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# Development of novel ground improvement methods

by

## Xinyi Jiang

A thesis submitted to the graduate faculty

## in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE

Major: Civil Engineering (Geotechnical Engineering)

Program of Study Committee: Cassandra Rutherford, Major Professor Bora Cetin Kaoru Ikuma Vernon Schaefer Michael Bartlett

The student author, whose presentation of the scholarship herein was approved by the program of study committee, is solely responsible for the content of this thesis. The Graduate College will ensure this thesis is globally accessible and will not permit alterations after a degree is conferred.

Iowa State University

Ames, Iowa

2019

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### ABSTRACT

Numerous ground improvement technologies have been developed over the last few decades to address problematic soils, marginal sites and geohazards. Soil erosion is a leading geohazard causing infrastructure damage during storm and flooding events. Researchers have studied various bio-treatment methods to decrease erosion susceptibility of coarse-grained soils. Bacterial Enzyme Induced Calcite Precipitation (BEICP) was explored in this study to increase undrained shear strength and decrease soil erosion from moving water. This research investigates the surface erosion control for the mixture of 20-30 standard Ottawa sand and Iowa Western loess silt stabilized by BEICP applied by a spray method. The results obtained in this study indicate that the higher enzyme concentrations increase the surface shear strength, and that the formation of the calcite precipitation provides increased resistance to erosion. The depth of the calcite precipitation into the soil specimen was also investigated.

Soft soils are also considered problematic soil due to their low undrained shear strength and compressibility. Various methods have been used to increase the shear strength such as addition of fibers, shredded rubber tires and geosynthetics. This research investigates adding magnetic particles and using a magnetic field to rotate the particle orientation to increase the shear strength of soft soils. A soft soil surrogate (laponite) which is also a transparent material, was used to visualize the rotation of the magnetic particles. The addition of the magnetic particles was shown to significantly increase the undrained shear strength. Preliminary work using a controllable electro-magnet to create a magnetic field to rotate the orientation of the magnetic particles at small scale is also presented.



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## **CHAPTER 1. INTRODUCTION**

Ground improvement methods are used to improve the geotechnical properties of problematic soils. Techniques have been developed to mitigate potential hazards (i.e. liquefaction, landslides, erosion) and improve soil conditions for marginal sites (i.e. soft soils, loose sands, etc.) Many ground improvement methods have been developed and are currently in use. These methods have been categorized into groups by different authors: Mitchell (1981) categorized based on the construction/function, Hausmann (1990) categorized based on process, Ye et al. (1994) categorized based on function, Chu et al. (2009) categorized based on soil type and inclusion, Schaefer and Berg (2012) categorized based on application, and Han (2015) categorized based on function. The proper selection of a ground improvement technology needs to focus on the necessity of the ground improvement level and site specific factors which include structural conditions, geotechnical conditions, environmental constraints, construction conditions and reliability and durability of the improvement (Han, 2015).

#### **1.1 Background**

Advances in ground improvement methods over the last decade have led to novel methods using bio-treatment/bio-cementation as well as various soil additives to improve geotechnical properties of problematic soils or reduce geohazards such as erosion. This research focuses on investigating two emerging applications of ground improvement, namely, biocementation and soil reinforcement.

#### **1.1.1 Bio-cementation**

Soil erosion due to moving water may cause damage to an environmental system such as a river or coastline create stability problems for infrastructure leading to an increase the maintenance cost (Rivas, 2006). Soil erosion could also be one of the main factors that cause the



levee and dam failures and slope stability problems (Foster et al., 2011; Li et al., 2013). There are two types of erosion control methods available, short-term control and long-term control. Vegetation is one of the most cost-effective long-term methods to provide erosion control for soil (Nichnadowicz, V. F. 2001). Some methods to reduce internal erosion include seepage induced sandy-clay mixtures reduction (Jiang et at. 2016), seepage flow control/reduction (Fell et al. 2005, Engemoen, 2012), and chemical stabilization (Adams et al. 2013).

Bio-treatment/bio-cementation for subgrade stabilization has been investigated as a ground improvement method that can be used to improve soil properties and reduce erosion. Biotreatment systems provide a network for chemical reactions to occur which could potentially alter soil geotechnical properties (DeJong et al., 2010). Improved soil geotechnical properties include permeability, stiffness, compressibility, shear strength, and volumetric behavior (DeJong et al., 2010). Multiple methods of bio-treatment have been investigated. One method, Microbial Induced Calcite Precipitation (MICP), is the process of using microbial urea hydrolysis and carbonate ions to react with calcium to form calcite precipitation which can lead to increased shear strength in granular materials (Shanahan et al. 2014, DeJong et al., 2010, Whiffin et al., 2007, Cheng et al. 2014). While the application of MICP for marine conditions is a sustainable way to increase the strength of sands in marine and coastal environments, it requires a large number of application cycles to reach the high undrained shear strength improvements (Cheng et al. 2014). Enzyme induced calcite precipitation (EICP) is another bio-inspired process which uses ureolysis as the reaction to produce carbonate mineral precipitation (Hamdan and Kavazanjian, 2016, Kavazanjian and Hamdan, 2015). Researchers have compared EICP and MICP and found that MICP provides for a higher strength for surface stabilization, greater



efficiency and more rapid carbonate precipitation (Kavazanjian & Hamdan, 2015, Kavazanjian et al. 2017).

Another method of bio-cementation that has been recently investigated by researchers (Hoang et al., 2018) is Bacteria Enzyme Induced Calcium Precipitation (BEICP). BEICP refers to the formation of a calcium carbonate precipitation during the reaction between a bacterial enzyme and a chemical solution. Limited research using BEICP to decrease soil erosion susceptibility has been investigated.

#### **1.1.2 Soil reinforcement**

Various additives such a fiber (natural and synthetic), shredded rubber, plastic and shredded rubber have been used to reinforce soft soils. The fiber reinforcement technologies use the wood, pioneer plants (Gray & Ohashi, 2008). Researchers have demonstrated that fiber reinforcement technology could effectively increase the undrained shear strength of soil. (Ahmad et al., 2010). However, the orientation of the particles and thus the soil fabric could not be modified as desired due to the particles' physical properties.

The combination of using a magnetic field and magnetic particles in geotechnical engineering may have the potential to modify the particles' orientation and thus allow for the engineer to "design" the soil fabric. Magnetic fields have been used to create membranes and films and reinforce synthetic composites (Akbari et al., 2016, He et al., 2016, and Erb et al., 2019). Magnetic particles/powder and magnetic fields have also been used to increase the strength of soils in triaxial tests and steel slag has also been used into increase soil strength and soil sustainability (Dayioglu et al., 2014). Although previous research has shown that the addition of magnetic particles and magnetic fields can increase the strength of the materials, there is not any reported studies on using magnetic field to rotate the orientation of the magnetic



particles to control the fabric. One study by Lin et al. (2017) demonstrated that suspended graphene flakes in water could be rotated and re-orientated with the application of magnetic field.

#### **1.2 Objectives**

This study explored two novel ground improvement techniques: (i) Bacterial Enzyme Induced Calcite Precipitation (BEICP) and (ii) the use of magnetic fields to rotate magnetic particle orientation within transparent soil. The primary research objective was to investigate the preliminary use of these two new ground improvement techniques to improve geotechnical properties of soils. The overall objective was broken into 6 specific research objectives (3 for BEICP and 3 for magnetic particle rotation):

- Investigate the use of BEICP to increase the shear strength of silty sand using a spray method for application,
- Determine the depth of calcite precipitation within a silty sand specimen using a spray method application,
- Determine the effectiveness of BEICP to decrease erosion susceptibility of silty sand using a flume by comparing the improvements,
- Design an experimental procedure to suspend magnetic particles (graphene flakes, steel slag and iron fillings) within a soft soil surrogate (Laponite),
- 5) Investigate the use of graphene flakes, steel slag and iron fillings as an additive to increase the shear strength of a soft soil surrogate (Laponite),
- 6) Design a methodology to experimentally test the use of magnetic fields to rotate magnetic particles within a soft soil surrogate (Laponite).



