

INVESTIGATING STRENGTH AND DEFORMATION CHARACTERISTICS
OF MICROBially INDUCED CALCITE PRECIPITATION TREATED
LOOSE SAND

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PRECIPITATION TREATED LOOSE SAND**

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ABSTRACT

INVESTIGATING STRENGTH AND DEFORMATION CHARACTERISTICS OF MICROBIALY INDUCED CALCITE PRECIPITATION TREATED LOOSE SAND

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Microbially induced calcite precipitation (MICP) is a recently proposed novel environmentally friendly ground improvement method that is alternating to conventional ground improvement techniques. MICP has been under intense investigation by researchers from different civil engineering sub-disciplines to mitigate problems related to crack propagation of concrete, insufficient soil strength characteristics, soil erosion, asphalt cracks, etc. This study focuses on the effect of MICP treatment on the strength and volumetric behavior of loose sands by performing isotropically consolidated drained triaxial tests under 100, 200, and 400 kPa effective confining pressures. Various injection schemes (different numbers of cementation solution treatments and cementation solution concentration) were investigated within the concept of this study. MICP treated sands exhibit higher peak deviatoric stress, stiffness, and dilation. An increase in peak shear strength is hindered by the increase in confinement as the ratio of the peak deviatoric stress of treated and untreated sand is lower at higher confinement pressures. Lastly, an increase in treatment numbers and concentration enhanced the abovementioned properties.

Keywords: Microbially Induced Calcite Precipitation, Drained Triaxial Test, Cemented Sand, *Sporosarcina pasteurii*

ÖZ

MİKROBİYOLOJİK TABANLI KALSİYUM KARBONAT ÇÖKELTİLEN GEVŞEK KUMLARIN MUKAVEMET VE DEFORMASYON KARAKTERİSTİKLERİNİN İNCELENMESİ

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Mikrobiyolojik tabanlı kalsiyum karbonat çökeltmesi (MTÇK) son zamanlarda önerilmiş çevre dostu, konvansiyonel zemin iyileştirme tekniklerine alternatif olan zemin iyileştirme metodudur. MTÇK, betonun çatlak yayılımı, yetersiz zemin mukavemeti, zemin erozyonu, asfalt çatlakları, vb. ile ilgili sorunları çözmek için farklı inşaat mühendisliği alt disiplinlerinden araştırmacılar tarafından yoğun bir şekilde araştırılmaktadır. Bu çalışma 100, 200 ve 400 kPa efektif çevre basınçları altında izotropik konsolidasyonlu drenajlı üç eksenli testler yaparak MTÇK uygulanmış gevşek kumların mukavemeti ve hacimsel davranışı üzerindeki etkisine odaklanmaktadır. Bu çalışma kapsamında çeşitli enjeksiyon şemaları (farklı sayıda çimentolaşma solüsyon enjeksiyonu ve çimentolaşma solüsyon konsantrasyonu) incelenmiştir. MTÇK uygulanmış kumlar daha yüksek pik deviatorik gerilme, rijitlik ve genleşme göstermektedir. MTÇK uygulanmış ve uygulanmamış kumun pik deviatorik gerilmelerinin oranı, daha yüksek çevre basınçlarında daha düşük olduğundan, pik kayma mukavemetindeki artış çevre basıncının artmasıyla kısıtlanmıştır. Son olarak, uygulama sayısı ve solüsyon konsantrasyonunun artışı yukarıda verilen özellikleri arttırmıştır.

Anahtar Kelimeler: Mikrobiyolojik Tabanlı Kalsiyum Karbonat Çökeltmesi,
Drenajlı Üç Eksenli Testi, Çimentolaşmış Kum, *Sporosarcina pasteurii*





Dedicated to my family...

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LIST OF ABBREVIATIONS

ABBREVIATIONS

MICP	Microbially Induced Calcium Carbonate Precipitation
BS	Biological Solution
CS	Cementation Solution
ATCC	American Type Culture Collection
PPRC	Polypropylene Random Copolymer
EDS	Energy Dispersive X-ray Spectroscopy
OD ₆₀₀	Optical Density at 600 nm wavelength
SEM	Scanning Electron Microscopy
UCS	Unconfined Compression Strength
CD	Consolidated Drained

CHAPTER 1

INTRODUCTION

1.1 Overview

Increasing population and migration to the cities result in the growth of infrastructural demand and lead to the necessity of construction within infeasible ground conditions. Such conditions may be enhanced by the application of ground improvement techniques, which have been widely accepted and successfully utilized for many decades. However, increasing environmental awareness, the expectations, and most importantly, the regulations force designers and researchers to question the feasibility of existing methods. At that point, the quest for better applications brought about bacteria-based methods considering their advantages. As the bacteria already live in the ground and are an inevitable part of nature, the external interferences are minimized. Bacteria can function even under extreme conditions.

Providing the necessary nutrition for bacteria to the soil, the formation of calcium carbonate takes place inside the pores. Formed calcium carbonate particles are effective in improving strength and deformation characteristics of soils, especially for sands. The whole procedure is known in the literature as microbially induced calcium carbonate precipitation (MICP), in which the method harnesses the bio-geochemical processes to plug calcium carbonate within the soil matrix (Stocks-Fischer et al., 1999). The method utilizes urea hydrolysis induced by ureolytic bacteria to precipitate calcium carbonate in the pores of the soil, which attracts especially geotechnical engineers' attention. Most of the geotechnical engineering problems are due to the inherent or non-inherent nature of the soils, which may have poor strength and deformation characteristics. As MICP might improve these properties it has caught the attention of the geotechnical engineers.

In addition to improving the mechanical properties of soils, the emergence of MICP comes from the need for more environmentally friendly solutions to replace existing ground improvement methods such as injection of micro-fine cement, epoxy, and silicates (DeJong et al., 2006) since artificial grouts have toxic constituents that can affect the quality of groundwater and soil. Moreover, the use of chemical grouts is banned in some countries (Karol, 2003).

In a sustainability-driven age, the importance of new improvement techniques like MICP have become more urgent for geotechnical engineers. Therefore, MICP is under intensive investigation in the geotechnical engineering field. To date, researchers have investigated the precipitation patterns (Gurbuz et al., 2015; Saricicek et al., 2019; Wang et al., 2019; Mujah et al. 2019), the effect of MICP on strength and deformation (DeJong et al., 2006, Feng & Montoya, 2015; Lin et al., 2015; Terzis & Laloui, 2019), hydraulic (Rowshanbakht et al., 2016; Li et al., 2018; Hataf & Baharifard 2019), and thermal (Venuleo et al., 2016; Martinez et al., 2019; Wang et al., 2020) characteristics of sands, mitigation of soil erosion (Jiang et al., 2016; Zomorodian et al., 2019), and liquefaction (Zhiguang et al., 2016; Zhang et al., 2020) problems. The outcomes in the literature are promising and show that MICP can be a viable solution. However, the studies listed above are mostly in the lab scale, which brings the question: “Can MICP be applied on field-scale applications?”. The MICP research has evolved around this question, transformed from element scale to meter-scale columns/tanks (Gomez et al., 2016; Nassar et al., 2018; Graddy et al., 2018) and field scale (van Paassen et al., 2010; Gomez et al., 2013; Terzis et al., 2020) applications. The expected challenges, such as nonhomogeneous precipitation of calcium carbonate, limited depth of influence due to inlet clogging, are still here and address the continuation of intensive research. Therefore, fully understanding the biogeochemical reactions is essential for the advancement of the MICP. Rather than focusing on the element or larger scales, some researchers (Wang et al., 2019; Elmaloglou et al., 2020) focused on the micro-scale investigation of the MICP to see the evaluation of precipitation in the pore scale. Yet, there is still room for understanding the mechanical behavior of MICP

treated sands. Therefore, this study aims to investigate the drained behavior of the treated sands with varying injection numbers and treatment concentrations.

1.2 Research Objective

This study aims to investigate the effect of MICP on loose sands. The main objectives are:

- to outline strength increase by conducting drained triaxial tests
- to observe the change in the mechanical behavior of the loose sands after the MICP treatments
- to examine the effect of different treatment numbers and chemical concentrations on the abovementioned properties
- to quantify the mass of calcium carbonate within the soil matrix and outline its role on these properties

1.3 Scope and Method

The scope of this research is utilizing *Sporasarcina pasteurii* (*S. pasteurii*) as urease positive bacteria to stimulate the necessary biogeochemical reactions to precipitate calcium carbonate, hence improve the mechanical and deformation characteristics of the loose sands. Within the context, the effect of different number of injections and chemical concentrations on these properties are investigated by conducting drained triaxial tests. In addition, precipitated calcium carbonate is quantified by the hydrochloric acid wash technique. Also, scanning electron microscopy images are taken to investigate how micro-scale behavior affected macro-scale behavior.

1.4 Thesis Outline

While this chapter provides the introductory remarks, the remaining of the thesis is structured as follows:

- Chapter 2 includes studies from the MICP literature, especially the ones which includes the effect of MICP on the mechanical properties of the sands
- Chapter 3 describes the procedures to produce MICP treated sand specimens and proceed to macro-scale experiments, quantify the calcium carbonate content. Also, the method for SEM imaging is included.
- Chapter 4 shows the results of the macro-scale tests, includes micro-scale images. The effect of different treatment numbers and chemical concentrations are evaluated in the light of dicussions.
- Chapter 5 includes a summary of the research, concluding remarks, future works.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

MICP is a sustainable and environmentally friendly ground improvement method that harnesses the biogeochemical processes to improve the mechanical characteristics of soils. The method uses the urease enzyme to facilitate a series of chemical reactions to precipitate calcium carbonate crystals at the soil grains' contact points. Urease positive bacteria initiate the urea hydrolysis in pore fluid by its metabolic activity and induce calcium carbonate precipitation in the pore matrix. Stocks-Fischer et al. (1999) made the pioneering study in MICP literature and explained the MICP pathway utilizing *Bacillus pasteurii* (now *Sporosarcina pasteurii*). The individual cell organisms hydrolysis the urea, produce ammonia, and create an alkaline environment in pore fluid. Then, two different chemical reaction initiates at the same time, ammonium and bicarbonate are produced in step 2. The local increase in pH of urea - CaCl_2 medium in the vicinity of the cells enables the precipitation around the organism and crystal growth. The pathway described in the literature is as follows:

1. $\text{CO}(\text{NH}_2)_2 + \text{H}_2\text{O} \rightarrow \text{CO}_2 + 2\text{NH}_3$
2. $2\text{NH}_3 + 2\text{H}_2\text{O} \rightarrow 2\text{NH}_4 + 2\text{OH}^-$
 $\text{CO}_2 + \text{OH}^- \rightarrow \text{HCO}_3^-$
3. $\text{Ca}^{2+} + \text{CO}_3^{2-} + \text{OH}^- \rightarrow \text{CaCO}_3 + \text{H}_2\text{O}$

Various factors can affect the efficiency of the MICP, and these factors should be understood well before designing any experimental program. Therefore, the next section will focus on this issue in detail.

2.2 Factors Affecting MICP

A chain of chemical reactions leads to precipitation of CaCO_3 crystals, as given in the previous subsection. Crystal size, shape, and precipitation patterns have been affected by several factors such as temperature, pH, urease activity, degree of saturation, treatment solution concentration, etc. (Mujah et al. 2017). This section focuses on the factors affecting the MICP and provides the studies from the literature.

2.2.1 Temperature

Temperature is found to have an effect on bacterial activity (Cheng et al. 2016; Omoregie et al. 2017; Sun et al. 2018), growth (Khodadadi & Birsal 2017; Sun et al. 2018), precipitation pattern (Cheng et al. 2014), content (Cheng et al. 2014; Kim et al. 2018), nucleation rate (Cheng et al. 2016) and strength of the MICP treated soils (Keykha et al. 2017).

Kim et al. (2018) studied precipitation effectiveness with varying temperatures of the urea- CaCl_2 solution, and the optimum temperature was observed at 30 °C in terms of the precipitation content.

Sun et al. (2018) investigated the effect of temperature on urease activity, bacterial growth, precipitation rate. Bacterial growth was accelerated with an increase in temperature (but the maximum temperature is 30 °C). The highest urease activity was achieved at 30 °C (temperature during incubation) for *S. pasteurii*, and it was developed in time and remain constant. Urease activity also affected the precipitation rate, which is the highest at 30 °C. Khodadadi & Birsal (2017) observed the highest rate at 50°C (temperature of urea – CaCl_2 source) and 108 cell/ml, respectively. Also, bacterial growth is inhibited at 50°C (temperature during incubation), so calcium carbonate precipitation, too, due to the lack of bacterial cells.

Cheng et al. (2014) set the initial temperature of the sand specimen to 25°C and 50°C. The amount of precipitated calcium carbonate was higher at 50°C than the specimen

treated at 25°C, yet it showed significantly lesser unconfined compression strength. On the contrary, Keykha et al. (2017) showed the strength wasn't significantly affected by the temperature of the specimen (30°C, 40°C, and 50°C). and the highest strength was observed at 40°C. Additionally, Cheng et al. (2014) showed that SEM images revealed a larger size of crystals at the 25°C case than that of 50°C, which can fill the gaps between sand grain and enhance the strength. This finding was also observed at Cheng et al. (2016). This is explained by variation in nucleation rate, in which higher temperature leads to greater nucleation rate that results in smaller precipitated crystals. The small size crystals were also seen in SEM images of 4°C case, even though the lower nucleation rate is achieved at the low temperatures. The precipitation of smaller crystals at 4°C is due to the low relative supersaturation degree due to the low hydrolysis rate.

Omorieg et al. (2017) examined the effect of growth conditions on the urease activity of isolated *Sporosarcina pasteurii* from the caves of Sarawak location. Maximum specific urease activity (mM urea hydrolyzed min⁻¹ OD⁻¹) was highest at incubation temperatures of 25°C and 30°C for four different bacteria.

2.2.2 pH

The pH of the pore fluid is an important factor for the initiation of chemical reactions since CaCO₃ precipitates in an alkaline environment more effectively (Stocks-Fischer et al. 1999). Also, the pH of the growth medium can affect the urease activity of the bacteria. Omorieg et al. (2017) revealed that the control strain (*S. pasteurii* DSM 33) showed the maximum activity at a pH value of 6.5.

Keykha et al. (2017) adjusted the cementation solution pH (5.0, 6.0, 8.0 and 9.0). The higher cementation solution pH led to an increase in strength (Figure 1). Kim et al. (2018) discovered the optimum pH as 7.0 (among 6.0, 7.0, 8.0, 9.0, and 10.0), considering the precipitated mass.

