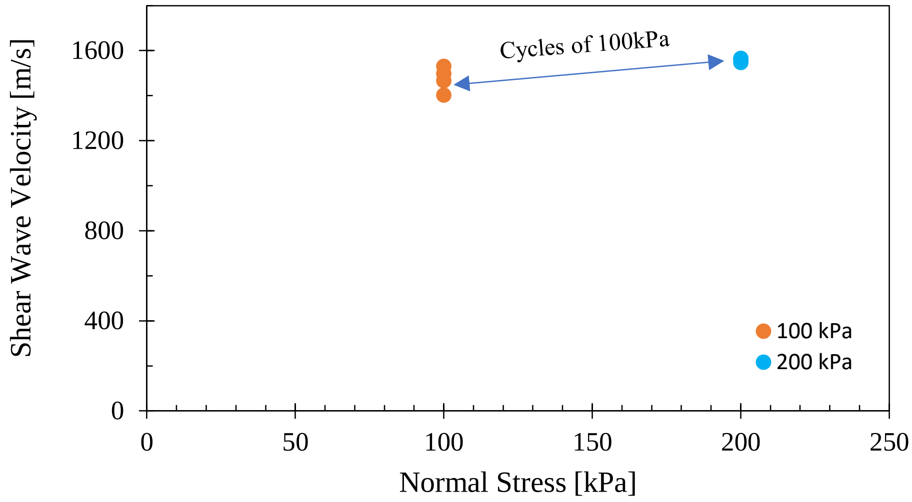

Figure 17. Vs values of samples with 1.0% expected precipitation under cyclic loading

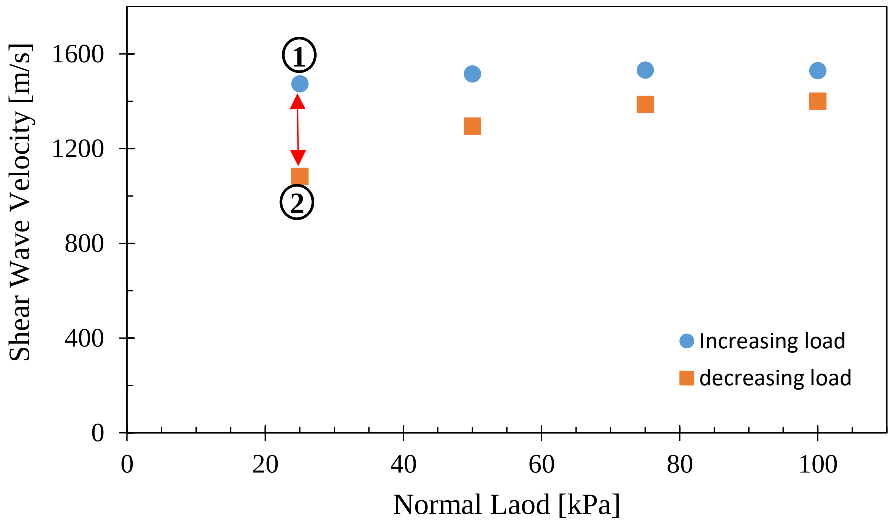

Figure 18. Comparison between Vs values at the beginning and end of the testing for samples with 1.0% expected precipitation

Finally, the cyclic test was performed on the sample with 1.5% expected calcite precipitation. The results showed no significant increase in Vs values as we increased the loading then applied the cycles on the sample. Figure (19) shows that the variations in the loading as we perform the cycles does not influence the values of Vs.