#### **1.3 Organization of Thesis**

Chapter 2 reviews the literature on traditional and state of the art ground improvement technologies focusing on the development bio-treatment methods and use of reinforcement (fiber, rubber, etc.) in soils. Previous research on the use of transparent material as a soil surrogate and the use of magnetic fields to rotate magnetic particles within soil is also presented.

Chapter 3 presents a detailed experimental plan for the use of BEICP to increase the undrained shear strength and decrease the erosion susceptibility of granular soil.

Chapter 4 describes the experimental plan, materials and procedures used in the magnetic particle rotation within transparent soil portion of the study.

Chapter 5 summarizes the results and provides discussions for each novel ground improvement technique. The first section focuses upon the bacterial enzyme induced calcite precipitation technology, and the second the addition of magnetic particles, rotation and orientation within transparent magnetic soil.

Chapter 6 provides the conclusions of the two novel techniques demonstrated and investigated within this study, and recommendations for further research.



## **CHAPTER 2. LITERATURE REVIEW**

#### **2.1 Ground Improvement Technology**

The use of ground improvement technology is necessary when improved geotechnical engineering performance is required for a site (USACE, 1999, Schaefer and Berg, 2012). Mitchell (1981) provides a comprehensive report on the state of the art in ground improvement. The main functions of ground improvement methods include: (i) increase shear strength and bearing resistance, (ii) increase density, (iii) decrease permeability, (iv) transfer loads to competent layers, (v) control deformations, (vi) acceleration consolidation, (vii) decrease imposed loads, (viii) provide lateral stability, (ix) form seepage cutoffs, (x) fill voids, and, (xi) increase resistance to liquefaction (Munfakh 1997a; Elias et al. 2006). Most ground improvement methods are summarized into eight categories: (1) densification, (2) consolidation, (3) load reduction, (4) reinforcement, (5) chemical treatment, (6) thermal stabilization, (7) biotechnical stabilization, and (8) miscellaneous (Schaefer and Berg, 2012). Ground improvement technologies can be applied to a variety of soils and site conditions including: soft soils, loose sand, earthwork construction, earth retention and grouting in construction. The novel ground improvement methods investigated in this study will fall under the biotechnical stabilization and reinforcement categories.

#### 2.2 Bio-treatment/bio-cementation to Increase Soil Strength

Bio-treatment or bio-cementation of soil is a method that is considered an emerging ground improvement technology. These methods have been shown to improve soil properties such as soil strength, permeability and stiffness (DeJong et al. 2010, Mitchell and Santamarina (2005). Bio-cementation stabilization methods can be used for the stabilization of subgrade soil in which a biological process is used with a chemical reaction to produce a connection between



soil particles and improve soil geotechnical properties. DeJong et al. (2010) illustrated how the bio-mediated improvement system could change geotechnical properties of soils (Figure 2.1). Calcite precipitation occurs between the particles of sands which alters the pore space. Bio-mediated treatment for soil improvement has the potential advantages of reducing cost and impact to the environment by using natural materials and can be adapted to various with soil conditions (DeJong et al., 2010).



Figure 2.1 Bio-mediated soil improvement system (Figure from DeJong et al., 2010)



One method of bio-cementation is microbially induced calcite precipitation (MICP) which uses microorganisms to form calcite between the soil particles. Qabany & Soga (2013) reported the reaction for MICP as

$$CO (NH_2)_2 + 2H_2O \rightarrow 2NH_4 + CO_3^{2-} pH \uparrow \qquad Equation 1$$

$$Ca^{2+} + CO_3^{2-} \leftrightarrow CaCO_3 \downarrow pH \downarrow$$
 Equation 2

DeJong et al. (2006) used *Bacillus pasteurii* as the MICP microbe to improve the strength and shear stiffness of Ottawa 50-70 sand prepared at a relative density at 35%. Whiffin et al. (2007) treated a five-meter sand column with the MICP technique to show the method can be used in field applications. The study demonstrated that MICP technique made a significant strength increase and a reduction in soil permeability. Cheng et al. (2014) investigated biocemented samples urease activity, crystal content, permeability and strength under the marine salt water conditions. The unconfined compressive strength and permeability of the seawater treated specimens were compared with specimens treated with the cementation solution as illustrated in Figure 2.2. The SEM images of the MICP treatment with seawater indicated the surface of the sand particles had formation of calcium carbonate between the particles, and the formation of the carbonate increased the unconfined compressive strength.





Figure 2.2 Bio-cemented sample treated with seawater and chemical solution of (a) UCS strength; (b) Permeability (Figure from Cheng et al., 2014)

Shanahan & Montoya (2014) used the *Sporosarcina pasteurii* as the biological organism to treat Ottawa 50-70 sand at a relative density of 40% for a sand dune model. The results show that prepared soil specimens treated with MICP technology had less erosion compare with the untreated soil. The average mass of the calcite for MICP treated specimens was as high as 6.6%. Gomez & DeJong (2017) investigated the MICP treatment of 14 different soils. Measurements of shear wave velocity, hydraulic conductivity, unconfined compressive strength, and calcite content were compared. The results from the study showed that post-treatment UCS and



hydraulic conductivity had significantly reduced for soil of D<sub>10</sub> particles size and fine content. They also showed that the final calcite content increased with a fine content up to 13%. The use of MICP to decrease the internal erosion control of sand-clay mixtures were also investigated by Jiang et al. (2016). Dry sand and kaolin-clay were used as the soil material. The results showed that MICP treatment for the sandy-clay mixtures has decreased permeability and increased volumetric contraction of the particles. However, there was insignificant improvement for the critical shear stress and erosion resistance due to the existing of the fine content.

Enzyme Induced Carbonate Precipitation (EICP) is another bio-treated ground improvement technology. Neupane et al. (2013) examined the technology by injecting the reagent and enzyme solution into sand specimens. The results indicated that the calcite precipitation for sand samples treated with reagent and enzyme solutions could be applied for the large scale in situ site. The EICP technology is the process using ureolytic from agricultural urease to precipitate the calcite. Kavazanjian and Hamdan (2015) used Ottawa 20-30 sand and F-60 sand mixtures treated by the EICP technology. The results indicated that treated Ottawa 20-30 sand obtained a peak strength at 529 kPa (76.73 psi), and the F-60 sand obtained a peak unconfined compressive strength of 391 kPa (56.71 psi). They also demonstrated that EICP could be used to form columns within the subgrade for large area treatment. Kavazanjian et al. (2017) also explored on forming vertical and inclined columns by using EICP technology. The vertical columns created by EICP technology were tested for dry and flooded condition, and inclined columns were tested with a variation of degrees inclined. The results show that both vertical and inclined columns treated by EICP technology can provide stabilization for cohesionless soils as a ground improvement technology.



#### 2.3 Soil Reinforcement

Soil reinforcement using various additives can also improve soil geotechnical engineering properties. Gray & Ohashi (2008) investigated natural and synthetic fiber reinforcement. The testing results showed a shear strength increase for both loose and dense sand. The application of fiber reinforcements also had the ability to control shear resistance post peak reduction for dense sand. Jiang et al. (2010) explored soil reinforcement by adding short polypropylene fiber. The fiber reinforced soil showed an increase of the unconfined compressive strength, cohesion and internal friction angle. Gray and Al-Refeai (1986) compared the stress-strain response of soils with continuous, oriented fabric layers and discrete, randomly distributed fibers. The testing results showed that the dry sand reinforced by both continuous, oriented fabric layers and distributed, discrete fibers had significantly increased strength and modified stress deformation behavior.