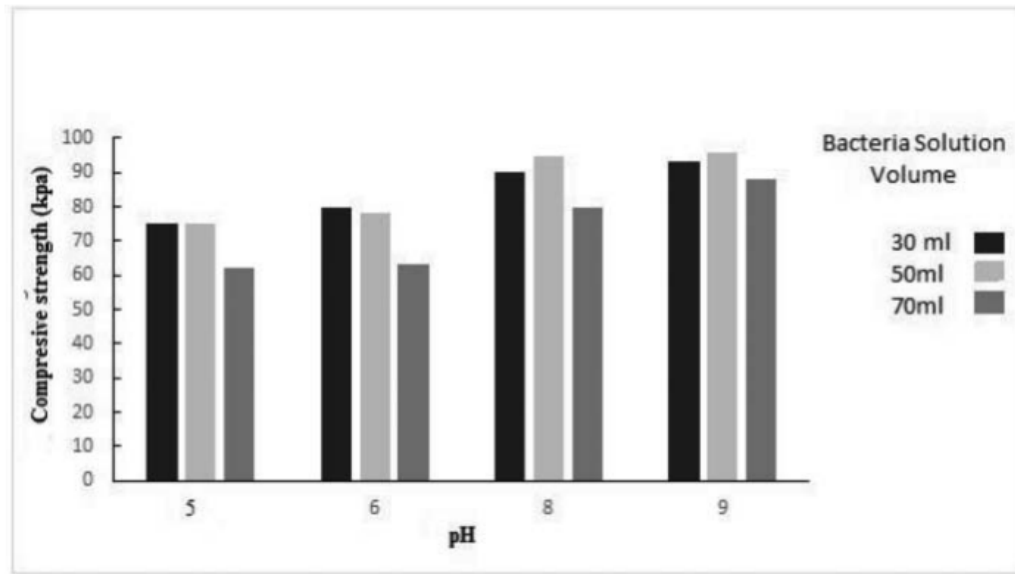


Figure 1 Compressive strength vs. pH, with varying bacteria solution volume (7-day curing) (Keykha et al. 2017)

The cementation medium contains pH stabilizers such as NH_4Cl and NaHCO_3 . Saracho and Haigh (2018) investigated the effectiveness of the stabilizers. 2.12 g/L of NaHCO_3 was enough to stabilize the pH, so NH_4Cl was removed from the recipe.

Cheng et al. (2019) proposed a new injection method that controls the lag phase of CaCO_3 precipitation by adjusting the pH. An injection solution called all-in-one solution (a mixture of cementation solution and bacterial solution) with initial pH 4.0. Having a low solution pH decreased the byproduct ammonium by 90% compared to the conventional MICP treatment method. Chemical conversion efficiency (percentage of urea that led to precipitation of CaCO_3) was not changed the pH values higher than 4.0. It is stated that having lower pH eliminates bio-floc production and provides a stable injection solution. Therefore, a low pH solution is suggested by the authors.

Cheng et al. (2014) adjust the pH of the specimen to 3.5, 9.5, and neutral. Specimens treated at both acidic and alkaline environments exhibited a lesser strength compared to the neutral case, even though the neutral case has the least CaCO_3 mass.

2.2.3 Urease Activity/Bacterial Concentration

MICP process utilizes urea hydrolysis, and urease activity is directly related to the hydrolysis rate of urea. Therefore, it is essential to work at optimal urease activity/bacterial concentration to achieve to enhance the geotechnical properties of the soils.

Cheng et al. (2016) investigated the effect of urease activity on unconfined compression strength of treated soils. For the same CaCO_3 content, the higher urease activity led to lower strength, whereas the lower rate led to the opposite. Cheng et al. (2019) stated that lower urease activity led to more uniform precipitation along the sand column.

Imran et al. (2018) examined crystal growth for an isolated bacteria, *Pararhodobacter* sp., by using a digital microscope under varying bacterial concentrations. The deposition rate, which is the ratio of average volume of crystal particles and time, is increased by an increase in bacterial concentration.

Martinez et al. (2013) worked on the optimization of calcite precipitation. An injection with a microbe concentration of 7×10^5 cells/mL injected at 10 mL/min for 1.5 pore volumes and retention time as 6 h found optimum for the augmentation of bacteria.

2.2.4 Degree of Saturation

The initial degree of saturation of soil is an important factor for calcium carbonate precipitation. Cheng et al. (2013) investigated the unconfined compression strength of the MICP treated sands with the varying initial degree of saturation. Specimens with a lower degree of saturation reached the same strength as the specimens with a higher degree of saturation, with a lesser CaCO_3 percentage. This could be explained by the nutrients forming menisci form in the pore space, which is leading CaCO_3 precipitation at the contact points. However, in a fully saturated case, the crystals can

precipitate in the pore space freely since the pores are filled with nutrients. An illustration is given below.

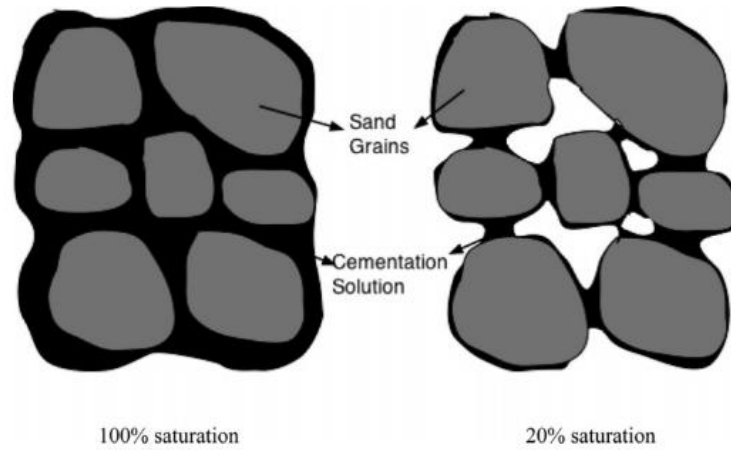


Figure 2 Precipitation patterns for both 100% and 20% saturation

Zeng et al. (2018) examined the MICP treatment on the specimens with different initial degrees of saturation. Specimens with 80 – 70% degree of saturation exhibited a lower unconfined compression strength than 100% degree of saturation. The dry state showed a higher strength compared to a fully saturated case, as the strength increased by lowering the degree of saturation incrementally from 80% to 0% (Figure 3).

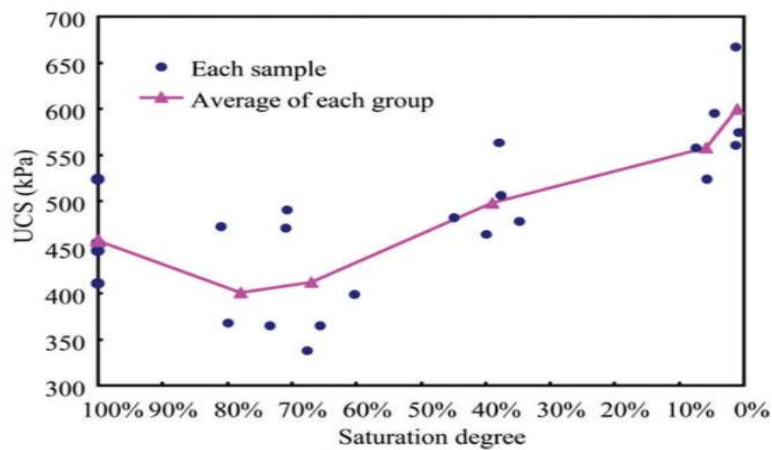


Figure 3 Unconfined compression strength vs. saturation degree for the specimens dried at 60°C (Zeng et al. 2018)

2.2.5 Grain Size and Relative Density

The pore throat and pore size should allow bacteria to move within the pore matrix of a soil freely. Therefore, grain size and relative density can affect calcium carbonate precipitation.

Hataf & Jamali (2018) investigated the effect of fine content on the strength enhancement by the MICP process. Consolidated drained direct shear tests revealed that the fine content hinders the increase in cohesion and angle of friction due to MICP treatments. Rebata-Landa (2007) states that bacterial activity is suppressed, and nutrient percolation is not sufficient in fine-grained soils. Furthermore, Jiang et al. (2016) examined the erosional behavior of MICP treated sand-clay mixtures (with different sand grain sizes) by conducting constant flow rate erosion tests. For the same fine content, having finer sand in the mixture led to no improvement in critical shear stress, yet, it is opposite for the coarser sand-clay mixtures.

Soon et al. (2013) investigated the effect of MICP on the strength of residual soil (sandy silt) and sand specimens with varying relative densities (85%, 90%, 95%). MICP treated residual soil exhibited a higher increase (40-164% increase) in unconfined compression strength compared to sand specimens (14-25% increase). This phenomenon is due to very high relative densities of the sand inhibits the movement of the bacteria freely in the pore space.

Tan et al. (2017) observed the bacteria attachment and permeability reduction in the sand columns with varying grain sizes. The highest reduction in permeability was observed in the finest sand, in which permeability is dropped by 97%. Larger grain sizes led to a poor bacterial attachment due to larger pore volume. Dhami et al. (2016) treated sand columns with a maximum grain size of 0.1, 0.2, 0.5, 0.75, 1, 1.5, and 2 mm and measured the rate of effluent flow within the specimen during the treatment process. The highest drop in the rate was observed in 0.5 mm case in which the rate was dropped from 12.2 ml/min to 1.4 ml/min in 10 days (88% decrease). Larger

grained sand columns exhibited a lower rate of reduction during the initial stages of the injection schedule, compared to finer sands.

Cheshomi & Mansouri (2019) performed direct shear tests on two different sands. Coarser sand (grain size varying between 0.075 – 2 mm) exhibited a peak shear stress of 380.56 kPa at 98 kPa of normal stress, which is 6.2 times the untreated sand's shear strength. However, fine sand showed a lesser increase in peak shear strength, which treated sand peak shear strength is 3.27 times the untreated one's strength. Amarakoon & Kawasaki (2016) treated two types of sands with D_{50} of 0.2 and 0.6 mm. Within the same treatment conditions, coarser sand exhibited higher unconfined compression strength. Nafisi et al. (2020) explained the same phenomenon with a straightforward analogy related to the cementation content and number of contact points. For the same calcium carbonate content, fine sand will have lesser effective calcium carbonate crystals (grain binding crystals) than coarser sand since it has a higher number of contact points. Therefore, the strength gained in fine sand will be lesser than the coarser sand.

2.2.6 Treatment Media

In a theoretical sense, hydrolysis of one mol urea should produce one mol calcium carbonate according to the MICP pathway. But, the MICP process is complex and dependent on various factors, as discussed in previous sections. However, the pathway is indicating that the chemical concentrations of urea and calcium source might affect the process.

Al Qabany et al. (2012) investigated the effect of concentration and retention time on the process. Treatments with equimolar 0.25 M urea- CaCl_2 solution were performed with retention times of 6 h, 12 h, 24 h, and 2 days. In addition to this set of trials, 0.5 M urea- CaCl_2 with 6 and 24 h retention time and 0.1 M urea- CaCl_2 with 3 h retention time were applied as an alternative. The efficiency (ratio of actual precipitated mass and theoretical precipitated CaCO_3 mass) was calculated for each

case. Efficiency was kept at almost 90% when the input rate is lesser than 0.042 mole/litre/hour. Changing the concentrations did not make any difference in the efficiency, as long as the input rate is kept at less than 0.042 mole/litre/hour (Figure 4).

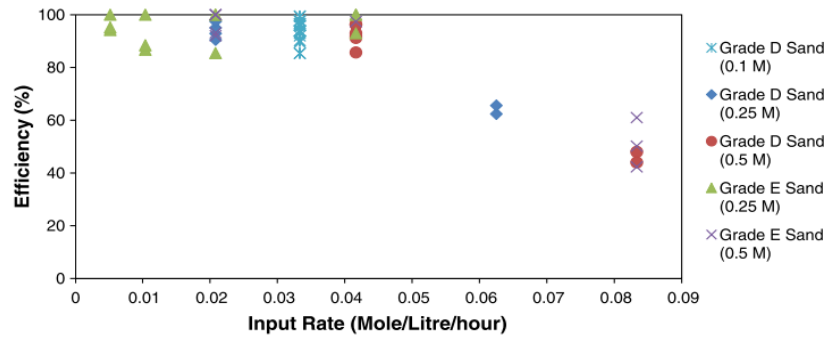


Figure 4 Efficiency vs. input rate (Al Qabany et al. 2012)

Al Qabany & Soga (2013) conducted rigid wall permeability, and unconfined compression test on MICP treated sands. Treatments were done with varying equimolar urea- CaCl_2 (0.1 M, 0.25 M, 0.50 M and 1.0 M) cementation solutions. The higher rate of reduction in permeability was observed due to localized clogging at greater cementation solution concentrations than lower concentrations. Localized clogging led to the inhomogeneous distribution of CaCO_3 , hence low unconfined compression strength. Therefore, a higher number of injections with lower concentrations of cementation solution is suggested.

Wen et al. (2019) investigated the relationship between initial urea concentration and hydrolysis rate. Urea was hydrolyzed for 0.25 and 0.50 M urea in 24 hours, yet only 60% of the 1.0 M urea hydrolyzed during the first 24 h. In terms of the hydrolysis rate, 1.0 M case showed the highest compared to others.

Mortensen et al. (2011) focused on the chemical recipe used in Stocks-Fischer et al. (1999). Higher ammonium chloride concentration (374 mM) led to a lower rate of CaCO_3 precipitation than the 174 mM ammonium chloride case. Having a lower rate is beneficial for achieving uniform CaCO_3 distribution.

Shahrokhi-Shahraki et al. (2015) performed unconfined compression, and falling head tests on MICP treated sands with different urea-CaCl₂ concentrations. Equimolar urea-CaCl₂ concentrations resulted in lower strength and higher permeability than a non-equimolar case (urea to CaCl₂ ratio greater than 1) (Table 1).

Table 1 Some properties of MICP-treated sand A using a bacterial concentration of 1×10^8 cells/ml (Shahrokhi-Shahraki et al. 2015)

Cementation Combination	Urea (M)	CaCl ₂ (M)	UCS (kPa)	Permeability coefficient (m/s)
1	0.10	0.10	50	2.1×10^{-4}
2	0.25	0.25	75	1.8×10^{-4}
3	0.46	0.25	110	1.4×10^{-4}
4	0.25	0.50	80	1.7×10^{-4}
5	1.00	0.50	180	8.1×10^{-5}
6	1.85	1.00	240	2.6×10^{-5}
Untreated	0	0	-	4.2×10^{-4}

2.3 Mechanical Response of MICP Treated Sand

Many researchers investigated the behavior of artificially cemented sands (Dupas & Pecker, 1979; Clough et al., 1981; Consoli et al., 1998; Schnaid et al., 2001). But MICP distinguishes itself from the conventional ground improvement methods since it requires an understanding of bio-geo-chemical processes. Due to the complex nature of MICP, further investigation about the behavior of MICP treated should be done. This section provides the studies done regarding this issue.

Dejong et al. (2006) made the pioneering study in MICP literature and investigated the undrained response of gypsum and MICP treated sand. Both cases exhibited higher q/p'_{cons} than untreated sand. Shear wave velocities were monitored during the

shearing and showed similar behavior for gypsum and MICP treated sands. A rapid drop in shear wave velocity due to cementation degradation was observed in the first 1% axial strain.

Montoya and Dejong (2015) conducted a series of undrained and drained triaxial tests on MICP treated sands to define the stiffness and strength properties of these soils with varying cementation levels (Figure 5). In undrained tests, moderately cemented sands showed greater shear strength and faster peak shear strength mobilization compared to loose sands. However, at large strains, treated sand was showed similar shear strength with loose sands since it exhibits strain softening. As the cementation increased, higher peak shear modulus and strengths were observed at low axial strains. Considering the behavior of moderately treated sand, it showed a dilative behavior. Additionally, the effect of loading path on strength and stiffness properties of the MICP treated sands were investigated in both drained and undrained triaxial tests. Radial extension, constant p , and axial compression cases were investigated, and in drained tests, it has been seen that more brittle behavior was observed when moving from axial compression to radial extension in treated sands. In undrained tests, stress-strain behavior was found similar in each loading case.