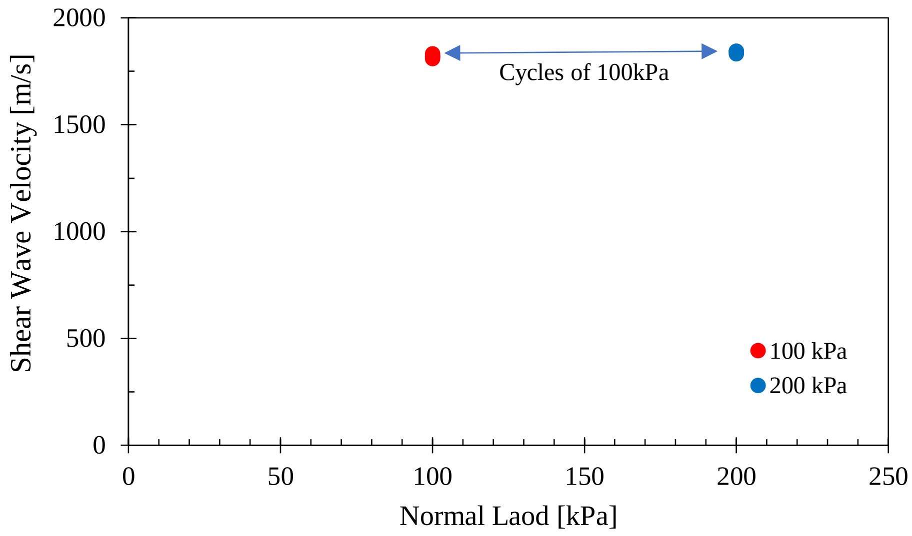

Figure 19. Vs values of samples with 1.0% expected precipitation under cyclic loading

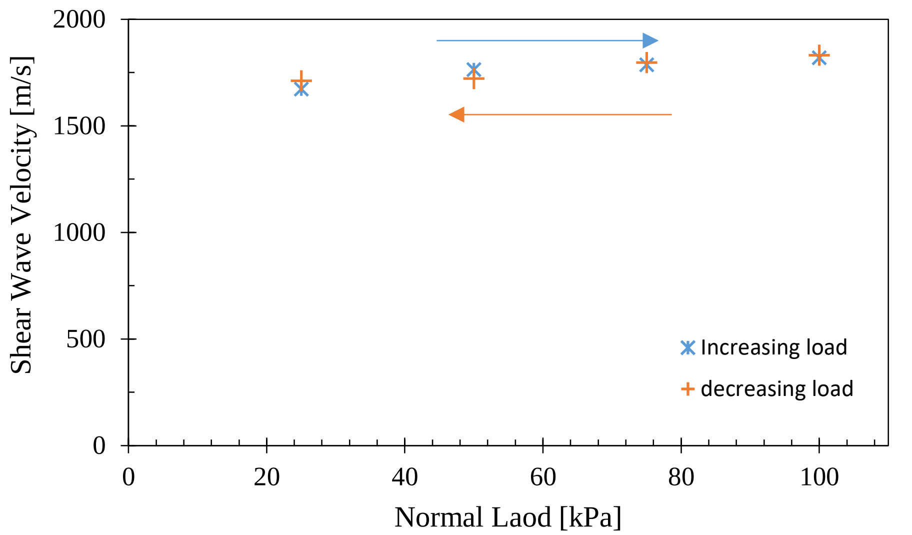

Figure 20. Comparison between Vs values at the beginning and end of the testing for samples with 1.5% expected precipitation

Similarly, figure (20) shows that Vs values at the beginning and end of the testing remain the same even after performing the cyclic loading.

3.5. Cone Penetration Test

The cone penetration test is conducted on samples prepared using the same procedure for the tall oedometer, using a cylindrical mold having the same dimensions as the oedometer. The samples prepared are replicates of the ones we conducted the S-wave testing on, having the same concentrations, to reach the same target of precipitated calcium chloride.

The miniature cone penetrometer, as described before, is made of a linear actuator, a load cell, and the cone probe. The cone probe has a diameter D = 6.5 mm and a height H = 5 mm as shown in figure (21).

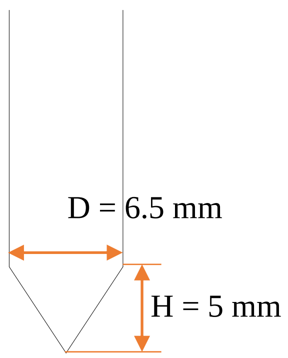

Figure 21. Cone probe dimensions

The sample is placed in the setup and the cone penetrates the same at a rate of 3mm/s. The recorded resistance by the load cell (in Kg) is converted to kPa and presented in figure (22).