#### 2.4 Magnetic Particle Soil Improvement

Previous work has used magnetorheological fluids (MRFs) and powder to suspend the micron sized magnetic particles in the fluid and within soils to increase the strength once a magnetic field had been applied. The use of MRFs to solidify coarse-grained soil and improve the engineering soil properties was investigated by Hryciw & Susila (2005); Aydar et al. (2010); Susan-Resiga et al. (2010) and Whiteley et al. (2010). The results of the study demonstrated that the application of magnetorheological fluid/powder combined with magnetic field can solidify the soil temporarily and increase strength. No previous studies on the use of magnetic fields to rotate particles within soils were found.



# CHAPTER 3. BACTERIAL ENZYME INDUCED CALCITE PRECIPITATION (BEICP) METHOD

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#### **3.1 Research Objective**

The main research objective of the BEICP method testing was to determine to what extent the calcite precipitation increased the undrained shear strength and decreased the erosion susceptibility at the surface of the silty sand mixture using a spray application method. Various concentrations of enzyme were used to determine the depth of the calcite precipitation into the sample specimens, increased strength and improved resistance to water erosion.

#### 3.2 Growth Media, Cell Harvesting and Enzyme Extraction

The source of the urease was Sporosarcina Pasteurii (ATCC11895) and was obtained from Dr. Karou Ikuma's lab prepared by Rayla Pinto-Vilar. The growth media consisted of tryptic soy broth (20 g/L), ammonium sulfate (0.08 M) and tris base (0.13 M pH 9). After incubation at room temperature, the liquid cultures were continuously agitated at 160 rpm for 48 hours to an OD<sub>600</sub> value of 1.2-1.3. Cells suspension were centrifuged for 5 min, at 20 C and 8000 RCF to harvest the cells. After the supernatant was discarded, the pellet of cells was re-suspended in 20 ml phosphate buffer 50 mM pH 8. A phosphate buffer was used to wash the cells twice. The obtained cell pellet was then stored at 4°C. To extract the enzyme, the stored cell pellet was re-suspended in 20 ml of phosphate buffer 50 mM pH 8. The cell suspension was sonicated for 18 cycles, each of 2 min continuous sonication and 1 min off to lyse the cells. After completion of all cycles, the suspension was centrifuged for 5 min, at 20 C and 8000 RCF. The supernatant was filtered through a 0.2  $\mu$ m filter to separate the total protein extract from cell debris and intact cells. The resulting protein extract was dialysed to reduce the background concentration ammonia. The obtained protein extract was stored in the freezer until further use. The concentration of total protein in the



extract was determined using NanoDrop measurements with absorbance at 280nm (A280) prior to freezing.

#### **3.3 Chemical Solution Preparation**

The chemical solution used for BEICP application is a mixture of Calcite Chloride Dihydrate (CaCl<sub>2</sub>·H<sub>2</sub>O) and Urea (CH<sub>4</sub>N<sub>2</sub>O). When preparing each of the chemical solutions, the specified amount of water was added to the chemical for the desired concentration and mixed thoroughly. For both Calcite Chloride Dihydrate and Urea solutions, the concentration was determined to be 0.3 M and mixed at 1:1 ratio (Hoang et al., 2018). After preparation of each of the chemical solutions, the mixtures were allowed to sit for 10 minutes.

#### **3.4 Soil Specimen Preparation**

PVC tube containers were prepared for soil column specimens. The height of each PVC mold was designed to be 8.89 cm (3.5 inches) high and 6.35 cm (2.5 inches) inner diameter. A piece of Scotch Brite sponge was used at the bottom of the PVC tube to keep the soil in while still allowing drainage of the solution though the base. The PVC tubes were then divided into two parts, the bottom part was filled with 3.81 cm (1.5 inches) of washed gravel and the top part with 5.08 cm (2 inches) of the soil. A piece of Scotch Brite sponge was used to separate the washed gravel and soil specimen layers. An example of the PVC mold with a prepared soil column specimen is shown below in Figure 3.1





Figure 3.1 PVC model soil column c

The soil column specimens were prepared with 20-30 standard Ottawa sand, silt and water. The Ottawa sand which contains high content of silica  $(SiO_2)$  is used as the coarse-grained soil material. The Iowa western loess soil is a fine-grained, unstratified accumulation of clay and silt deposited by wind (Alpers et al., 2005), which may cause collapse with high loads (Peck et al., 1974). The silt used in the experiment was obtained by conducting a sieve analysis on the Iowa western loess soil and collecting the materials that passed No. 200 sieve (open size of 0.074mm). The gradation curve of the mixed soil material is shown in Figure 3.2. The gradation curve shows that soil is classified as a poorly graded soil based on the Unified Soil Classification System (USCS). The soil specimen columns with a weight of W were prepared by mixing 90% W of the 20-30 standard Ottawa sand with 10% W of the prepared silt, and 5% W of water. After mixing the soil, the PVC mold was filled to approximately 1/3 the height and the soil was compacted using tamping (25 tamps) with the tamping energy of 16.61 kJ/m<sup>3</sup>. Another two layers of soil were compacted to fill the entire PVC mold. A constant tamping compaction energy was applied to obtain similar relative densities and void ratios throughout the entire specimen. The dimensions for the prepared soil column specimens are shown in Figure 3.3. The



specific gravity of the soil column specimens was calculated to be  $G_s = 2.66$  based on the ASTM D854 – 14 Standard Test Methods. For the prepared soil column specimens, the geotechnical properties including moist unit weight, water content, dry unit weight and void ratio were calculated. Example calculations are shown below:

Moist unit weight of the original soil specimens:

$$\gamma = \frac{W}{V} = 112 \ pcf = 17.59 \ kN/m^3$$
 Equation 3 Moist unit weight

Water content of the original soil specimens:

$$w = \frac{W_w}{W_s} \times 100\% = 4.79\% \qquad Equation 4 Water content$$

Dry unit weight of the original soil specimens:

$$\gamma_d = \frac{W_s}{V} = 106.75 \ pcf = 16.77 \ kN/m^3$$
 Equation 5 Dry unit weight

Void ratio of the original soil specimens:

$$e = \frac{G_s \gamma_W}{\gamma_d} - 1 = 0.55$$
 Equation 6 Void ratio

Void volume of the original soil specimens is;

$$V_v = e \times V_s = 49 ml$$
 Equation 7 Void volume

Where:

W = weight of the total soil specimen

V = volume of the total soil specimen

W<sub>w</sub>= weight of water in the soil specimen

W<sub>s</sub>= weight of oven dries soil in the soil specimen

 $\gamma_w$  = unit weight of water



 $V_s =$  volume of oven dried soil



Figure 3.2 Grain size distribution of soil material (90% sand mixed with 10% silt)







Figure 3.3 Dimensions of the Soil Specimens (a) Height of the specimen and (b) Diameter of the specimen

#### **3.5 BEICP Technique**

The Bacteria Enzyme Induced Calcium Precipitation (BEICP) method refers to the formation of a calcium carbonate precipitation during the reaction between a bacterial enzyme and chemical solution. The BEICP technique was applied to the soil column specimens' surface to increase surface strength and decrease the erosion rate of the soil in the flume. The BEICP technique was applied with a spray method in this study because this would be the likely method applied in the field. During the spraying process, the angle between the spray bottle head and the soil column specimen's surface was in the range of 45° - 60°, as shown in Figure 3.4. The total amount of enzyme applied for each specimen was calculated to fill the void spaces in soil column specimens. Based on the void volume calculated in equation 5, a total of 50 ml enzyme



was applied to each soil column specimen. In order to allow time for the calcite precipitation to form, the total of 50 ml enzyme was applied to each specimen in 5 cycles. The amount of enzyme and chemical solution to be sprayed each time is shown in Table 3.1. The amount of the enzyme and chemical solution for each cycle was determined based on the previous experience. Prior to spraying the solution, the initial weight of specimens was recorded. After each spray cycle, a 3-4 hours wait time was allow so that the specimens' surface was totally dry. After the 5th spraying cycle was applied, the specimens were left at room temperature for 5 days. Finally, the specimens were placed in the oven at 60°C for 48 hours and the specimens' oven-dry weight was recorded.