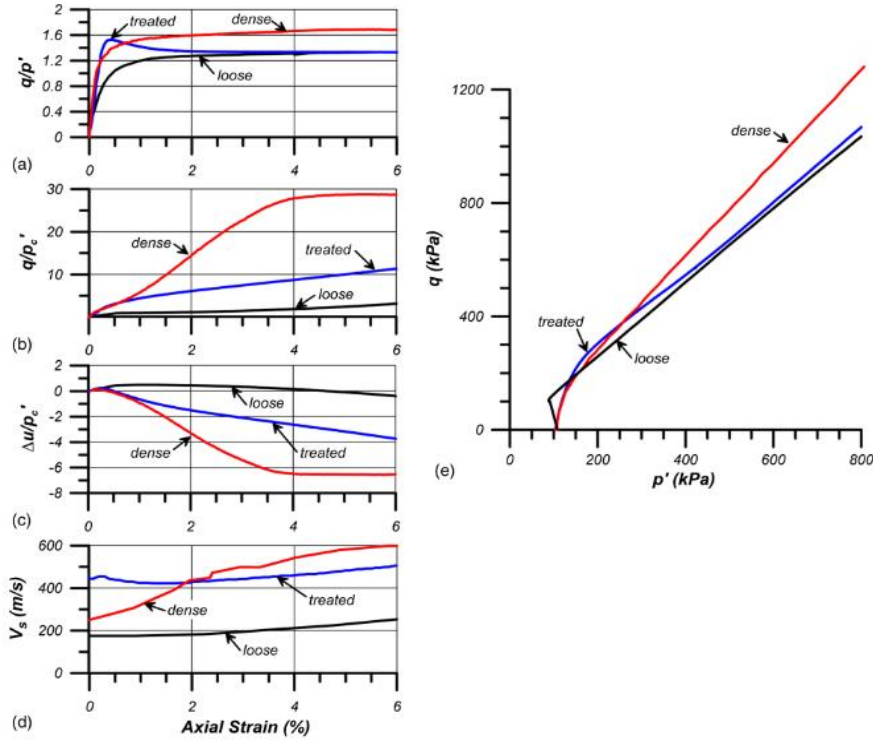


Figure 5 Undrained triaxial compression tests of MICP treated and untreated specimen (Montoya and DeJong 2015)

Cui et al. (2017) were also conducted isotropically consolidated undrained triaxial tests to observe the effect of cementation level on the strength properties of MICP treated sands. A total of 11 groups of loose and dense specimens were treated with different injection numbers, and their average calcite content was determined by the hydrochloric acid washing technique. At relatively high calcite contents, loose sand specimens were showed strain hardening and softening behavior, which implies that specimens became denser and more brittle. After failure, a visible shear plane was observed (Figure 7). However, at relatively low calcite content, this behavior was not found. Additionally, as the calcite content was increased, effective cohesion and angle of friction were increased too. This trend was observed linearly in effective friction, exponential in effective cohesion (Figure 6).

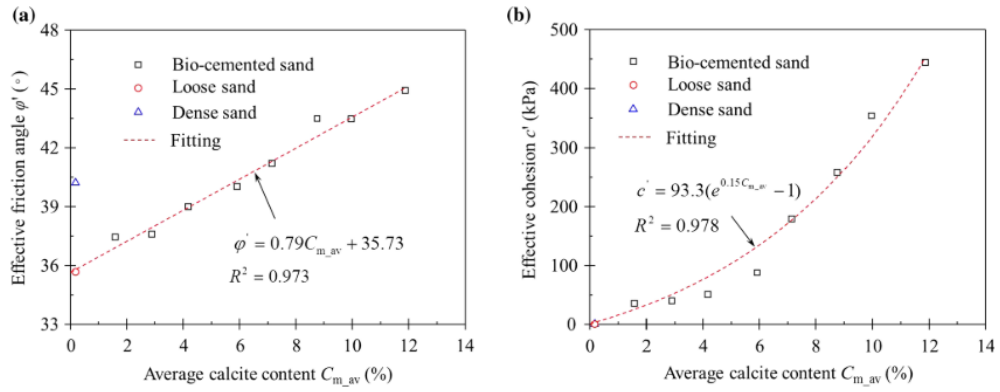


Figure 6 Variation of strength parameters of bio-cemented sand with average calcite content: **a** effective friction angle; **b** effective cohesion (Cui et al. 2017)

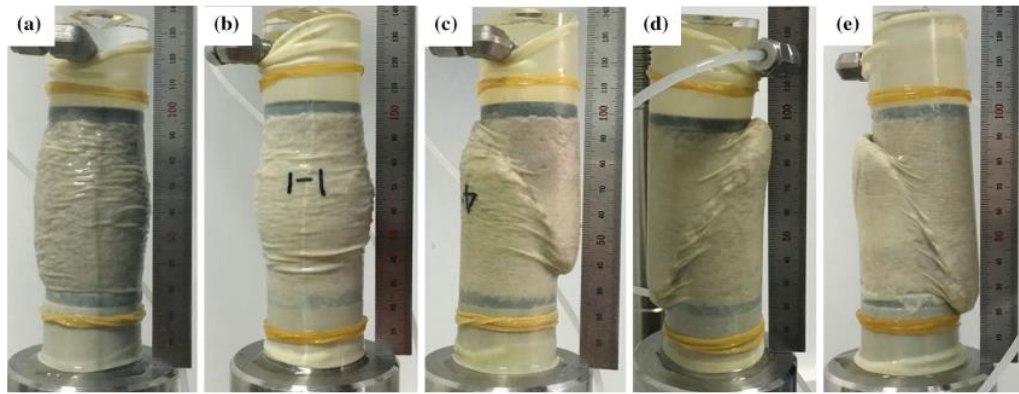


Figure 7 Failed specimens (confining pressure 100 kPa) with different treatment numbers (a) Loose untreated sand (b) N = 2 (c) N = 8 (d) N = 12 (e) N = 16 (Cui et al. 2017)

Cheng et al. (2013) investigated the mechanical behavior of coarse and fine sands under different degrees of saturation by performing consolidated undrained triaxial tests. With an increase in calcium carbonate content, effective friction and cohesion were increased, and at the same calcium carbonate content, the resulting increase was observed higher in a lower degree of saturation for both fine and coarse sands. At a lower degree of saturation, the treatment solution forms a meniscus between grains, which leads to producing calcite bonding at this location. However, at fully saturated conditions, MICP can be produced both between grains and on the surface of the grains. Because of this mechanism, at a lower degree of saturation, it is more likely to have more effective calcium carbonate particles. Additionally, at the same

degree of saturation, fine sand had a lower angle of friction, but higher cohesion since it has more particle contacts compare to coarse sand, meaning that contact stresses are less and more effective calcium carbonate can precipitate (Figure 8 and Figure 9).

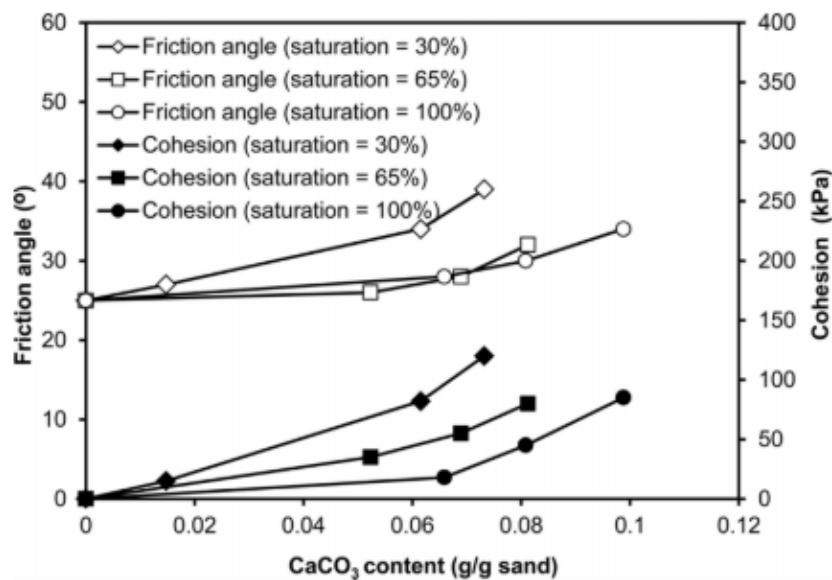


Figure 8 Effect of saturation conditions on shear strength parameters of coarse silica sand having different amounts of CaCO_3 (Cheng et al. 2013)

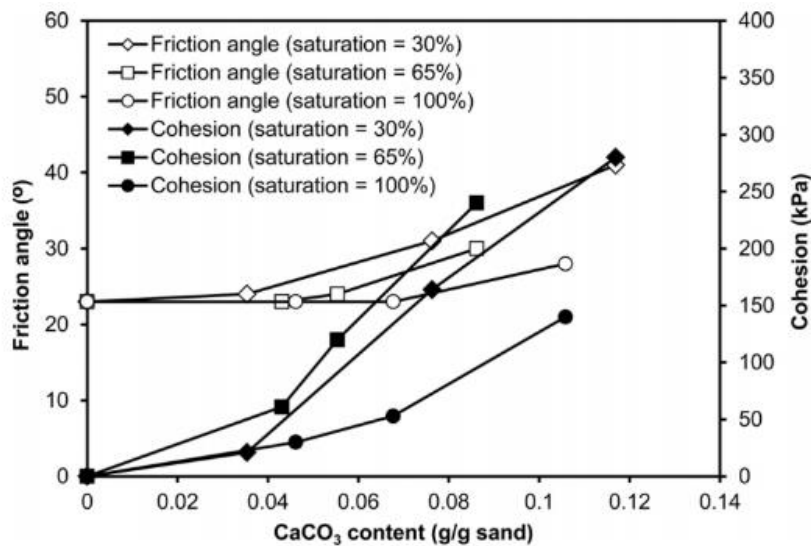


Figure 9 Effect of saturation conditions on shear strength parameters of fine silica sand having different amounts of CaCO_3 (Cheng et al. 2013)

A study by Lin et al. (2016) revealed the drained response of treated Ottawa 20/30 and 50/70 sands under different confinement pressures with injections of different molarities of cementation solution (0.1 M CaCl_2 and 0.3 M CaCl_2). Unlike the untreated sand response, treated sands showed strain-softening behavior for both 0.1 M CaCl_2 and 0.3 M CaCl_2 injection cases, and treated sands showed a more dilative tendency. Furthermore, Poisson's ratio was determined by using P-wave and S-wave velocities. After cementation treatments, Poisson's ratio was decreased, which justifies the trend of moving towards to dilative behavior. Also, after reaching peak strength, deviator stress was decreased to the deviator stress of untreated sand at 10% axial strain for the Ottawa 50/70 sand, 0.1 M CaCl_2 case. However, an increment of 14 to 71% was observed in residual strength for Ottawa 20/30, 0.1 M CaCl_2 case, which can be due to residual cohesion since residual cohesions were observed as 1 and 7 kPa for Ottawa 50/70 and 20/30. In the case of 0.1 M CaCl_2 injection, peak angle of friction and cohesion were obtained as 32° and 31° , 41, and 58 kPa for Ottawa 50/70 and 20/30 sands.

Feng & Montoya (2015) investigated the drained response of MICP treated sands with varying cementation levels (lightly, moderately, and heavily cemented sands) under different confining pressures (100, 200, and 400 kPa). An increase in cementation level led to dilative behavior, yet dense untreated sand exhibited a higher dilatancy than heavily cemented sand. Also, peak deviatoric stress significantly increased for moderately and heavily cemented sands, but lightly cemented sand showed similar strength with untreated sand. The increase in peak strength was suppressed by increasing confining pressure, as the ratio of peak strength of treated and untreated case was decreased with increasing confining pressure. Furthermore, higher cementation levels made the initial tangent modulus to become less sensitive against effective confining pressure.

Gao et al. (2019) performed drained triaxial tests on MICP treated sands under 100 kPa effective confining pressure with varying treatment numbers (1, 2 and 4) and relative densities (30, 50, 70, and 90%). Increase in treatment number led to higher shear strength and dilatancy. Also, MICP has been found to be a more effective

improvement method than densifying the soil by compaction. Increasing the relative density from 30% to 90% increased the peak shear strength by 22%. However, 1, 2, and 4 MICP treatment cycles improved the shear strength by 13%, 36%, and 39%, respectively.

Do et al. (2019) performed drained triaxial tests on treated and untreated sands and looked into the theoretical stress-dilatancy relation proposed by Zhang & Salgado (2010) for non-associated Mohr-Coulomb soils. The results were evaluated in d - η space (dilatancy ratio and stress ratio), which is defined in (1).

$$d = \frac{9(M - \eta) - 3m_c}{9 + M(3 - 2\eta) + m_c} \quad (1)$$

which d is the ratio of plastic volumetric strain rate and plastic deviatoric strain rate ($-\frac{d\epsilon_{vol}}{d\epsilon_q}$), η is stress ratio (q/p'), and M is the slope of the critical state line of untreated sand in q - p' space. m_c is a cohesion factor and defined as:

$$m_c = \frac{6(3 - M)(c/p)^2}{3 - \eta} - \frac{2c(3 - M)}{p} \sqrt{\left(\frac{(3c/p)^2}{3 - \eta}\right) + \frac{3 + 2\eta}{3 - \eta}} \quad (2)$$

The dilatancy ratio for untreated sand is less than 1, indicating that the specimen exhibits contractive behavior. For treated sand, d is starting around 1.4-1.5 during the shearing. A decrease in d is associated with strain hardening behavior, yet, d is increasing when strain-softening behavior was observed (Figure 10).

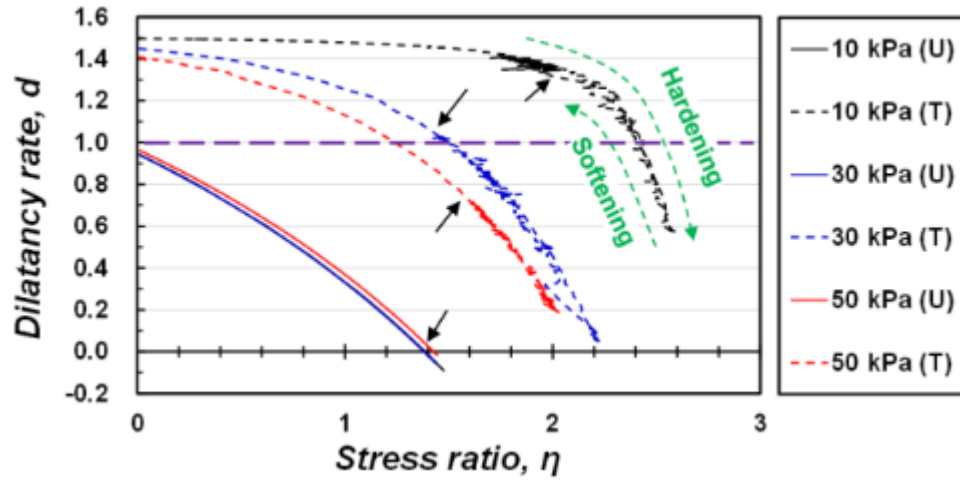


Figure 10 Dilatancy rate vs. stress ratio for treated and untreated sand under varying confining pressures (Do et al. 2019)

Nafisi et al. (2020) conducted drained triaxial tests on MICP treated sands under 10, 100, and 400 kPa confining pressure to assess the failure envelope of these soils. Linear, bilinear, and nonlinear failure envelopes were fitted. At lower confining pressures, the cohesion was overestimated in linear Mohr-Coulomb failure envelope, as the cohesion is 26, 12, and 14 kPa in linear, bilinear, and nonlinear cases, respectively.