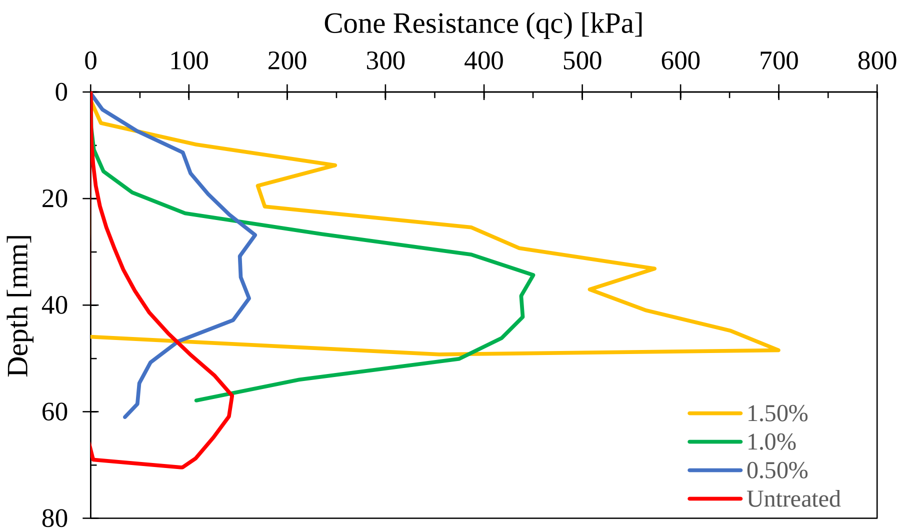

Figure 22. Cone resistance (qc) variation with the depth of sample

The samples with more calcite content present higher qc values as the cone penetrates through the depth of the sample. The sample with 1.5% expected cementation precipitation yielded the highest qc value of 700kPa, while 1.0% reached 450kPa and 0.5% reached 170kPa. The results show significant increase in the resistance of the sand compared to the untreated samples (140kPa). This increase in the resistance is due to the densification and the bonds created in the sand after the cementation treatment.

The cone penetrometer also gave an image about the distribution of the cementation solution through the samples. The precipitation is distributed along the sample with accumulation at the lower half.

CHAPTER 4

DISCUSSION AND RECCOMENDATIONS

The laboratory experiments confirmed that the biocemented sand samples were improved, as a result of the EICP treatment. The S-wave velocity measurements proved to be a convenient method to quantify the improvement encountered in the sand samples. A comparison between 3 treated samples, using different expected percentage of calcite precipitation (0.5%, 1%, and 1.5%) behaved in a similar manner. The Vs gradually increased and then plateaued. It is important to keep in mind that all three samples behaved similarly, yet reached different S-wave values. As expected, the precipitation did not impact the trend, because the experiment stops and stabilizes at a certain stage in all three samples, causing a constant shear velocity. It was in accordance with the percentage of calcite precipitation, that the precipitation would increase, and the Vs would increase. This is due to the fact that as the percentage of precipitation increased, the density increased, which increased shear velocity. This is in compliance with the findings of Qabany et al., 2011 which shows good correlation between Vs and the amount of calcium carbonate precipitation.

Furthermore, when comparing all three samples, Vs at 0.5% had a mild slope compared to 1.5% which has a steep slope. This proves that the reaction is producing more calcite during the same period due to the presence of higher quantities of urea, calcium chloride, and urease enzyme. The hydrolysis of urea and the creation of calcium carbonate caused the shear velocity to increase, and this is due to densification. The presence of the calcium carbonate was proved by SEM and EDX analysis, It is known that densification resists liquification, and cyclic loading causes liquification. After reaching the plateau, cyclic loading was performed. This was done to track the behavior of biocemented sand when performing cyclic loading. As expected, the biocementation resisted the liquification because biocementation does densification. At 1.5% precipitation, no variations in shear wave velocity were spotted after multiple cycles of loading and unloading which proves the resistance of biocementation. I assume that biocementation resisted due to the fact that the bonds between the particles of the sand are stiff. This is evident by the increase in the size of these particles which was observed under SEM. Thus, the bonds are naturally strong; yet, it is further in accordance with the increase of size that the bonds grew stiffer and more resistant. Also, the concentration of the solution helped. This is because, the more the cementation solution, the more calcium carbonate is produced, which aligns with densification. Thus, pores were filled, samples were compact, and there weren't much of spaces to allow the particles to move.

In the case of lower levels, where percentages were 1% and 0.5%, there was a similar behavior and trend; yet, a slight difference in resistance to cyclic loading was indicated. This is because less quantiles of calcium carbonate were generated. This allows for more space for sand to move. The movement of sad allows the bonds to break under repeated loading cycles. Therefore, the trends were similar but not identical.

However, when comparing low cementation with untreated samples, they presented a significant resistance in comparison to the untreated samples. Thus, it is evident that cementation resisted the cyclic loading in a significant manner. Also, as the quantity of cementation increases, the resistance to cyclic loading increases. In addition, as the quantity of cementation increased, the drop in the shear velocity decreased which further indicated resistance. This is because drops in shear velocity will result in bond breakage.