Figure 3.4 Spray angle in BEICP process



	Cycle 1	Cycle 2	Cycle 3	Cycle 4	Cycle 5
Enzyme amount (ml)	15	15	10	6	4
Chemical solution amount (ml)	20	20	10	8	5

Table 3.1 Enzyme and Chemical Solution Spray Cycles

#### **3.6 Experimental Plan**

Four types of tests were conducted in this research study: (1) undrained shear strength test improvement; (2) erosion susceptibility; (3) depth of calcite precipitation within soil specimen based on spraying method, and (4) the use of Scanning Electron Microscope (SEM) imaging to determine calcite formation between soil particles. In the undrained shear strength and erosion testing, five different enzyme concentrations were compared. The enzyme concentration with the largest increase in surface shear strength was used to determine the depth of calcite improvement based on the spray method for application. These specimens were also used in the SEM images.

#### **3.7 Surface Shear Strength Prior to Erosion Testing**

The undrained shear strength was measured at the surface of soil column specimen using a pocket penetrometer. In this research, four different enzyme concentrations were used to treat the soil column specimens and were compared with the control of untreated soil. Three replicates of each concentration were tested. Because the BEICP technique was applied by a spray method, it was hypothesized that a majority of the calcite formation would be on the surface of specimen and therefore the specimen would not have a uniform increase of strength with depth into the specimen. The pocket penetrometer was used to test the surface treated soil column since the triaxial test was not suitable for non-uniform specimens (soil at the bottom of the specimen



would likely be untreated). Before conducting the surface strength test, a small amount of loose sand on top of the specimens' surface remained and therefore was removed because it would likely affect the pocket penetrometer testing results. The four concentrations of enzyme tested were C = 0 mg/mL, C = 0.3 mg/mL, C = 0.7 mg/mL, C = 1.5 mg/mL, compared with a control of untreated soil.

#### **3.8 Erosion Testing**

A recirculating flume (9.1 m long by 0.6 m wide), as shown in Figure 3.5, used for erosion test. A constant head tank regulated flow velocity, while a honeycomb structure following the inflow pipe broke down large structures and conditioned the flow. The depth of the water flow was controlled by a tailgate of 3 m at the downstream of the test section. The soil column specimens were attached to a plywood board and placed within the flume channel. Two concrete columns were placed on the plywood board to avoid any moving during the erosion test. The existence of the concrete column may cause affect a little of the flume velocity, but it was not explored in this research. During erosion testing, the height of water was controlled to be right above the soil column surface with a mean flume velocity of 23.2 cm/s. Figure 3.6 shows the erosion testing of the soil column specimens in the flume channel. The soil specimens prepared for erosion test were also treated with the same enzyme concentrations as in the strength improvement tests. Three replicates of each enzyme concentration were tested. In this study, treated specimens were subjected to the water erosion for a specified time and the weight loss of each specimen was measured after the test. The criterion of terminating the test was determined as any obvious visual erosion on the specimen's surface. Even if the specimens started to erode in less than 20 minutes, they were kept in the flume water for 20 minutes and the weight loss for each specimen was compared. After the erosion test in the flume channel, the testing soil column



specimens were placed in the oven at 60°C for 48 hours. The specimen's weight was recorded before and after the erosion testing to measure the weight loss. The shear strength prior to and after the erosion testing was also measured.



Figure 3.5 Flume channel used for erosion test



Figure 3.6 Soil Specimen in Erosion Test



#### 3.9 Depth of Calcite Formation and Strength Improvement

The depth of the calcite formation and undrained shear strength improvement was measured to determine the effectiveness of BEICP treatment by spray application. In order to test the strength improvement for different depths within the specimen, a new soil column mold was designed. A 10-cm tall PVC tube was cut into 5 short sections. Each tube section had a height of 2 cm. Electrical tape was used to connect the tube sections to prevent leaking of the solutions during the spray process and also allow for the sections to be separated after BEICP treatment. The bottom end of the column mold was closed with a piece of Scotch Brite sponge which would allow drainage of the excess solution (Figure 3.7). There soil column specimens were prepared with enzyme concentrations of C = 1.5 mg/mL, C = 0 mg/mL, and a control of untreated soil. The C = 1.5 mg/mL was the concentration provided the highest increase in shear strength at the surface of the soil and also had the lowest weight loss during erosion testing. The C = 0 mg/mL enzyme concentration was used as a comparison to determine if the solution had any effect on improved soil properties. The untreated soil material was used as a control having the original soil properties. The pocket penetrometer was used to measure the shear strength for each layer of the specimens. After the strength testing, the amount of calcium carbonate precipitation was measured by adding a 2M concentration hydrochloride. The chemical reaction between hydrochloride and calcium carbonate released carbon dioxide gas. The mixture was poured into a funnel made by the 09-801E filter paper. A vacuum filter was used (Figure 3.8) to allow the solution to move through the filter paper. The soil left on the filter paper was collected and placed in the oven at 60°C for 24 hours together with the filter paper. The weight difference before and after adding the hydrochloride to the soil sample was measured.





Figure 3.7 Dimensions of the precut tube layers (a) Diameter of precut tube, (b) Height of precut tube, (c) Height of each layer



Figure 3.8 Vacuum filter for calcium concentration test



#### 3.10 SEM Imagery & EDS Testing

Scanning electron microscope (SEM) imagery was performed by The Materials Analysis and Research Laboratory of the Iowa State University Office of Biotechnology (MARL). The SEM images were taken using FEI Quanta 250 FE-SEM shown in Figure 3.9. Images of the calcite precipitation within the treated soil and in the soil structure were taken. During the process, the energy dispersive x-ray spectroscopy (EDS) was also performed based on the backscattered electrons images. The EDS test was using a crystal to detect the x-rays by isolating from the SEM chamber from a thin window. The x-ray maps from EDS testing is a mapping system that shows where the element is abundant and where it is not. In the SEM & EDS tests, the soil samples were treated with C = 1.5 mg/mL enzyme concentration. Soil samples from the top layer and fourth layer were selected and compared with the untreated soil material.



Figure 3.9 FEI Quanta 250 FE-SEM



# CHAPTER 4. MAGNETIC PARTICLE SOIL IMPROVEMENT 4.1 Materials

#### 4.1.1 Transparent Soil

Transparent materials were developed and customized to simulate the soft soils. There are two groups of transparent materials, one group is silica gels to simulate the sand behavior, and the other group used is to simulate natural clays (Iskander et al., 1994; Iskander 2010). Iskander (2002) explored the geotechnical behavior and deformation of transparent materials to simulate natural clay under normally consolidation and overconsolidation conditions.