Liu et al. (2019) treated the calcareous sands with a different approach. A single cementation solution with varying volume (in terms of pore volume) is recirculated through the specimen three times for achieving 90% chemical conversion. Injections of larger volumes led to an increase in peak shear strength, secant modulus, and faster dilation mobilization.

2.4 Mechanical Behavior of Naturally Cemented Sands

Cemented sand deposits are found all around the world quite often. The cementation process in nature occurs due to the existence of cementing agents such as byproducts of nearby weathering or processes like welding of sand grains at the contact points (materials like volcanic ash exhibits this phenomenon) (Milstone 1985).

Researchers investigated the behavior of this type of soils. O'Rourke & Crespo (1988) took cemented volcanic silty sand specimens from Southern Colombia and performed drained triaxial tests under 0, 60, 120, 200, and 300 kPa confining pressures (Figure 11). The specimens exhibited a significant brittle behavior at lower confining pressure, but with an increase in the pressure, the behavior transformed to ductile. Also, peak deviatoric stress occurred at the axial strain, which exhibited the highest rate of dilation.

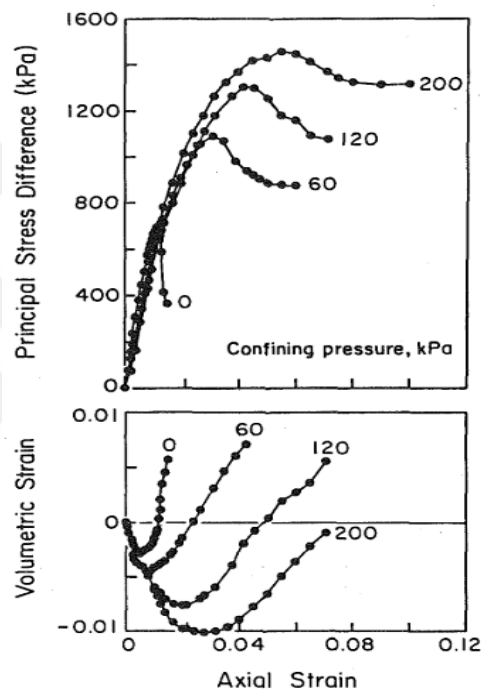


Figure 11 Principal stress difference and volumetric strain versus axial strain, sample 7

Clough et al. (1981) investigated the behavior of weakly, moderately, and strongly cemented sand specimens from San Francisco Peninsula. Drained triaxial tests were performed under 35, 104, 207, and 414 kPa confining pressures (Figure 12). Specimens exhibited dilative behavior at lower pressures, but the behavior was suppressed at the higher pressures. Strongly and moderately cemented sands showed significant strain-softening behavior, but it was not the case for weakly cemented soil. Weakly cemented soil only showed a brittle failure at the unconfined

compression test. Additionally, an increase in confining pressure led to higher peak deviatoric stress and stiffness for naturally cemented sands.

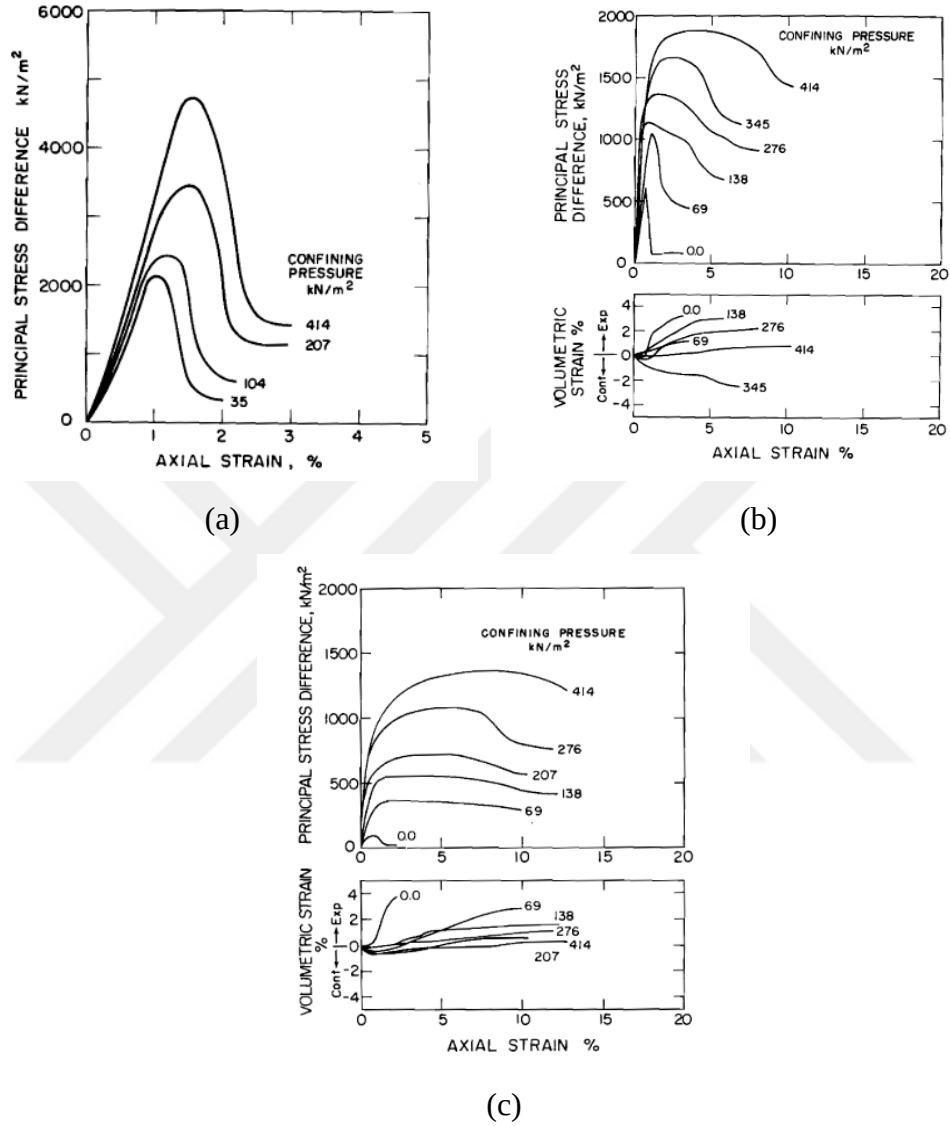


Figure 12 Principal stress difference vs. axial strain and volumetric strain vs. axial strain graphs (a) strongly cemented (volumetric behavior is not available) (b) moderately cemented (c) weakly cemented



CHAPTER 3

MATERIALS AND METHODS

This chapter focuses on describing the materials and methods for biological and cementation solution (BS and CS) preparation, bacteria cultivation, MICP treatments, triaxial testing, and determination of calcium carbonate content of the treated specimen. Some of the methods were adopted from the literature and evolved in a trial and error fashion since there is no exact procedure for macro scale testing of MICP treated sands. The problems and drawbacks of the methods are also discussed and included in this chapter.

3.1 Sand Properties

Fine quartz sand (POMZAEXPORT Mine Industries & Trade Company) was used for this study. The basic properties of this sand were obtained from Ahmadi-Adli (2014), Sariçiçek (2016) and Tunalı (2019). The relevant ASTM standards are ASTM D854 for specific gravity, ASTM D6913 for grain size distribution, ASTM D4254 for minimum and maximum void ratio of soils. Figure 13 and Table 2 illustrates these properties. The motivation behind choosing this sand is fine-grained sands are relatively harder to improve. Nafisi et al. (2020) showed that in order to reach the same cementation level, fine-grained sands require more treatment cycles compared to coarse-grained sands.

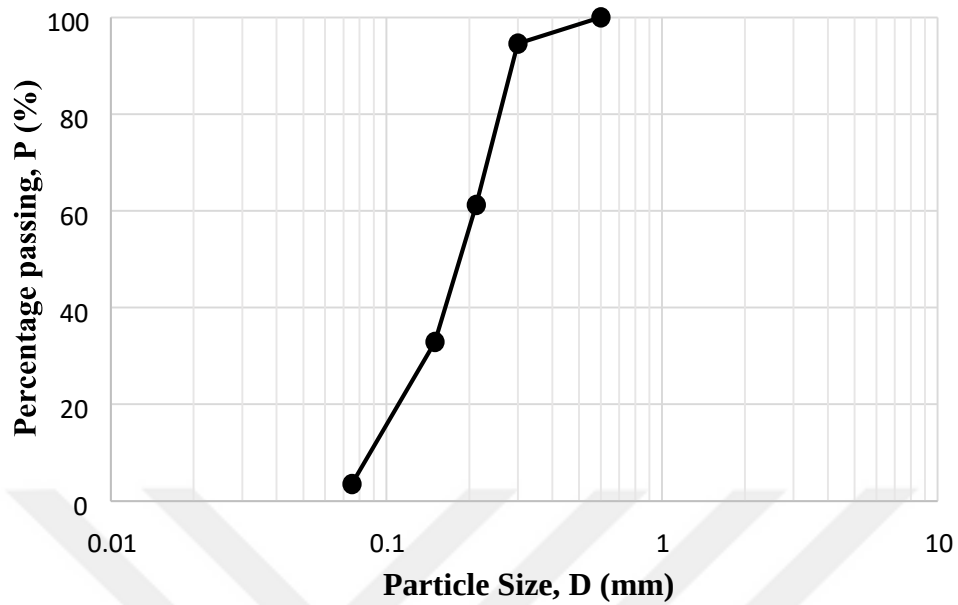


Figure 13 Particle size distribution of sand

Table 2 Sand properties

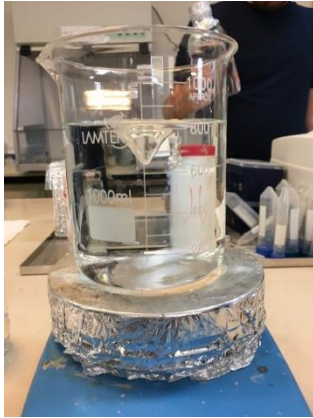
Sand property	Value
D ₆₀ (mm)	0.202
D ₃₀ (mm)	0.14
D ₁₀ (mm)	0.09
C _u	2.24
C _c	1.08
Soil Classification	SP
G.S	2.66
e _{max}	1.00
e _{min}	0.62

3.2 Bacteria Cultivation

Sporosarcina pasteurii (ATCC 11859) was used as urease positive bacteria, which are frequently used in the literature (DeJong et al. 2006; DeJong et al. 2013) due to their high urease activity (Mujah et al. 2017). The bacteria were initially precultivated in ammonium-yeast extract (NH₄-YE) medium (ATCC 1376) at 30 °C with a shaking incubator at 200 rpm, for approximately 24 hours. The growth medium consists of 20 g/L yeast extract, 10 g/L ammonium sulfate [(NH₄)₂SO₄], 0.13 M Tris buffer (pH = 9.0). The medium is prepared as follows (Figure 14):

- 20 g yeast extract and 10 g ammonium sulfate were dissolved in 100 mL distilled water
- 15.75 g Tris buffer was dissolved in 800 mL, and its pH was adjusted to 9.0 by adding 2 M HCl solution
- The ingredients were separately autoclaved for 15 mins at 121°C as ATCC 1376 suggests that no growth occurs when the ingredients are autoclaved together.
- After autoclaving, the ingredients were cooled off and mixed in a sterile environment.

Precultivated bacteria medium was inoculated in fresh growth media (1% v/v), and then the bacteria were recultivated under the same conditions for 24 hours (Figure 15, Figure 16). The final solution was diluted to the optical density (OD₆₀₀) of approximately 1.0 and centrifuged at 4000 g for 15 minutes in 15 mL sterile tubes (Figure 17). Following that, the supernatant was removed (Figure 18), and pellets were stored at -20°C prior to use.



(a)



(b)



(c)

Figure 14 Growth medium preparation (a) tris buffer solution (b) yeast extract and ammonium sulfate (c) mixing the ingredients

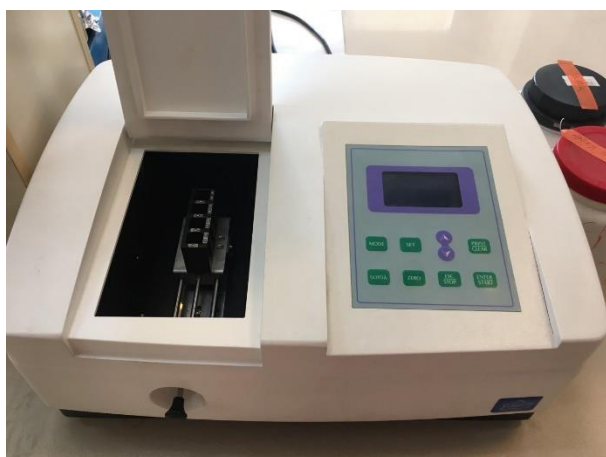


(b)

Figure 15 Bacteria inoculation (a) precultivated medium (b) bacteria inoculation in growth medium



Figure 16 Shaking incubator



(a)



(b)

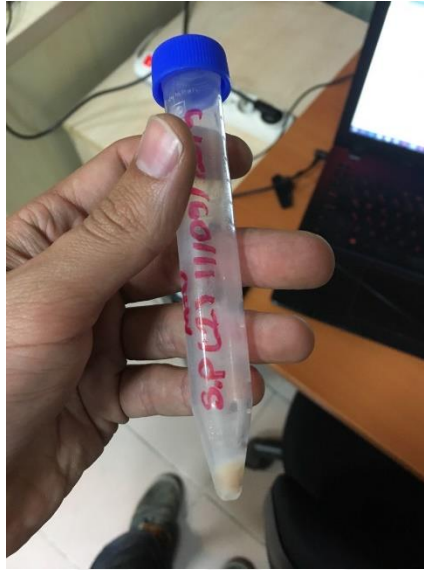
Figure 17 OD₆₀₀ measurement (a) spectrophotometer (UV-5100, SOIF, China) (b) bacterial solution in micro cuvette



(a)



(b)



(c)

Figure 18 Centrifugation the inoculated bacteria (a) Centrifuge (Spectrafug 6C, LABNET, U.S.A.) (b) bacterial pellet and supernatant (c) only pellet after removing the supernatant

3.3 Biological and Cementation Solution Preparation

Biological media constituents, representing the solution used for the introduction of bacteria to the samples, and those for cementation solution are shown in Table 3. The solutions were prepared 2 L at once, and preparation steps (Figure 19) are listed as:

- 120.12/60.06 g urea (Set1/Set2) was dissolved in approximately 300 mL distilled water
- 147.01/73.505 g calcium chloride dihydrate ($\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$) and 20 g ammonium chloride (NH_4Cl) were dissolved in 400 - 500 mL distilled water
- 6 g nutrient broth was dissolved in 200 mL distilled water
- $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, NH_4Cl , and nutrient broth were mixed in 2 L graduated cylinder, and water was added to complete the volume as 1700 mL. The solution was divided into 2 bottles, each containing 850 mL.