Moreover, a destructive test was performed to establish correlations between nondestructive and destructive tests to prove my results to be reliable. This test was the Cone penetration test. Under the cone penetration test, the values of qc increased as quantities increased. Also, under cementation of 1.5%, the highest strength was obtained. This deems the results reliable because they align with the findings of the nondestructive shear velocity tests.

The precipitation was distributed along the sample when conducting the cone penetration test, with accumulations at the lower half. This can be because of evaporation of the solution at the top layers, and the cementation reaction was not taking place throughout the duration of the curing process at these layers.

The level of increase was 5 times between the untreated and treated sample (1.5%) in the S-wave velocity. On the other hand, the level of increase between the untreated and treated sample (1.5%) in the cone penetration test was 4.7 times. This shows a similar compatibility of levels increasing between the destructive and nondestructive tests, at 1.5%. The latter applies to the 1.0% expected calcite precipitation tests, where we have an increase of 3.6 times in the destructive verses 3.2 times for the nondestructive tests. Thus, a similar compatibility of levels increasing was shown between the destructive and nondestructive tests, at 1.0%.

However, at 0.5%, this was not witnessed. This can be explained by the fact that at low cementation content, the quantity and size of precipitation did not affect the strength of samples. In the cone penetration test, the bonds did not resist the small surface penetration of the cone, which resulted in low strength. This was presented by no significant increase in qc between 0.5% treated samples and untreated ones. On the other hand, in the S-wave velocity measurements, the presence of precipitation caused the increase in S-wave velocity between 0.5% treated samples and untreated one. This proves a different compatibility of levels increasing between the destructive and nondestructive tests between 0.5% treated samples and untreated one. I assume that as the compatibility between destructive and nondestructive tests happens at higher cementation content.

Previous studies done on the matter did not perform a continuous shear velocity measurement. In this study, the Vs was measured continuously through the reaction time. It was performed as such in order to better understand how calcite is developing inside the sample. It was detected that maximum reaction happened in the first three hours of the experiment, whereby the biggest amount of calcium was obtained. Yet, the remaining amount of calcium took more time to develop and crystalize. Thus, the continues measurements of S-wave velocity helped in understanding of calcite production in the samples.

Although this presented work demonstrates benefits in understanding the behavior of biocemented sands under cyclic loading, there are still existing challenges in the field. This study will lead the way to more studies on biocementation in order to effectively handle the matter of interest. This project was executed on isolated molds; thus, in real life applications, we are unaware if the same results will be generated under natural conditions and factors. Also, all the tests were performed on dry sands of which the cementation solution was injected; yet, what would be the case if a saturated medium was there, and we needed to conduct the improvements on. We are unaware how the solution would behave and how it would disperse, or whether the calcite precipitation will bond to sand or float. All these interests are encouraged to be worked on for us to make maximized benefits from the topic at hand.

CHAPTER 5

CONCLUSIONS

Nowadays, construction work on fragile soils became common and inevitable, this is due to the increasing worldwide shortage of land. This weak soil accumulations tend to be characterized with being low in strength and high in compressibility. Thus, new soil developmental techniques will necessitate evolutionary methods of thinking, this is to ensure efficient and effective improvement in the soil settlements. It is also important for the new techniques to involve sustainable and eco-friendly characteristics. On this matter, using calcite-inducing reactions to produce natural cementation in soils is a promising technique to the geotechnical and environmental arena. It is evolutionary, sustainable, ecofriendly, and replicable. Although cementation is naturally existing in the aged soils, it does not tend to be considered in designs because its characteristics are still not well understood. This is why our approach was to inject cementation solutions with known characteristics in the field. This study explored the behavior of biocemented sands under cyclic reactions of loads and unloads. The experimental observations indicated that treated samples had an improved properties such as increase in resistance to liquefaction, in comparison to non-treated samples. Also, among the treated samples, samples with higher percentage of precipitation gave an improved properties, increased resistance, and increased stiffness. Although we currently have a well-rounded understanding of how treated and untreated sands behave under cycles of loads and unloads, the adoption of such techniques in the fields is still limited until we expand our research into understanding how other factors impact their behavior. Models connecting lab experiments, numerical simulations, field applications, and theories about soil mechanism are needed in order for these techniques to be attained in the field.

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