The LAPONITE RD was the transparent soil material used as surrogate of clay in this study. The laponite is a 2:1 layered silicate structural material with the similar natural clay mineral structure (Wallace and Rutherford, 2015). BYK Additives and Instruments (2014) reported laponite particles have a geometry of a 25nm diameter and with a height of 0.92nm, and the specific gravity is 2.53 (BYK Additives and Instruments, 2014) The laponite is in powder form as shown in Figure 4.1. The results of the geotechnical properties of the laponite indicated that the transparent clay prepared by laponite obtained comparable geotechnical properties to soft cohesive soils, and it could be used as soft clay surrogate for further non-intrusive observing testing (Wallace and Rutherford, 2015). Laponite were used in this study to simulate the soft clay geotechnical properties.





Figure 4.1 Laponite Powder

### **4.1.2 Magnetic Particles**

The magnetic particles used in this study were graphene flakes, steel slag which is a recycled material, and fine iron filings. The graphene flakes were flat sheets that are formed by the pure carbon atoms linked together. The graphene flakes passed through a number 100 mesh sieve. The purity of the graphene was reported by the manufacturer 99.9% (Flake graphite fact sheet). Figure 4.2 shows the graphene flakes and the SEM images of the flakes that were used in the study.

The steel slag particles are a by-product produced during the steel making process. The slag was solidified from the silicates and oxides upon cooling. The major source of the steel slag aggregate is produced during the primary stage of steel production (AASHTO et al. 2008). The grain size distribution curve for the steel slag used in this study is shown in Figure 4.3, which is similar to the material used in other studies by Dayioglu et al. (2014) as shown Figure 4.4. There were two size of the steel slags obtained in this study. The first has a gradation between the No. 60 sieve and the No. 80 sieve (opening size between 0.18mm and 0.25mm), and the second were



sieved passing the No. 80 and retained on No. 120 sieve (opening size between 0.125mm and 0.18mm). The second group of the steel slag particles were determined to use in this study because the larger particle size would be easier to track. Figure 4.5 shows the steel slag particles that were used in this study.

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Iron filings were also used as a magnetic material in this study. Two types of the iron filing particles were obtained, coarse (size of 20 mesh) and fine (size of 100 mesh) sized particles. The fine iron filing particles were used since the particle size for the coarse iron filling were too large to fit in the small-scale testing mold. Figure 4.6 shows the fine iron filling particles that were used in this study.



(a)

(b)

Figure 4.2 Graphene Flakes and SEM Image (Reference: <u>https://graphene-supermarket.com/images/XC/HC%20Graphite/Flake-Graphite-I-SEM.jpg</u>) (a) Graphene flakes (b) SEM image for graphene flakes




Figure 4.3 Grain size distribution curve for steel slag



Figure 4.4 Steel slag particle size distribution from Dayioglu et al., (2014)







Figure 4.5 Steel Slag Particles

Figure 4.6 Iron Filing Particles

#### **4.2 Sample Preparation**

Acrylic cubes (10 cm x 10 cm x 10 cm) were used as molds for the transparent soil samples. The transparent soil specimens were prepared with laponite powder, sodium pyrophosphate decahydrate (Na4P2O7·10H2O), and distilled water. Wallace and Rutherford (2015) stated that a concentration of 4.5% of laponite obtained the highest strength and transparency. After mixing, the transparent soil specimens were allowed to self-weight consolidate at room temperature for 7-days to gain the strength. Other research by Worthing (2016) reported that a concentration of 12% by mass of silicate powder provide high transparency and strength therefore various concentrations of laponite were prepared to study the undrained shear strength and transparency. In this study, laponite concentrations by mass of 4.5%, 7%, 10%, and 12% were tested. A total of 400 g of water was used to prepare the samples and different weights of laponite powder were added to water prior to mixing. A mixer with three rotating speeds was used for the mixing process. The laponite powder was poured into the water, the mixer was started at the medium rotation speed for 30s and then mixed for another 10s



at the lower rotation speed. A total mixing time of 40 s was used to effectively reduce the air voids and create a uniform transparent soil slurry. However, there were still some small air voids in the transparent soil slurry which may affect the visualization for the further tests. Sodium pyrophosphate decahydrate with an initial concentration of 1.258% by mass of water was added to the slurry over the mixing process to minimize the amount of the air voids in the transparent soil slurry (Killen, 2016). After pouring the mixed transparent soil slurry into the acrylic mold, the mold was first covered with the plastic wrap, and then the lid was placed on top of the wrapped cube to prevent the moisture loss. It could be observed that after the transparent soil slurry was set in the mold for about 20 mins, most of the air bubbles were rose to the surface and disappeared since the sodium pyrophosphate decahydrate increase the dispersal of the laponite powder and allowed for sufficient time for the de-airing process. Figure 4.7 shows the 12% laponite concentration of transparent soil slurry prepared for further testing.



Figure 4.7 Transparent Soil Specimen prepared with 12% laponite Concentration

Different particles types (graphene flakes, steel slag and iron filings) were tested to

investigate the ability to rotate the particles with a magnetic field and see to what extent the



additional material improved the engineering properties of the soil. Higher undrained shear strength was expected for the transparent soil samples solely due to the addition of the particles. To determine the extent of the particle rotation, various laponite concentrations were tested to obtain the optimum undrained shear strength and transparency for tracking the rotation of the particles. Four different laponite concentrations were tested in this research: 4.5%, 7%, 10% and 12% by mass of water. The three particle options were added to the transparent soil specimens by a suspension process. The concentration of the particles added to the transparent soil was also varied, starting at 0.025% by mass of water and laponite powder, and increasing to higher concentrations of 0.075% and 0.1% to compare the specimens' transparency. The concentration of 0.05% and 0% for the magnetic particles were also tested to compare the undrained shear strength difference with 0.025% particles concentration. The particle concentration that obtained the optimum material strength with transparency were used in the particle rotation tests.

## 4.3 Suspension of Particles

Because of the higher specific gravity of the particles compared with laponite, the particles would settle to the bottom of the container therefore a suspension process for the particles was developed. Paton et al. (2014) used a surfactant solution to suspend graphene flakes in water. Dishwashing fluid was used as the surfactant. Three types of the dishwashing fluid were tested in this study. A kitchen blender was used to perform the suspension of the graphene flakes particles in water. The dishwashing fluid shown in Figure 4.8 was used in the particle suspension process because a limited number of bubbles formed during mixing compared with the other two types of dishwashing fluid. The ratio of the dishwashing fluid to graphene flakes concentration was 8 based on recommendations in Paton et al., (2014). In this study, the ratio of 8, 10, 20 were tested to compare effectiveness. A ratio of 8 was able to



provide the suspension for particles by mixing with the kitchen blender, and therefore used for the particle suspension process in the further tests. The blending time applied in this research varied from 10 mins to 30 mins, with the most efficient time that suspended the graphene flakes in water as as15 to 20 minutes. The same blending process was applied for both the steel slags and the iron fillings for particles suspension in water. The blended solution (water and dishwashing fluid) was when mixing the laponite powder and sodium pyrophosphate decahydrate to prepare the transparent soil specimens. Particle suspension is shown in Figure 4.9.



Figure 4.8 Soap with surfactant used for particle suspension (a) Type of soap used (b) Fresh soap surfactant





Figure 4.9 Particle suspension process

## **4.4 Shear Strength Testing**

The prepared transparent soil slurry samples were tested for the undrained shear strength with miniature vane shear laboratory testing equipment as shown in Figure 4.10. The vane is 25mm x 25 mm square with a blade thickness of 0.70 mm. The mini-vane shear tests were conducted at the middle depth among the specimens, a depth of 30 mm below the transparent soil specimens' surface, according to the ASTM D4648. The rotated degree of the vane was recorded and used to calculate the undrained shear strength of the specimen. A total of 56 transparent soil specimens were tested. Figure 4.11 shows the mini-vane testing process in the prepared transparent soil specimens. The concentration of the laponite powder and magnetic particles that obtained the highest undrained shear strength with sufficient transparency was then used in the investigation of applying a magnetic field to rotate the particles.