- All the constituents were autoclaved together at 121 °C for 15 min, except urea, as it may be decomposed under high temperatures. Instead, urea was sterilized through a 0.22 µm filter.
- Following the end of autoclaving and filtering stages, the components were mixed in bottles under aseptic conditions.

Table 3 Chemical components of biological and cementation media

Components	Biological medium concentrations (M)	Cementation medium concentrations (M)
Urea	1.0/0.50*	1.0/0.50*
CaCl ₂ .2H ₂ O	-	0.5/0.25*
Nutrient Broth	3 g/L	3 g/L
NH ₄ Cl	0.187	0.187

*Urea and CaCl₂.2H₂O concentrations are different for Set1 and Set2. Their concentrations are reported as Set1/Set2.

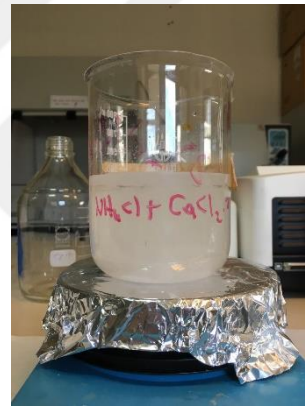
Calcium chloride dihydrate (CaCl₂.2H₂O) was not included in the biological medium in order to avoid the possibility of early precipitation. Furthermore, sodium bicarbonate (NaHCO₃) was not preferred in cementation medium as sodium bicarbonate could react with calcium chloride dihydrate.

Urea – calcium molarities are used differently in the literature. Some of the combinations are 0.25 M – 0.125 M (Gomez et al. 2013), 0.25 M – 0.25 M (Shahrokhi-Shahraki et al. 2014; Zhao et al. 2014; Venuleo et al. 2016; Wen et al. 2019; Terzis & Laloui 2019), 0.333 M – 0.050 M (Mortensen & DeJong 2011; Montoya & DeJong 2015; Feng & Montoya 2016; Zamani & Montoya 2019), 0.333 M – 0.10 M (Lin et al. 2015; Nafisi et al. 2020), 0.350 M – 0.250 M (Gomez et al. 2016), 0.375 M – 0.25 M (Saracho & Haigh 2018; Wang et al. 2019), 0.5 M – 0.5 M (Tsukamoto et al. 2013; Xiao et al. 2019; Gao et al. 2019; Zomorodian et al. 2019) and 1.0 M – 1.0 M (Cheng et al. 2013; Mahawish et al. 2018; Zhang et al. 2020).

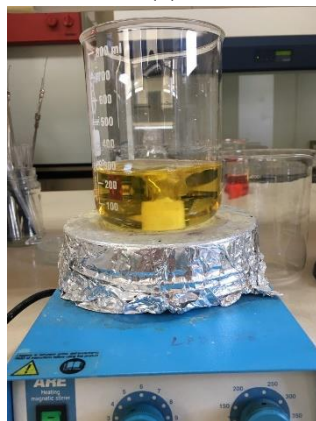
Very low CaCl_2 molarities are not used in this study to achieve rapid improvement. The studies with 0.050 M and 0.100 M CaCl_2 showed that almost 40 injections require to reach heavy cementation levels (Feng & Montoya 2016; Nafisi et al. 2020). Very high ones also disregarded, since it leads to nonuniform calcium carbonate distribution (Al Qabany & Soga 2013). Urea: calcium ratio is kept at two, as Martinez et al. (2013) suggest that it should be higher than unity. Lower ratios will have less ammonium byproduct, which is environmentally beneficial. However, urea consumption is essential to have the necessary biogeochemical processes for MICP. Therefore, the ratio is decided to be 2 to 1.



(a)



(b)



(c)



(d)

Figure 19 Biological and cementation solution preparation (a) urea solution (b) NH_4Cl and $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ solution (c) nutrient broth solution (d) urea filtration

3.4 Specimen Preparation and MICP Treatments

There are different approaches for MICP treatments, and two frequently used methods exist in the literature. The first one is injecting the biological solutions to the specimen through the drainage lines of the triaxial base while the specimen is under a specific confinement pressure (DeJong et al. 2006; Lin et al. 2015; Montoya & DeJong 2015; Feng & Montoya 2015; Feng & Montoya 2017). The second one is injecting biological solutions to specimens that are prepared in PVC columns and under no confinement pressure (Xiao et al. 2018; Cheng et al. 2019; Mahawish et al. 2019; Terzis & Laloui 2019; Liu et al. 2019). The first one is more realistic as it represents real life conditions where the soils are confined by a pressure changing with depth. Both approaches are used in this study and will be discussed in upcoming parts.

3.4.1 Injections within Triaxial Cell

For the study, loose specimens (relative density $\approx 30\%$) with 49.5 ± 0.5 mm diameter and 99.5 ± 0.5 mm height were prepared. Undercompaction method was adopted for specimen preparation, as suggested by Ladd (1978). The method allows the specimen to have uniform density along with the height of the specimen. During the compaction of upper layers with tamping rod, lower layers were also compacted as expected. Yet, in undercompaction, the height of layers was adjusted based on the principle that each layer is going to be compacted equally at the end. In this study, specimens were prepared in 6 layers. Initially, a vacuum was attached to the split mold to provide full contact of the membrane with split mold (Figure 20). After completing compaction, vacuum was utilized for the specimen from the top cap, and split mold is removed. Then, 50 kPa cell pressure was gradually applied while decreasing the vacuum slowly to avoid overconsolidation of the specimen. After applying the cell pressure, the biological solution is injected into the specimen via peristaltic pump (Figure 21a and Figure 21b). Injection rates between 1.33 mL/min

and 20 mL/min were adopted in the literature for triaxial testing. (Dejong et al. 2006; Burbank et al. 2012; Lin et al. 2015; Xiao et al. 2018). Therefore, 3 mL/min was decided for the initial trials. The retention time was 5 hr. After retention, cementation solution injections were made every 6 hours from with 1.3 pore volume (PV) of the specimen. The injection scheme described above is referred to as a two-phase injection (Martinez et al. 2013), and it prevents early precipitation of CaCO_3 . The direction of the flow was changed (bottom to top or top to bottom) at every subsequent injection to achieve more uniform calcium carbonate distribution (Nafisi & Montoya 2018), and the pH of the effluents was measured. As expected, pH values are decreased as time passes, and pH values are shown in Table 4. This might possibly be due to a decrease in bacterial activity and the flushing of the bacteria. After the 8th injection, pH value dropped below 8, as also observed similarly in Feng & Montoya (2016). For keeping the activity high, 2 mL of fresh bacteria was injected at every injection after the 8th one (except the last two injections). Additionally, in this method, only 0.25 M $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ and 0.50 M urea combination was used.

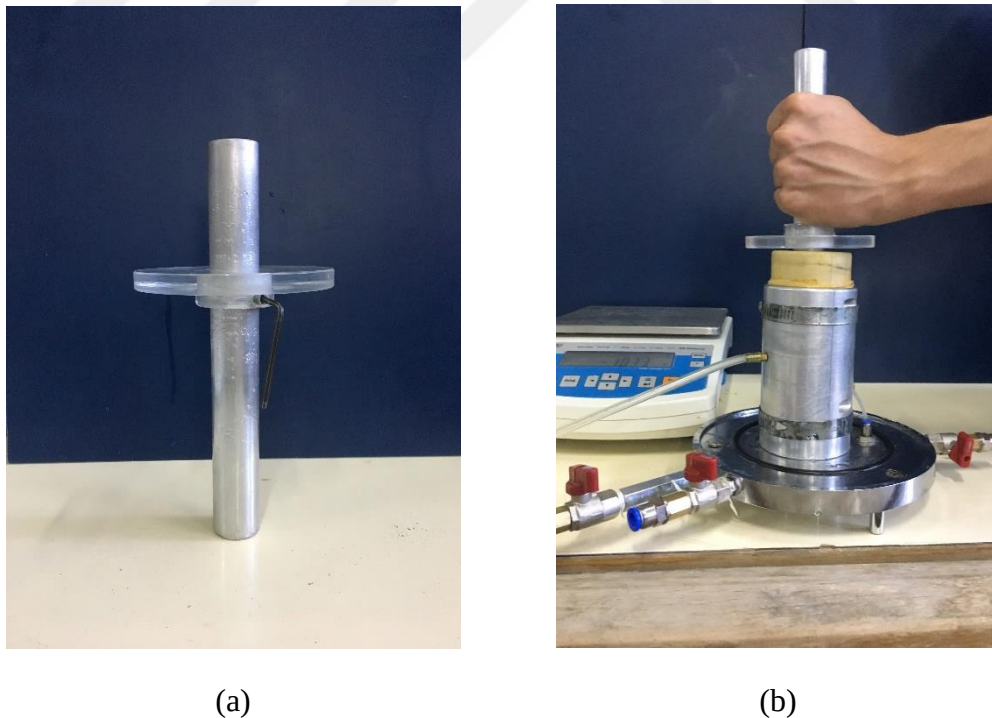


Figure 20 (a) Tamping rod with adjustable height (b) compaction of layers (Tunalı et al. 2019)



(a)

Oil water constant
pressure unit



(b)

Injection
solution

Peristaltic
pump

Effluent collection



(c)

Figure 21 Biological and cementation solution injection (a) side view (b) front view (c) precipitated calcium carbonate in drainage lines

Table 4 Effluent pH values

#CS	Effluent pH	#CS	Effluent pH
1	8.55	15 (2 mL bacteria)	8.02
2	8.52	16 (2 mL bacteria)	8.02
3	8.55	17 (2 mL bacteria)	7.94
4	8.36	18 (2 mL bacteria)	7.93
5	7.98	19 (2 mL bacteria)	8.06
6	8.26	20 (2 mL bacteria)	8.20
7	8.01	21 (2 mL bacteria)	8.21
8	7.70	22 (2 mL bacteria)	8.22
9 (2 mL bacteria)	7.8	23 (2 mL bacteria)	8.21
10 (2 mL bacteria)	8.01	24 (2 mL bacteria)	8.25
11 (2 mL bacteria)	8.18	25 (2 mL bacteria)	8.10
12 (2 mL bacteria)	8.11	26	7.65
13 (2 mL bacteria)	7.90	27	7.79
14 (2 mL bacteria)	7.96		

This method was not followed for the rest of the study for various reasons, and they are listed below.

- It takes approximately one and a half months to complete one test. There was only one triaxial cell available in the METU Geotechnical Laboratory. Unfortunately, it was not possible to prepare multiple specimens at the same time.
- Due to the low injection rate, calcium carbonate precipitation occurred at the drainage lines (Figure 21c) and on the pedestal. The effectiveness of the calcium carbonate precipitation in the specimen was in question, and there was not significant improvement in the specimen. Therefore, an injection scheme in which the injection solutions directly reach the soil matrix was chosen. This problem might be overcome by increasing the injection rate and volume. However, due to the time constraints, these alternatives were not tried and can be taken into consideration for future studies.

3.4.2 Injection within the External Molds

In this method, Polypropylene Random Copolymer (PPRC) split molds with 50.5 mm diameter and 115 mm height were used (Figure 22a, Figure 22b). Specimens with the same dimensions were prepared by using undercompaction method as already described in the previous section. An impermeable plastic sheet was placed inside the mold (Figure 22c), and then filter paper was placed on the plastic sheet to prevent sand from sticking to the sheet (Figure 22d). The plastic was placed in order to prevent leakage from the sides.



(a)



(b)



(c)



(d)

Figure 22 PPRC molds (a) bottom fitting (b) split mold (c) plastic sheet placed inside the mold (d) filter paper placed on the sheet

After the completion of specimen preparation, a granular layer was placed on top of the specimen for filtering purposes as seen in Figure 23a. Injections were made from top to bottom as the solutions were leaked through the granular layer to the specimen (Figure 23b, Figure 23c). Initially, the biological solution was injected with 5 mL/min (increased from 3 mL/min to accelerate the process), 1.3 PV, and

recirculated once. Then, a fixation fluid (0.025 M $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, 1.3 PV) was injected subsequently to increase the adherence of bacteria to the sand grains (Harkes et al. 2010). For cementation solution injections, 8 and 16 injections were made. For the 8 injection case, a biological solution (1.0 PV) was injected after the 4th cementation injection. For the 16 injection case, a biological solution (1.0 PV) was injected after the 8th injection to maintain the bacterial activity. Both injection schedules were applied for Set1 and Set2 and shown in Table 5 and Table 6. Cementation solutions were done in every 12 hours, and the injection rate was lowered to 1 mL/min after 2nd biological solution injection due to the decrease in permeability.



(a)



(b)



(c)

Figure 23 (a) gravel layer (b) injection setup (c) effluent collection

Table 5 8 cementation solution injection schedule

#Injection and Type	Volume and Rate
1. BS	1.3PV – 5 mL/min
Fixation	1.3PV – 5 mL/min
1. CS	1.3PV – 5 mL/min
2. CS	1.3PV – 5 mL/min
3. CS	1.3PV – 5 mL/min
4. CS	1.3PV – 5 mL/min
2. BS	1.0PV – 5 mL/min
5. CS	1.3PV – 1 mL/min
6. CS	1.3PV – 1 mL/min
7. CS	1.3PV – 1 mL/min
8. CS	1.3PV – 1 mL/min

Table 6 16 cementation solution injection schedule

#Injection and Type	Volume and Rate	#Injection and Type	Volume and Rate
1. BS	1.3PV – 5 mL/min	2. BS	1.0PV – 5 mL/min
Fixation	1.3PV – 5 mL/min	9. CS	1.3PV – 1 mL/min
1. CS	1.3PV – 5 mL/min	10. CS	1.3PV – 1 mL/min
2. CS	1.3PV – 5 mL/min	11. CS	1.3PV – 1 mL/min
3. CS	1.3PV – 5 mL/min	12. CS	1.3PV – 1 mL/min
4. CS	1.3PV – 5 mL/min	13. CS	1.3PV – 1 mL/min
5. CS	1.3PV – 5 mL/min	14. CS	1.3PV – 1 mL/min
6. CS	1.3PV – 5 mL/min	15. CS	1.3PV – 1 mL/min
7. CS	1.3PV – 5 mL/min	16. CS	1.3PV – 1 mL/min
8. CS	1.3PV – 5 mL/min		

3.5 Consolidated Drained Triaxial Testing

After completing the injections, three pore volumes of distilled water were flushed. Split mold and the plastic sheet were removed, and the specimen was extracted. For the drained triaxial testing, ASTM D7181 – “Standard Test Method for Consolidated Drained Triaxial Compression Test for Soils” was followed.

The specimen was mounted on the triaxial cell as following the steps shown below (Figure 24a, Figure 24b, Figure 24c, Figure 24d):

- The specimen was oven-dried at 60 °C until there was no change in the mass and placed on the pedestal.
- The membrane was stretched by the membrane stretcher and made full contact with the tube by vacuuming the air with blowing dust ball.
- The membrane was placed around the specimen.

- O-rings were placed on the pedestal, then top porous stone and top cap were placed. O-rings were placed on the top cap.