Figure 4.10 Miniature-vane shear test equipment



Figure 4.11 Miniature-vane Shear Test Procedure

# 4.5 Acrylic Mold Design

Due to the limitation of the available electro-magnetic, small scale acrylic molds were designed to hold the transparent materials with suspended magnetic particles. Three different dimensions of the acrylic molds were designed to investigate the magnetic field efficiency from the controllable electro-magnetic within the transparent soil. A 1/8 inches (0.3175 cm) acrylic sheet was used as the wall of the designed acrylic mold, and the bottom of the molds was closed with the 3/8 inch (0.9525 cm) thick acrylic sheet. The dimensions for the three types of acrylic



molds were 1 cm x 1 cm x 1 cm, 2 cm x 1 cm x 1 cm, and 3 cm x 1 cm x 1 cm. The drawings for the acrylic mold by AutoCAD are shown in Figure 4.12



(a)



(b)





(c)

Figure 4.12 Small scale Acrylic Mold (a) 1 cm x 1 cm x 1 cm; (b) 2 cm x 1 cm x 1 cm; (c) 3 cm x 1 cm x 1 cm

# **4.6 Rotation of Magnetic Particles**

A controllable electro-magnet from the Iowa State University Soft Materials and Structures Lab was used in the study. Seven samples were tested as shown in Table 4.1. A fresh transparent soil slurry specimen, and a self-consolidated specimen were tested each with either a suspension of graphene flakes particles or steel slags particles. Fresh specimens were tested with fine iron filing particles. Magnetic fields were applied to the soil specimens to rotate the particles suspended in the slurry to a desired orientation.



Magnetic particles	Specimen size	Status
type		
Graphene flakes	10 cm x 10 cm x 8 cm	Fresh
Graphene flakes	10 cm x 10 cm x 8 cm	Self-consolidated
Steel slags	10 cm x 10 cm x 8 cm	Fresh
Steel slags	10 cm x 10 cm x 8 cm	Self-consolidated
Iron fillings	1 cm x 1 cm x 1 cm	Fresh
Iron fillings	2 cm x 1 cm x 1 cm	Fresh
Iron fillings	3 cm x 1 cm x 1 cm	Fresh



# **CHAPTER 5. RESULTS AND DISCUSSION**

#### **5.1 BEICP Application for Soil Improvement**

The research objectives of the BEICP study were to: (1) Investigate the use of BEICP to increase the shear strength of silty sand using a spray method for application; (2) Determine the depth of calcite precipitation within a silty sand specimen using a spray method application; and (3) Determine the effectiveness of BEICP to decrease erosion susceptibility of silty sand using a flume.

#### 5.1.1 Results of Surface Strength Tests

#### 5.1.2.1 Strength Tests Before Erosion Testing

The surface undrained shear strength was conducted on BEICP treatment soil column specimens' surfaces prior to the erosion testing. A surface strength of 191.52 kPa (27.78 psi) was measured for the soil column specimen with no BEICP treatment (control specimen). While applied BEICP treatment to the soil column specimens, the undrained shear strength of the BEICP treated specimens showed an increase with the enzyme concentration increase. The specimens treated with the enzyme concentration of C = 1.5 mg/mL reached the highest surface strength of 449.65 kPa (65.22 psi). Figure 5.1 shows the average surface strength before erosion testing against the enzyme concentration increase. As shown in the graph, the undrained shear strength of the specimens treated with a concentration of C = 0 mg/mL and C = 0.3 mg/mL did not have an increase of the surface strength in comparison with higher enzyme concentration of C = 0.7 mg/mL and C = 1.5 mg/mL. The surface strength obtained more than two times of the untreated soil column specimens for the higher concentrations. The error bars indicate the range of the results of the three replicates tested.





Figure 5.1 Before Erosion Surface UCS vs. Enzyme Concentration

#### 5.1.2.2 Strength Tests After Erosion Testing

The surface strength of the specimens after erosion testing was compared for the BEICP treated specimens. The untreated soil column specimens had an initial strength of 151.62 kPa (21.99 psi) and had the largest decrease in strength after erosion testing. The highest surface strength after erosion was 399 kPa (57.87 psi), for the highest enzyme concentration of C = 1.5 mg/mL. Figure 5.2 shows the surface strength after erosion testing for the different enzyme concentrations treated soil column specimens. Specimens treated with C = 0.7 mg/mL and C = 1.5 mg/mL had the highest surface strength after erosion testing, whereas the specimens treated C = 0 mg/mL enzyme concentration and the control of untreated showed no strength increase. The error bars in the figure indicate on the standard deviation of the three replicated tests.

The results for erosion testing of BEICP treated specimens are consistent in comparison with previously published research studies. Kavazanjian and Hamdan (2015) found specimens consisting of mixed and compacted sand columns treated with EICP technology obtained the peak strengths of 529 kPa (76.73 psi) for Ottawa sand, and 391 kPa (56.71 psi) for F-60 sand.



Jiang et al. (2017) found sandy-clay mixtures treated with MICP methods decreased external and internal erosions.



Figure 5.2 After Erosion Surface UCS vs. Enzyme Concentration

### 5.1.2 Results of the Erosion Testing

In this study, the weight of the specimens before and after erosion testing were used to evaluate the erosion susceptibility of the soil. Figure 5.3 shows the average weight loss after erosion testing of the soil specimens treated with different enzyme concentrations. The graph shows that for the untreated soil specimens (control) tested in the water erosion environment, the average weight loss of the three replicates was around 50 grams. The soil specimens treated with the enzyme concentration of C = 1.5 mg/mL only had the average weight loss of around 7 grams. A significant decrease of the average weight loss for the BEICP treated soil column specimens was found compared with the untreated soil specimens. The error bars illustrate for the standard deviation of the three replicates weight loss after erosion. The error bars for the untreated column specimens are much larger than the others, which may due to the position of the replicates in the



water flume. During installation of the specimens in the flume for the erosion testing, a concrete column was placed behind the soil column to stabilize the plywood board in the flume channel. This may have reduced the erosion effect to the soil specimen. For all the enzyme treated soil column specimens, the error bars are smaller than the untreated soil specimens which indicated that there was no obvious variation of the weight loss between the replicates. The results of the weight loss test demonstrated that the soils treated with high enzyme concentrations experienced almost no weight loss while the weight loss increase significantly (from 2.4 % to 15.5%) with a decrease in enzyme concentrations. The results from the surface strength and weight loss after erosion testing illustrates that the BEICP technology could provide effective erosion control for sand-silt mixture soil.



Figure 5.3 After Erosion Weight Loss vs. Enzyme Concentration



### 5.1.3 Results of Depth of Calcite Precipitation Tests

The tests to determine the depth of the calcite precipitation were conducted to evaluate the effectiveness of the BEICP treatment by application using a spraying method. The enzyme concentration of C = 1.5 mg/mL C = 0 mg/mL were compared to a control specimen of untreated soil. The soil specimens were prepared in a mold which allowed each layer to be cut. Because the Scotch Brite sponge was used at the tube end, the bottom layers of the specimens were not tested.

## 5.1.3.1 Layer Strength Test

The soil columns treated with BEICP technology gained significant strength on the surface. Figure 5.4 shows the strength for each layer of the soil column specimens treated with the two types of enzyme concentrations (C = 0 mg/mL and C = 1.5 mg/mL). The top layer of the specimens treated with the enzyme concentration of C = 1.5 mg/mL had the average strength of 383 kPa (55.55 psi), while the soil specimens treated with the enzyme concentration of C = 0 mg/mL had the average strength of 262.5 kPa (38.07 psi). Compared with the untreated soil columns, all the layers had the similar strength of 191.52 kPa (27.78 psi). With application of BEICP technology that the increasing depth into the specimen, the strength increases from the BEICP treatment decreased.







## 5.1.3.2 Calcium Content Testing

The formation of calcium carbonate precipitation from the BEICP technology was the main reason for increase in strength and decrease in erosion susceptibility for the soil specimens. The untreated soil specimens were also tested for calcium content test, since the original silt sieved from Iowa western loess soil contains small amount of calcium content. The results from the untreated soil specimens were subtracted from the results for the BEICP technology treated specimens, which presents the formation of the calcite precipitation. The calcium content tested results for the soil specimen layers shows that the top layer of the specimen gained the highest average calcium content of 1.124% for the soil specimens treated with enzyme concentration of C = 1.5 mg/mL, and 0.776% for the soil specimens treated with enzyme concentration of C = 0 mg/mL. Figure 5.5 shows the calcium content results for each of the four layers treated with the two different enzyme concentrations. For the top layer of the soil specimens, calcium content tested tested had increase significantly for the specimens treated with both enzyme concentrations, and the specimen treated with higher concentration gained more calcium content. From the second



layer, which was lower than 2 cm from the top surface, the tested calcium content continually decreases from 0.24% to 0.136%.



#### Figure 5.5 Layer Calcium Concentration vs. Depth

In several studies published in the literature, the bio-treated soil columns are not uniform, with the calcite content varying with increasing depth (Jiang et al. 2017). The calcite content is also dependent upon the porosity of the material, and maximum calcite content decreased with the distance from injection point increase (Whiffin et al., 2007). A higher strength has also been shown to be a results of a higher calcite content. (Gomez and DeJong, 2007; Qabany and Soga, 2013). As observed in this study, the BEICP treated soil column specimens had an increase of calcite compared to the untreated soil.

### 5.1.4 Results of SEM and EDS

The reaction of the enzyme and chemical solution is the key factor to the formation of the calcite precipitation for BEICP treated soil specimens. The increase of the strength and erosion resistance of the soil is due to the formation of calcite precipitation binding the sand-silt particles. The SEM images and EDS map shows the detail structure between the soil particles,



and the element content for the different depth of layers within the soil specimens. The magnification used for the SEM testing was 300x, and the backscattering electron images were used for the analysis. The SEM images was tested for different depth layers of the BEICP treated specimens, and it showed that there were some calcite precipitation binding especially at the edges of the sand and silt particles of the top layer soil material. The part of the soil material which contains sufficient calcite precipitation binding were chosen for element analysis in the EDS map. From the EDS map for the top layer, it is obvious that there were some calcium element present, which may indicate that there was calcite precipitation binding existing. The large amount of the silicon element in the EDS map represent the existence of Ottawa sand. Because of the spray method applied in the BEICP technology, there were some chlorine spots shown in the EDS map of the top layer material. The area of the chlorine compare with the calcium area on the EDS map was very small which indicated the formation of calcite. The second and fourth layer of the BEICP treated soil specimens were also tested. Figure 5.6 and 5.7 shows the SEM image taken illustrating the calcite precipitation binding between soil particles was not sufficient as the top layer for the second and fourth layer soil materials. The area was tested for the EDS map for the second and fourth layer also show that the presence of the calcium element for the tested soil material were lower than that in the top layer. In the SEM and EDS test, the untreated starting soil were also tested as a comparison. It was shown in the SEM image that there was no obvious calcite precipitation binding the particles, and the presence of calcium in the EDS map is also shown.





Figure 5.6 SEM images and EDS maps for layer 1 and layer 2(a) Layer 1 SEM image; (b) Layer 1 EDS map;(c) Layer 2 SEM image (d) Layer 2 EDS map





(c) (d) Figure 5.7 SEM images and EDS map for layer 4 and starting material (a) Layer 4 SEM image (b) Layer 4 EDS map (c) Starting material SEM image; (d) Starting material EDS map



#### **5.2 Magnetic Particle Rotation Technique for Soil Improvement**

The goals of the magnetic particle rotation were: (1) Design an experimental procedure to suspend magnetic particles (graphene flakes, steel slag and iron fillings) within a soft soil surrogate (Laponite); (2) Investigate the use of graphene flakes, steel slag and iron fillings as an additive to increase the shear strength of a soft soil surrogate (Laponite); and (3) Design a methodology to experimentally test the use of magnetic fields to rotate magnetic particles within a soft soil surrogate (Laponite).

## 5.2.1 Transparency of Specimen

To optimize strength and transparency, laponite specimens were prepared with different laponite concentration and graphene flakes particles. The variation of the graphene flakes particles' concentrations was compared to determine the optimum particle concentration that obtained the highest undrained shear strength with adequate transparency. The transparent soil specimens prepared with the concentration of 4.5%, 7%, 10%, 12% and 14% were compared as shown in Figure 5.8. Because samples that were prepared with the 12% or lower laponite concentration had more transparency than the specimen prepared with the 14% laponite concentration, the 14% laponite concentration was not used in the particle suspend testing. The transparent soil prepared at the laponite concentration of 4.5% by weight had the highest transparency as shown by the visualization of the grid line behind the specimen. As the laponite concentration increased, the specimens were not as clear as the specimen prepared with the 4.5%. The graphene flakes particles were used to determine the particle concentration that provides the optimum transparency within the specimens. The transparent soil specimens were prepared with the 12% laponite powder concentration and suspended with 0.025%, 0.075% and 0.1% graphene flake particles concentrations. Figure 5.9 (a), (b), and (c) show the transparent



soil specimen suspended with 0.1%, 0.075%, and 0.025% graphene flakes concentrations respectively. The specimen prepared with the 0.1% and 0.075% particle concentration had very low transparency and therefore could not be used for magnetic particles rotation.

The lower laponite powder concentration of 4.5%, 7% and 10% were also prepared. The transparent soil specimens prepared with 4.5%, 7% and 10% laponite powder concentration with 0.025% suspended particles were compared. Figure 5.10 (a), (b), and (c) show the 4.5% laponite concentration specimens suspended with (a) graphene flake particles, (b) steel slag, and (c) iron filling particles. Figure 5.11 shows the transparent soil specimens prepared with 7%, 10% and 12% laponite powder suspended with the magnetic particles. The transparent soil specimens prepared with the 4.5%, 7%, and 10% laponite concentrations all provided sufficient transparency for magnetic particles.