Upon the completion of the previous steps, the triaxial cell was filled with water, and 30 kPa of all-around pressure was applied. Then, CO₂ and de-aired water were subsequently flushed (Figure 24e). As the last step of the saturation process, the specimen was back pressurized until a B value of 0.95 or higher was achieved. Then the specimen was consolidated under the desired effective stress and sheared at 0.1 mm/min till 15% axial strain.



(a)



(b)



(c)



(d)



(e)

Figure 24 Specimen placement on the pedestal (a) specimen (b) specimen placed on pedestal (c) o-ring stretcher, membrane stretcher, blowing dust ball and flexible membrane (d) membrane placed around the specimen (e) CO₂ flushing

3.6 Gravimetric Acid Washing Technique

The gravimetric acid washing technique is a method that has been used frequently in the literature to quantify the calcium carbonate within the specimen. The specimen was washed and rinsed with 2 M hydrochloric acid (HCl) solution since HCl reacts with calcium carbonate and produces CO₂. Change in mass was assumed as the mass of the calcium carbonate and, CaCO₃ content was calculated as shown below.

$$CaCO_3 \text{ content (\%)} = \frac{(M_2 - M_1)}{M_2}$$

where:

M₂ = dry mass of the specimen after completing the triaxial test

M₁ = dry mass of the specimen after washed and rinsed with HCl

The procedure was done after completing the triaxial test. The failed specimen was generally in two parts; the top part was still intact, nondeformed, and the bottom one

was weak, deformed/destroyed form. The calcium carbonate content of these parts was measured separately. The procedure was done in three steps:

- The failed specimen was oven-dried at 105 °C until there was no change in mass.
- The parts were washed and rinsed with HCl separately in a baker until there was no air bubble produced. The specimen was oven-dried with the same conditions.
- The final mass was measured, and the change in mass was calculated.

In some of the tests, specimens were divided into four pieces, as seen in Figure 25. The first three sections are approximately 2 cm, and section 4 is the remaining of the specimen.



(a)



(b)



(c)

Figure 25 Acid washing (a) before cutting sections (b) after cutting the sections (c) during the acid washing

This chapter presented laboratory works to perform drained triaxial tests on MICP treated sands. Specimen preparation, triaxial testing, and calcium carbonate content determination were successfully done, and the results will be discussed in the next chapter.

CHAPTER 4

RESULTS AND DISCUSSIONS

Quality assessment of a ground improvement application is a vital issue in geotechnical engineering. Laboratory tests can be effectively used for determining the quality of the method, as well as in situ tests. UCS tests have been used frequently in quality assessment of jet grouting, deep soil mixing (His & Yu 2005; Arroyo & Gens 2009; Madhyannapu et al. 2009; Tinoco et al. 2011; Şengör 2011; Toraldo et al. 2016). These are well-established methods and contain rich literature. UCS tests might be well suited for well-established methods since there are many case histories, field data about UCS testing in jet grouting, and deep soil mixing applications. However, MICP is a relatively new method, and the field application is limited. The behavior of MICP treated soils should be understood in depth. UCS tests might be applicable for quality assessment of MICP treated soils, and many researchers followed this approach (van Paassen et al. 2010; Saricicek 2016; Oliveira et al. 2016; Gomez & DeJong 2017; Mahawish et al. 2018; Cheng et al. 2019; Hoang et al. 2020). Yet, for such a new method, a more insightful approach requires understanding the behavior fully. At this point, CD triaxial testing is preferred for investigating the drained response of MICP treated loose sand since a limited number of studies (Lin et al. 2015; Montoya & DeJong 2015; Feng & Montoya 2015; Gao et al. 2018; Liu et al. 2018; Terzis & Laloui 2018; Nafisi et al. 2020) regarding the CD triaxial testing are available in MICP literature. This chapter herein represents the results obtained from the CD triaxial tests, and discuss the effect of MICP on loose quartz sand.

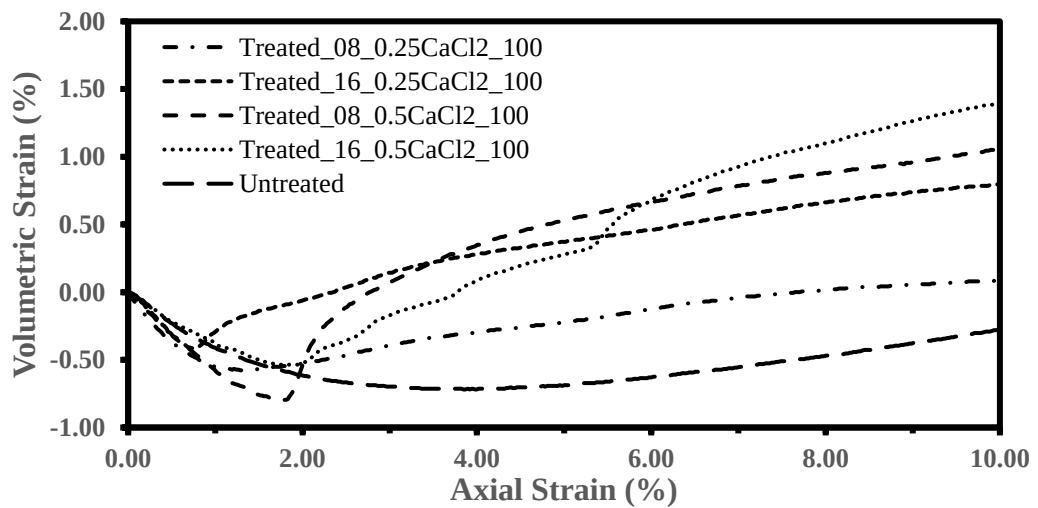
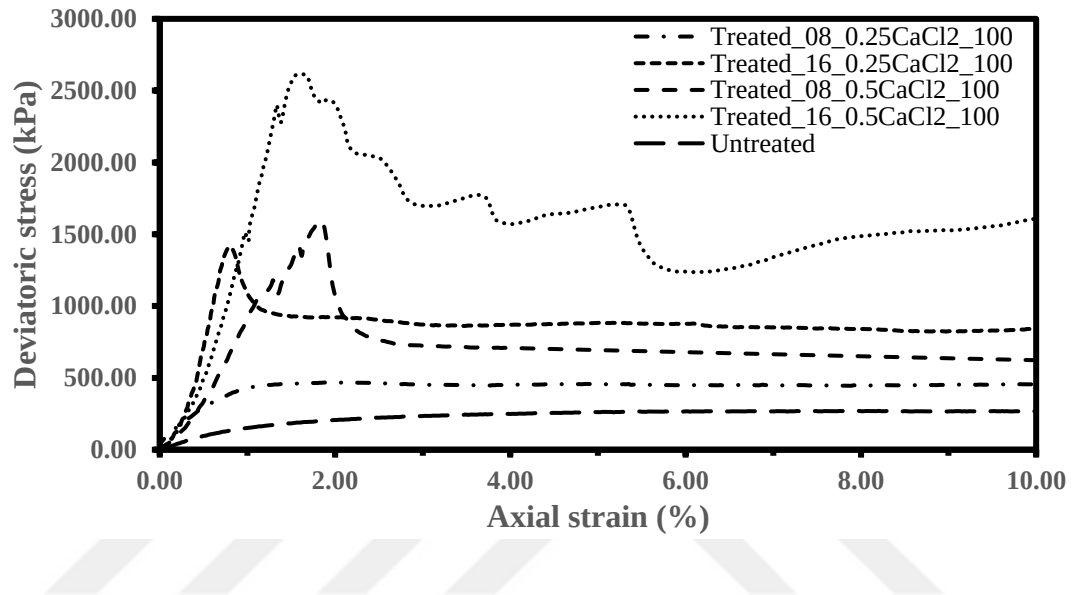
4.1 Drained Response of Untreated and Treated Sands

All of the specimens were sheared under 100, 200, and 400 kPa effective stresses. Untreated sand showed strain hardening behavior in all of the tests. As the effective stress increased, peak deviatoric stress and stiffness were increased, and dilation was suppressed gradually, as expected. Table 7 shows the initial relative densities of the specimens. Post-treatment void ratios are determined by taking $\rho_{CaCO_3} = 1.62 \text{ g/cm}^3$ (Weil et al. 2012). Since the $CaCO_3$ mass was determined in section 4.6, post-treatment void ratios can be determined and seen in Table 7.

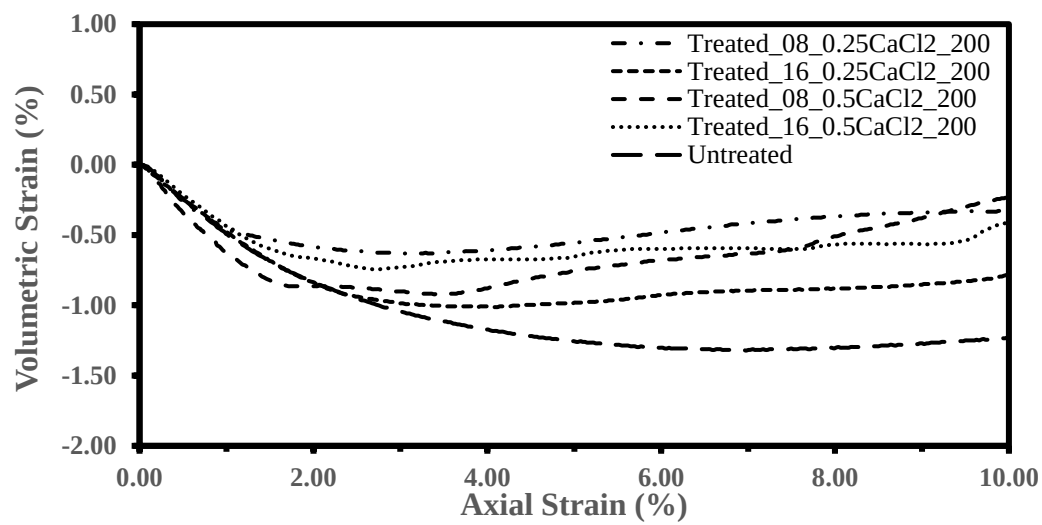
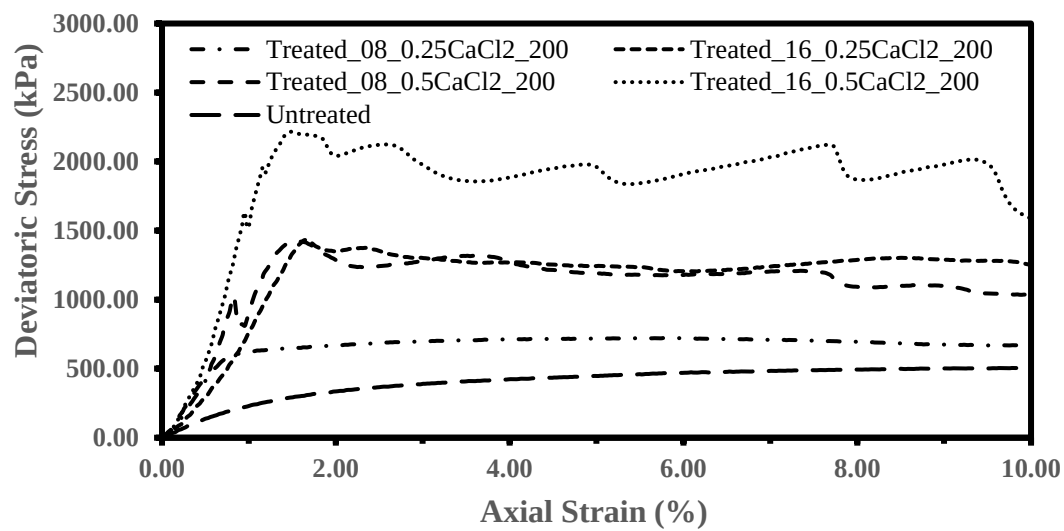
Table 7 Initial relative densities and void ratios

Specimen	Confining Pressure (kPa)	Initial Relative Density (%)	e_{initial}	$e_{\text{post treatment}}$
Untreated	100	27.8	0.894	-
	200	28.5	0.892	-
	400	31.6	0.880	-
0.25 M	100	32.6	0.876	0.783
CaCl ₂ - 8	200	31	0.882	0.793
Injections	400	33.2	0.874	0.781
0.25 M	100	34.3	0.870	0.721
CaCl ₂ - 16	200	33.9	0.871	0.728
Injections	400	31.1	0.882	0.765
0.50 M	100	31.2	0.881	0.746
CaCl ₂ - 8	200	29.2	0.889	0.754
Injections	400	33.3	0.873	0.749
0.50 M	100	32.1	0.878	0.640
CaCl ₂ - 16	200	34.1	0.871	0.655
Injections	400	31.7	0.879	0.711

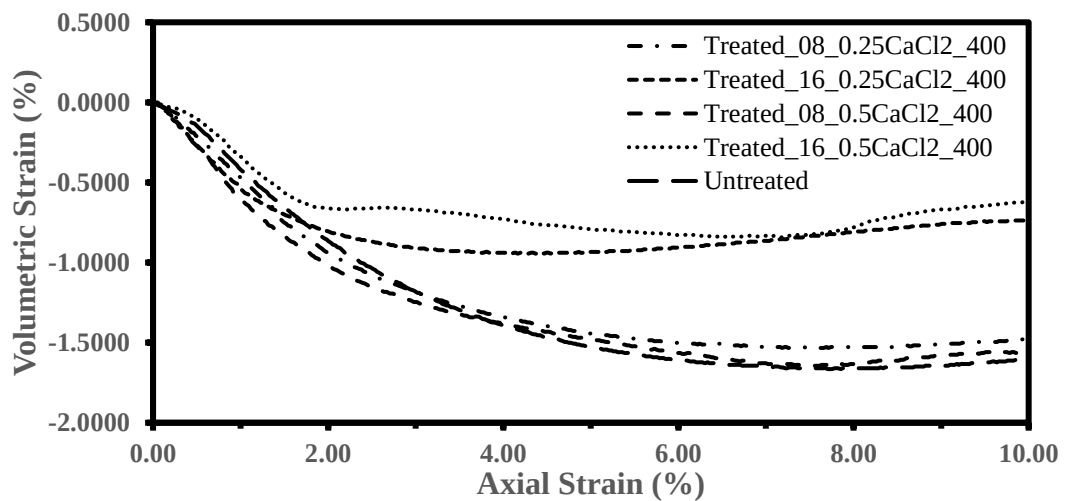
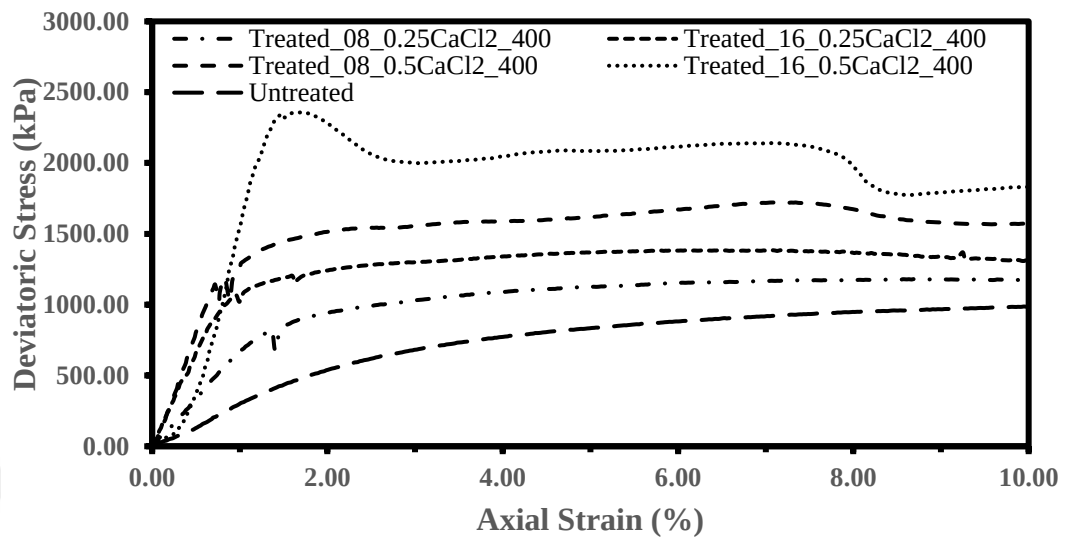
Stress-strain graphs of untreated and treated sands are shown below in Figure 26 for each effective stress case. The MICP treated sands are described as Treated_ #of Injections_CaCl₂ Molar concentration_Confining pressure. The tests were continued till axial strain reached 15%, but the graphs only include 10%.