(a) 4.5% laponite



(b) 7% laponite





(d) 12% Laponite

(c) 10% laponite



Figure 5.8 Transparent Soil Specimens prepared with (a) 4.5% laponite, (b) 7% laponite, (c) 10% laponite, (d) 12% laponite, and (e) 14% laponite





Figure 5.9 12% Laponite Suspended with different Graphene Flakes concentration: (a) 0.1% graphene flakes; (b) 0.075% graphene flakes; (c) 0.025% graphene flakes



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Figure 5.10 4.5% Transparent soil suspended with magnetic particles (a)Suspended with graphene flakes (b) Suspend with steel slags (c) suspend with iron filings





Figure 5.11 Transparent soil prepared with different laponite concentrations suspended with 0.025% Graphene Flakes: (a) 7% laponite; (b) 10% laponite; (c) 12% laponite



#### **5.2.2 Results from Shear Strength Test**

There were 56 transparent soil specimens tested by mini-vane to determine the undrained shear strength. A total of 14 specimens for each concentration (4.5%, 7%, 10%, and 12%) were prepared. Two of the specimens were mixed with graphene flake particles, two of the specimens were mixed with the steel slags particles, two of the specimens were prepared with the iron filling particles, and two specimens with no magnetic particles. Figure 5.12(a) shows the undrained shear strength for 4.5% laponite concentration mixed with different magnetic particles. The figure indicates that the transparent soil specimens set for 7-day self consolidation process gained the undrained shear strength of around 0.48 kPa (0.069 psi). The addition of magnetic particles significantly increases the undrained shear strength up to 0.83 kPa (0.12 psi). Figure 5.12(b) shows the undrained shear strength for 7% laponite concentration mixed with different magnetic particles. The result shows that the initial transparent soil specimens set for 7days self-consolidation process obtained the undrained shear strength of around 0.7 kPa (0.10 psi) which had significantly increase when the magnetic particles added. The specimens suspended with iron filling particles had an undrained shear strength as high as 1.33 kPa (0.19) psi) with the particle concentration of 0.05%. The specimens suspended with 0.025% particles also obtained undrained shear strength of 1.30 kPa (0.19 psi). Figure 5.13(a) and (b) show the undrained shear strength for the 10% and 12% laponite concentration mixed with different magnetic particles. The results show the trend of the undrained shear strength increase with magnetic particles added, and the specimens suspended with iron filling particles obtained the highest undrained shear strength of 1.44 kPa (0.21 psi) for 10% laponite concentration specimens and 1.51 kPa (0.22 psi) for 12% laponite specimens. When compared with the transparent soil specimens prepared with no magnetic particles added, the addition of magnetic particles



significantly increased the undrained shear strength. It was determined to optimize transparency and strength, a specimen with 10% laponite concentration would be used for the magnetic particle rotation trials.



Figure 5.12 Undrained Shear Strength vs. magnetic particle concentrations. (a) 4.5% laponite (b) 7% laponite



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*Figure 5.13 Undrained Shear Strength vs. Magnetic particle concentrations (a) 10% laponite concentration; (b) 12% laponite concentration.* 



## 5.2.3 Results from Particle Rotation Testing

The particle rotation testing was performed on a fresh transparent soil specimen prepared by 10% laponite powder and suspended with fine iron filling particles at 0.025% concentration. The specimens tested were prepared in the designed small scale acrylic mold. The specimens that were prepared for particle rotation testing are shown in Figure 5.14.

The magnetic field was applied on the two sides of the specimens to rotate the magnetic particles within the transparent soil specimens. The process of the particles' rotation was recorded by a video camera viewed through a microscope. From the microscope, the fine iron filling particles could be observed clearly. Figure 5.15 (a) shows the specimen prepared in the 1cm x 1cm cube before rotation. The iron filling particle (*a*) was used to track the rotation process. Figure 5.15 (b) shows the particle *a* before and after applying the magnetic field. During application of the magnetic field, the particle was recorded rotating back and forth. Figure 5.16 (a) shows the specimen prepared in the  $2\text{cm x 1cm x 1cm cube before rotation test, and the iron filling particle$ *b*was tracked. When the particle*b*was focused, the rotation of the particle*b*was recorded with the same magnetic field applied. Figure 5.16 (b) shows the degree changed for particle*b*after rotation process. The specimen prepared in the <math>3cm x 1cm x 1cm before rotation is shown in Fig 5.1ure7 (a), and the particle *c* was focused during the rotation process. While applying the same magnetic field to rotate the particle *c*, there was some rotation as shown in Figure 5.17 (b), but not as strong as observed for particle *a* and *b*.

The results from the particle rotation test show that the idea of by controlling the magnetic particles' orientation to improve the soil properties, especially for clay material could be achieved with undrained shear strength increase and reduction of air voids. While the



magnetic particles have their own strong magnetic fields, it will be easier to control the orientation by applying proper magnetic field.



Figure 5.14 Transparent Soil Slurry for Rotation Test (a) 1 cm x 1 cm x 1 cm (b) 2 cm x 1 cm x 1 cm (c) 3 cm x 1 cm x 1 cm



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Figure 5.15 Iron filling particles suspended in 1cm x 1cm x 1cm unit specimen (a) before and (b) after rotation





Figure 5.16 Iron filling particles suspended in 2 cm x 1 cm x 1 cm unit specimen (a) before and (b) after rotation





Figure 5.17 Iron filling particles suspended in 3 cm x 1 cm x 1 cm unit specimen (a) before and (b) after rotation



# **CHAPTER 6. CONCLUSION AND RECOMMENDATION**

#### **6.1 Conclusions**

The outcomes of the bacterial enzyme induced calcite precipitation (BEICP) illustrate that the method could be used to help with soil improvement specifically in relation to increased strength and decreased erosion susceptibility. The following outcomes can be summarized:

- The surface strength of soil specimens was effectively increased by applying the BEICP treatment through a spraying application. The BEICP treated specimens had 3 times the strength increase compared with the untreated soil specimens. The formation of the induced calcite precipitation provided the connection between low-plasticity particles, which help to increase the strength at the surface of the specimen.
- BEICP treated soil specimens had a lower percentage soil weight loss in comparison with the untreated soil specimens during water erosion testing.
- The surface of the BEICP treated soil specimens after erosion testing did not have a significant reduction. The BEICP treated soil specimens obtained a similar or even higher strength after erosion while the untreated soil specimens had a reduction of the strength.
- The determination of calcite precipitation with depth testing of the soil specimens showed that the formation of the induced calcite precipitation had a decreased with depth into the specimen from the top surface while using the spray method to apply the BEICP technology.
- The SEM images and EDS map for the various depths of the BEICP treated soil specimens and the untreated starting materials showed the degree of calcite precipitation formation. Both the SEM images and EDS map showed that the top layer of the BEICP treated soil specimen had the highest content of the calcite precipitation.



• The BEICP method could effectively increase the strength of the soil, and also provide an effective reduction of water erosion effect to soil.

The study of using magnetic fields to rotate magnetic particles within a transparent soil obtained the outcomes as listing below:

- The transparent soil prepared with 12% laponite powder obtained an optimum undrained shear strength with sufficient transparency without magnetic particles suspend.
- The suspension of the magnetic particles which included graphene flakes, steel slags, and iron fillings required a surfactant as an agent. The proper mixing time of the surfactant and the ratio 8 of the surfactant to magnetic particles provided the best performance of the particle suspension process.
- The addition of magnetic particles to transparent soil specimens showed a significant strength increase. The undrained shear strength results for the three types of magnetic particles indicated that the addition of the iron fillings particles and steel slag particles obtained higher strength compared with the transparent soil specimens and the graphene flakes.
- The rotation of particles tested in the small scale cubes demonstrated that the iron fillings suspended in the transparent soil slurry could be re-orientated with magnetic fields. As the cube size increased, it was more difficult to rotate the magnetic particles.

## **6.2 Recommendations**

The limitation of the BEICP study is that all the tests were performed in the lab, and the variation of the water conditions from the flume were not considered. The various flow velocity during the erosion test with respect of water velocity in levees or rivers will be considered in future work. Determinations on how to efficiently apply the technology in the field and



construction sites and the BEICP spray method compare with the mixing method would be needed in future research.

The rotation of particles within transparent soil slurry were only successful in small scale cubes. Additional work is needed to increase the size of the specimens. The undrained shear strength for the specimens after particle rotation was not tested due to the small size of the cubes. The magnetic particles used in this study were the fine iron filling particles. Since steel slag is a recycled material it could be considered more environmentally friendly to be used in future work.


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