(a)



(b)



(c)

Figure 26 Stress vs. strain and volumetric behavior of MICP treated and untreated sands under different effective confinement pressures (a) 100 kPa (b) 200 kPa (c) 400 kPa

MICP treated sands showed a significant increase in peak deviatoric stress, as seen in Figure 26. Also, treated sands reached to peak deviatoric stress at lower axial strains compared to untreated sand. However, increment in the confinement pressure

has suppressed the increase in deviatoric stress. Yet, the increase is still evident at the higher effective stresses. Peak deviatoric stresses and ratio of peaks of MICP treated and untreated samples are shown in Table 8 and Figure 27. Deformed shapes also can be seen in from Figure 36 to Figure 49. At the 100 kPa effective confining pressure, strain hardening behavior was followed by a strain softening. Strain softening behavior tends to disappear with the increase in confinement pressure, except the Treated_16_0.5CaCl₂_400 kPa case, as strain softening still exists at 400 kPa confining pressure. For all other cases, strain-softening behavior was gradually decreased with an increase in confinement, which agrees with MICP treated sands of Feng & Montoya (2015) and naturally cemented sands of Clough et al. (1981). Additionally, residual strength (defined as the strength at 10% axial strain) has increased possibly due to increased frictional resistance, since CaCO₃ precipitation increases particle roughness. Furthermore, cemented materials are not purely cohesive or frictional materials. Shear strength of cemented soils contains both frictional, cohesive, and dilatancy components (Zhang & Salgado 2010). The cohesive component degrades during shearing due to bond breaking. Free-flowing CaCO₃ particles densify the soil and enhance the frictional resistance, hence residual strength too. Treated_16_0.5CaCl₂_100 case exhibited a strain-softening behavior after reaching the peak stress. Yet, the deviatoric stress increased after an axial strain of 6%. Increase in frictional resistance might lead to this phenomenon. However, residual cohesion might increase the residual strength, too, as Clough et al. (1981) reported residual cohesions from the drained triaxial tests on artificially cemented sands.

The control specimens (contains no bacteria injections) could not be tested since they cannot withstand its weight. Therefore, they failed during transferring to the pedestal (see Figure 36).

Table 8 Peak deviatoric stresses for each test

Specimen	σ'_3 (kPa)	Peak σ_{dev} (kPa)	Ratio of Peak σ_{dev}
Untreated	100	270.3	-
	200	511.4	-
	400	1024.7	-
0.25 M CaCl ₂ - 8 Injections	100	469.5	1.7
	200	721.9	1.4
	400	1180.1	1.2
0.25 M CaCl ₂ - 16 Injections	100	1412.9	5.2
	200	1432.0	2.8
	400	1384.0	1.4
0.50 M CaCl ₂ - 8 Injections	100	1596.4	5.9
	200	1421.4	2.8
	400	1721.5	1.7
0.50 M CaCl ₂ - 16 Injections	100	2614.7	9.7
	200	2216.4	4.3
	400	2358.3	2.3

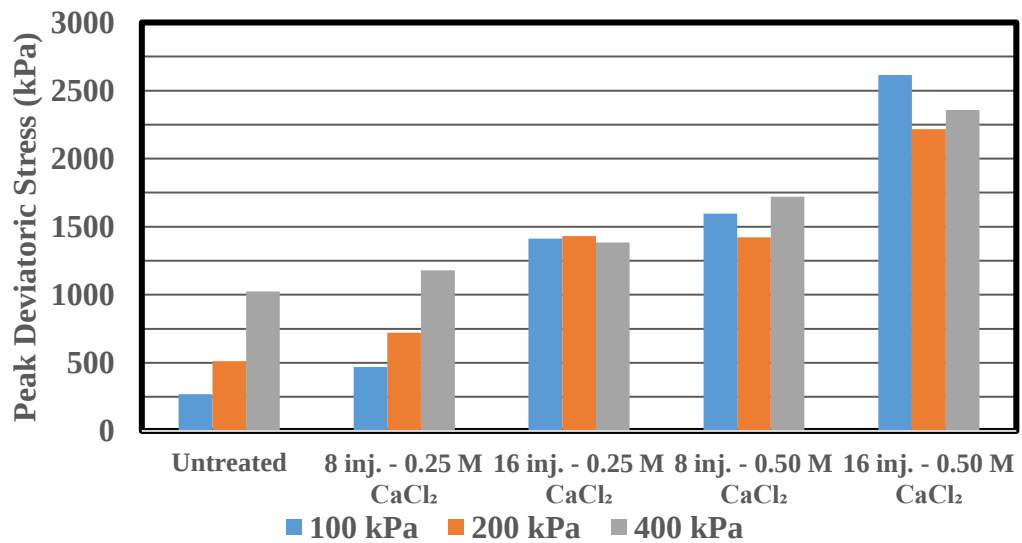


Figure 27 Peak deviatoric stresses for each case at each confining pressure

The volumetric behavior of the sands significantly changed due to MICP treatments, as seen in Figure 26. Positive volumetric strain represents dilation, negative is contraction. Untreated sands mostly exhibit contractive behavior with a very low rate

of dilation at the higher strains. However, MICP treated sands transform into dilative behavior at the lower axial strains, compared to untreated sands. Also, the dilation angle can be determined from the volumetric strain graph, as Vermeer & Borst (1984) idealize the graph bilinearly (Figure 28).

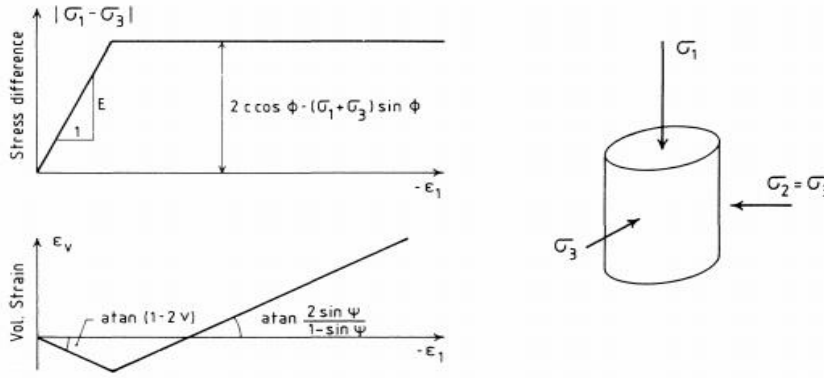


Figure 28 Bilinear idealization of triaxial test results (Vermeer & Borst 1984)

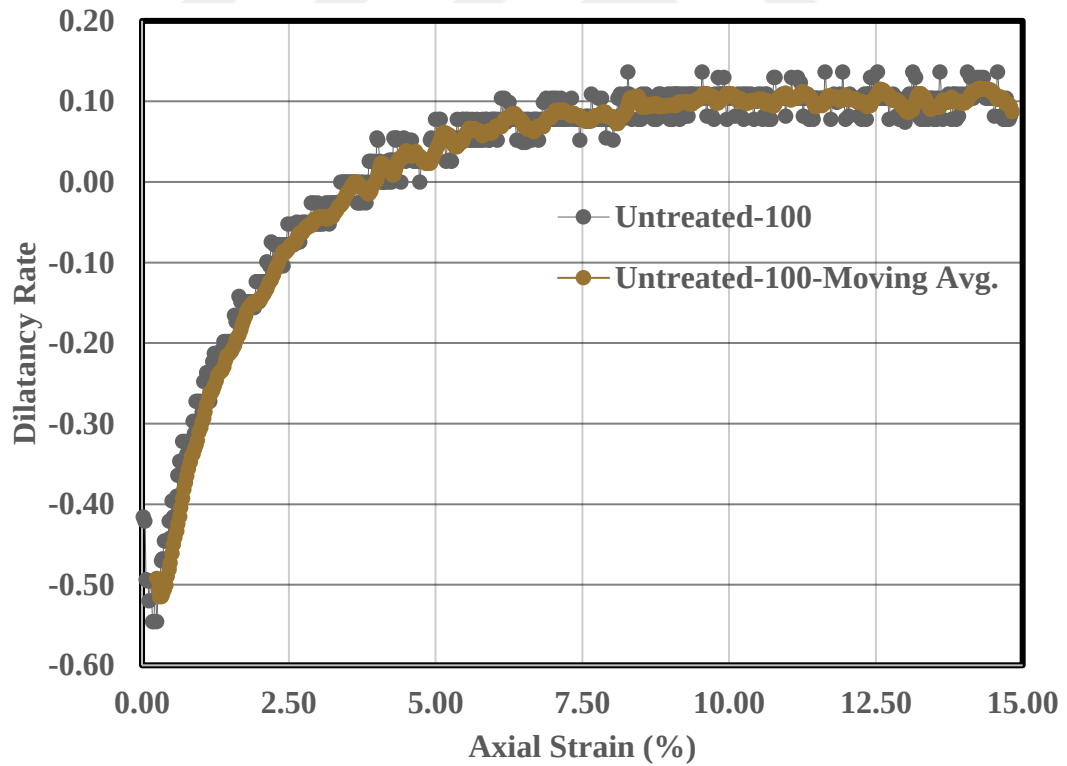
The dilatancy rate is defined as:

$$\frac{d\epsilon_v}{d\epsilon_{Ax}} = \frac{2\sin\psi}{1 - \sin\psi} \quad (3)$$

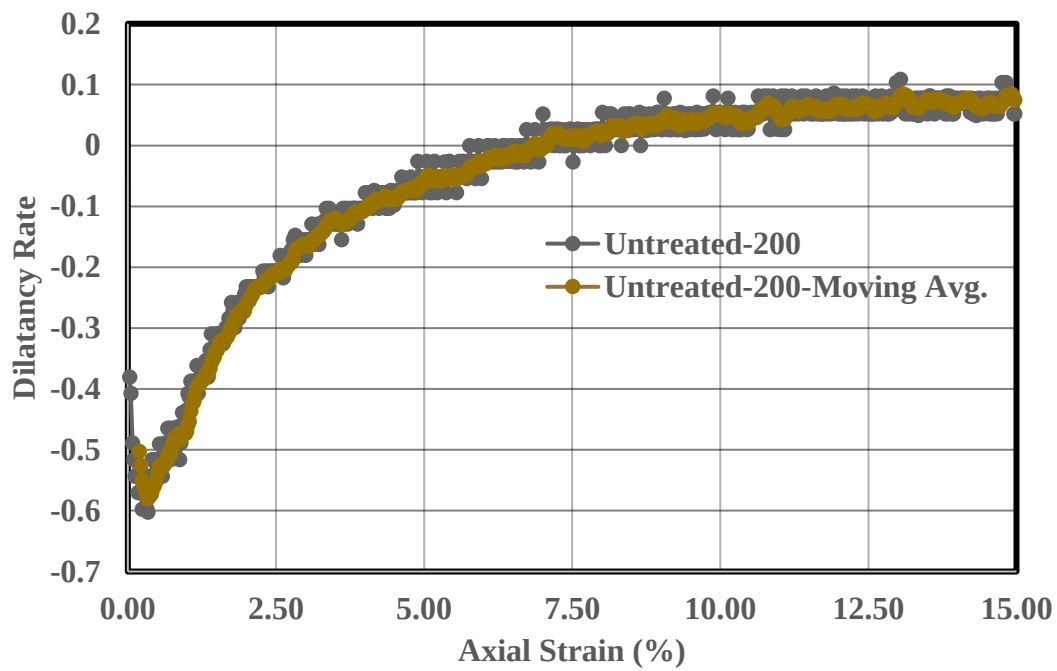
where ψ is the dilatancy angle. From this relation, dilatancy angles were determined for the highest rate, as also determined the same as Maranhã & Maranhã das Neves (2019). The highest rate also occurred at the axial strain which the peak deviatoric stress occurred for the treated 100 kPa tests, except Treated_16_0.25CaCl2_100 and Treated_16_0.50CaCl2_100 tests. This finding is similar to the naturally cemented silty sands of O'Rourke & Crespo (1988), in which the highest rate of dilation occurred at the axial strain of peak deviatoric stress. But, the dilatancy rate at the axial strain corresponding to peak deviatoric stress was still used for these tests. For other confining pressures, the highest rate for chosen at any axial strain. The graphs for the dilatancy rate vs. axial strain can be seen in Figure 29, Figure 30, Figure 31, Figure 32, and Figure 33. A moving average procedure was applied to have a smoother fit as also done in Maranhã & Maranhã das Neves (2019). Dilatancy angles for each test can be seen in Table 9.

Table 9 Dilatancy angle for each test

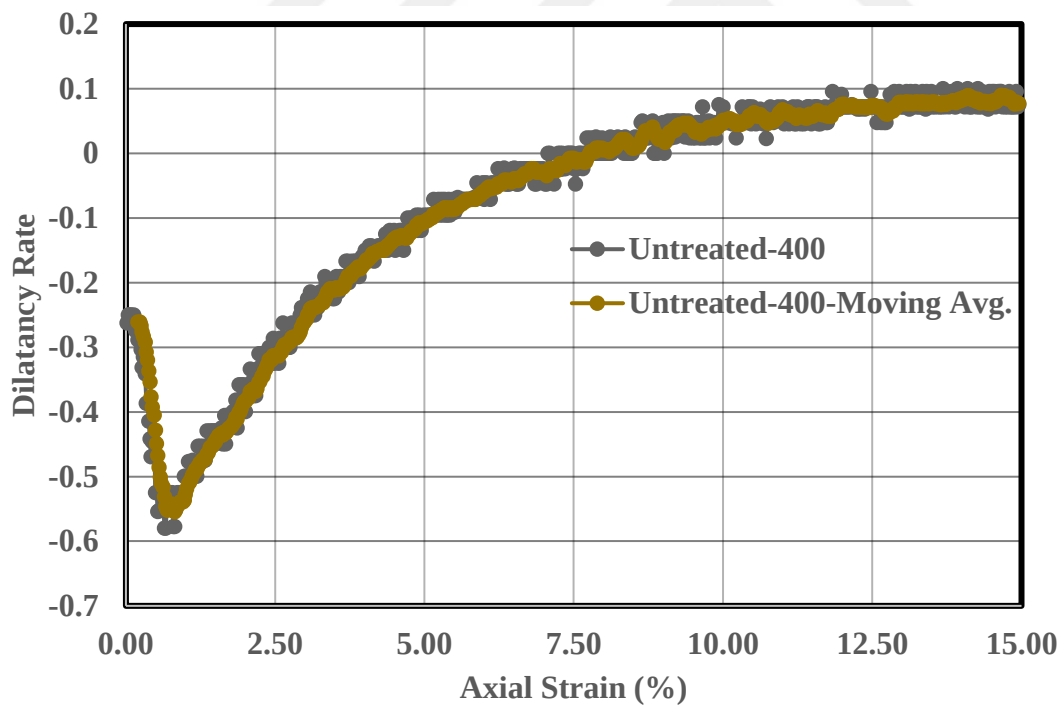
Specimen	σ'_3 (kPa)	Dilatancy Angle (°)
Untreated	100	3.2
	200	2.3
	400	2.3
0.25 M CaCl_2 - 8 Injections	100	4.0
	200	2.2
	400	1.8
0.25 M CaCl_2 - 16 Injections	100	10.7
	200	6.0
	400	1.6
0.50 M CaCl_2 - 8 Injections	100	23.6
	200	5.4
	400	1.7
0.50 M CaCl_2 - 16 Injections	100	10.6
	200	7.7
	400	4.5



(a)

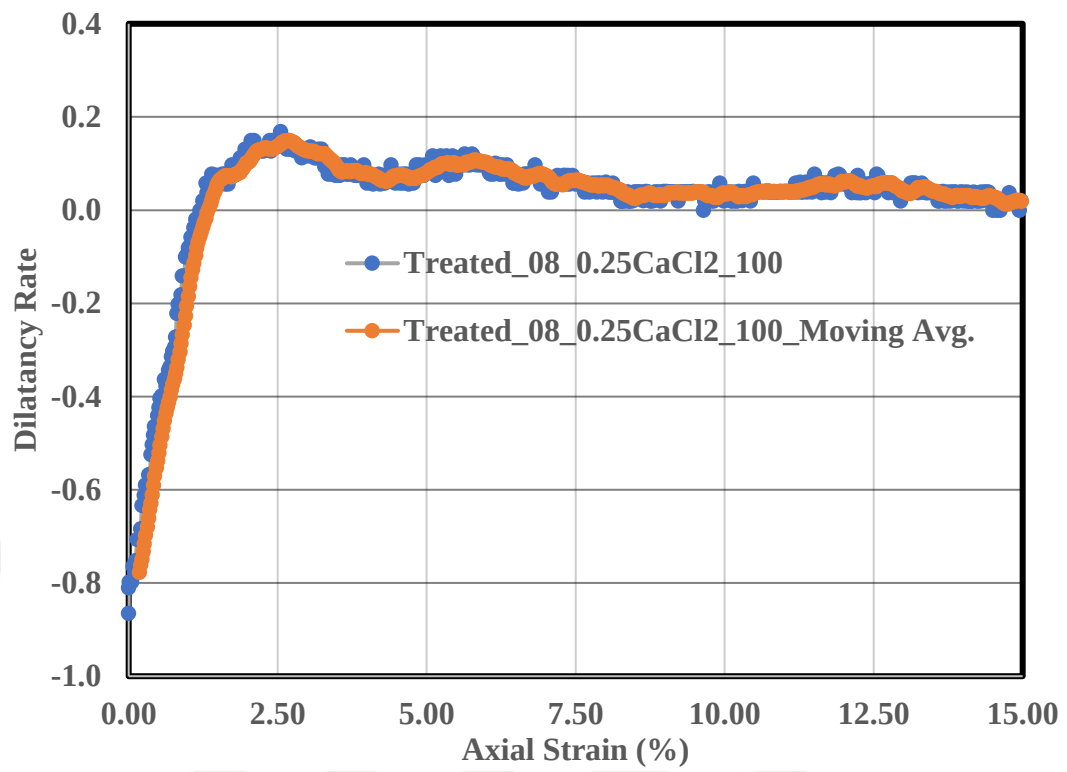


(b)

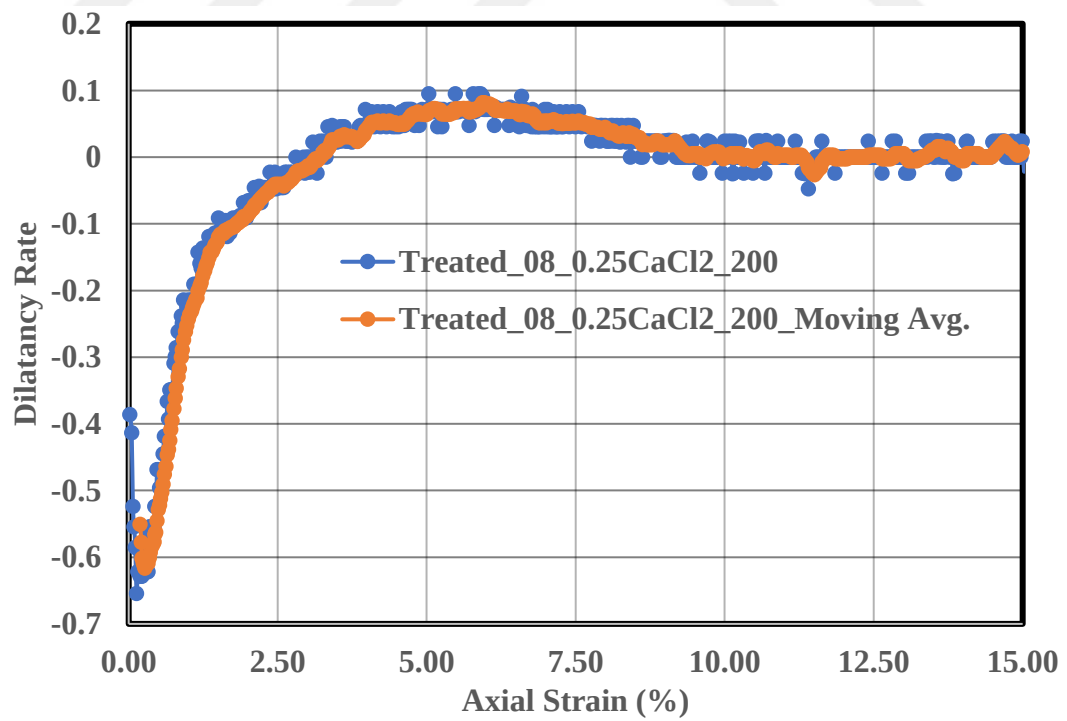


(c)

Figure 29 Dilatancy rate vs. axial strain (%) for untreated case (a) 100 kPa (b) 200 kPa (c) 400 kPa



(a)



(b)

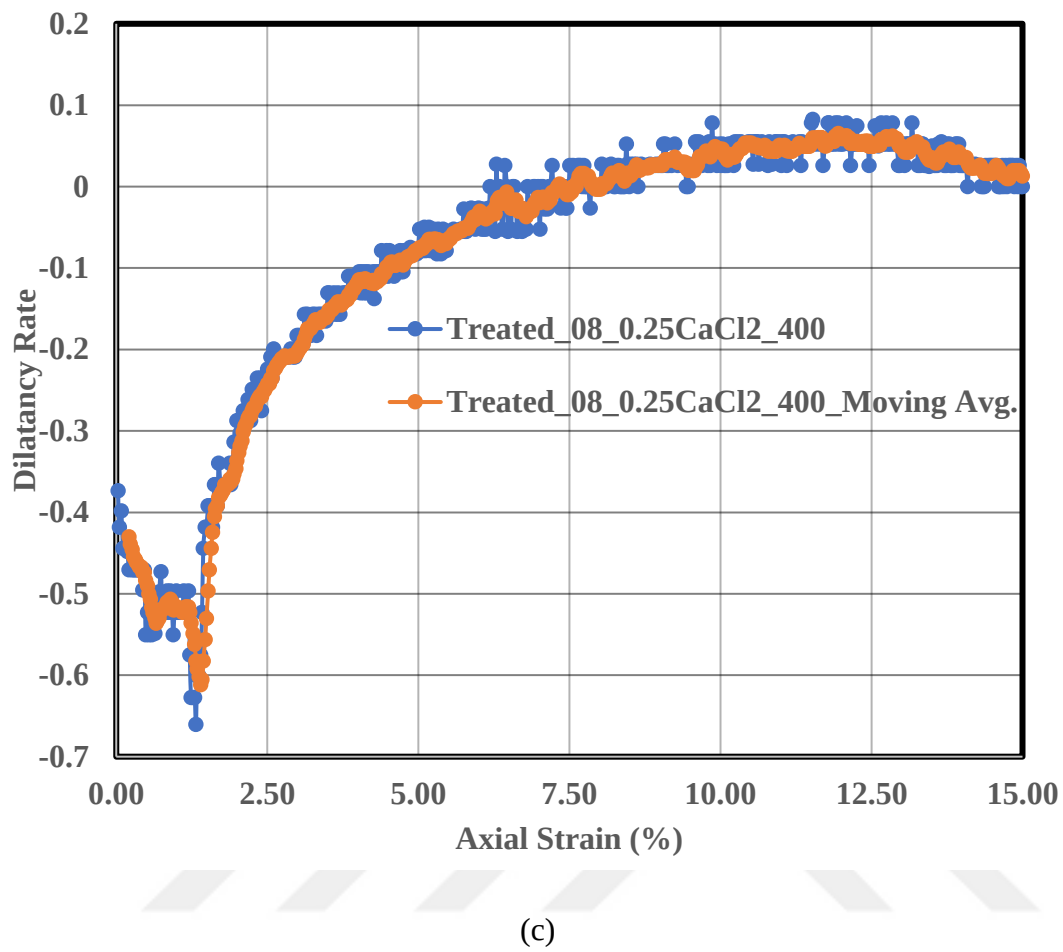
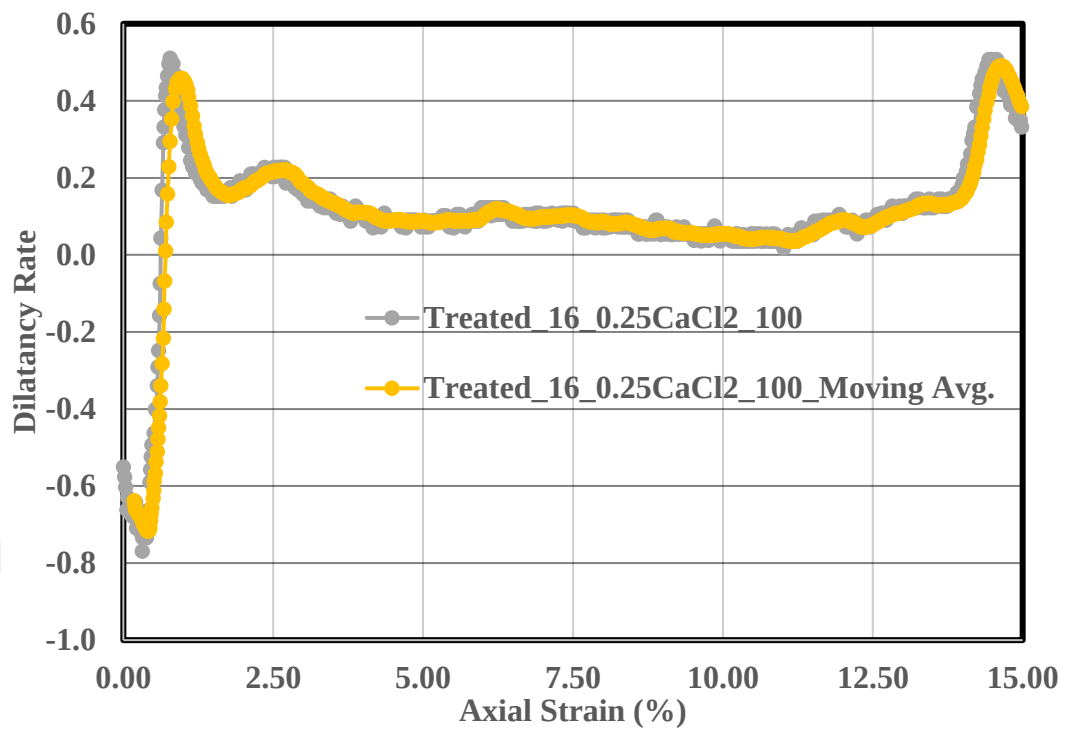
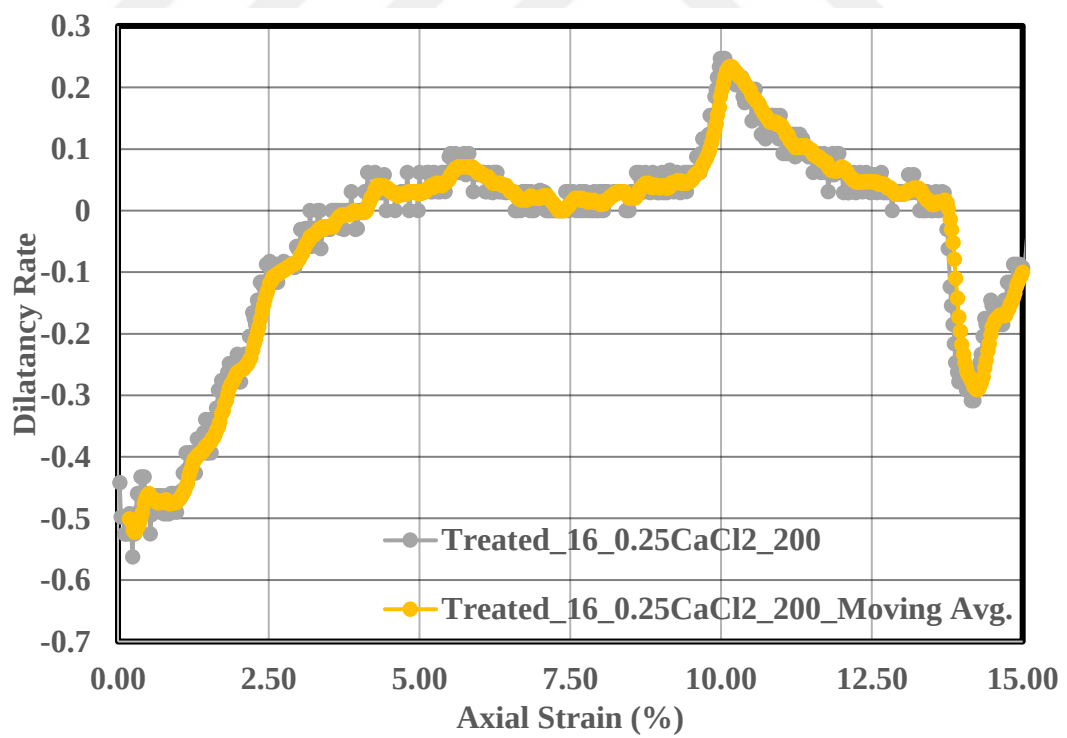


Figure 30 Dilatancy rate vs. axial strain (%) for 8 injections - 0.25 M CaCl_2 case
 (a) 100 kPa (b) 200 kPa (c) 400 kPa



(a)



(b)

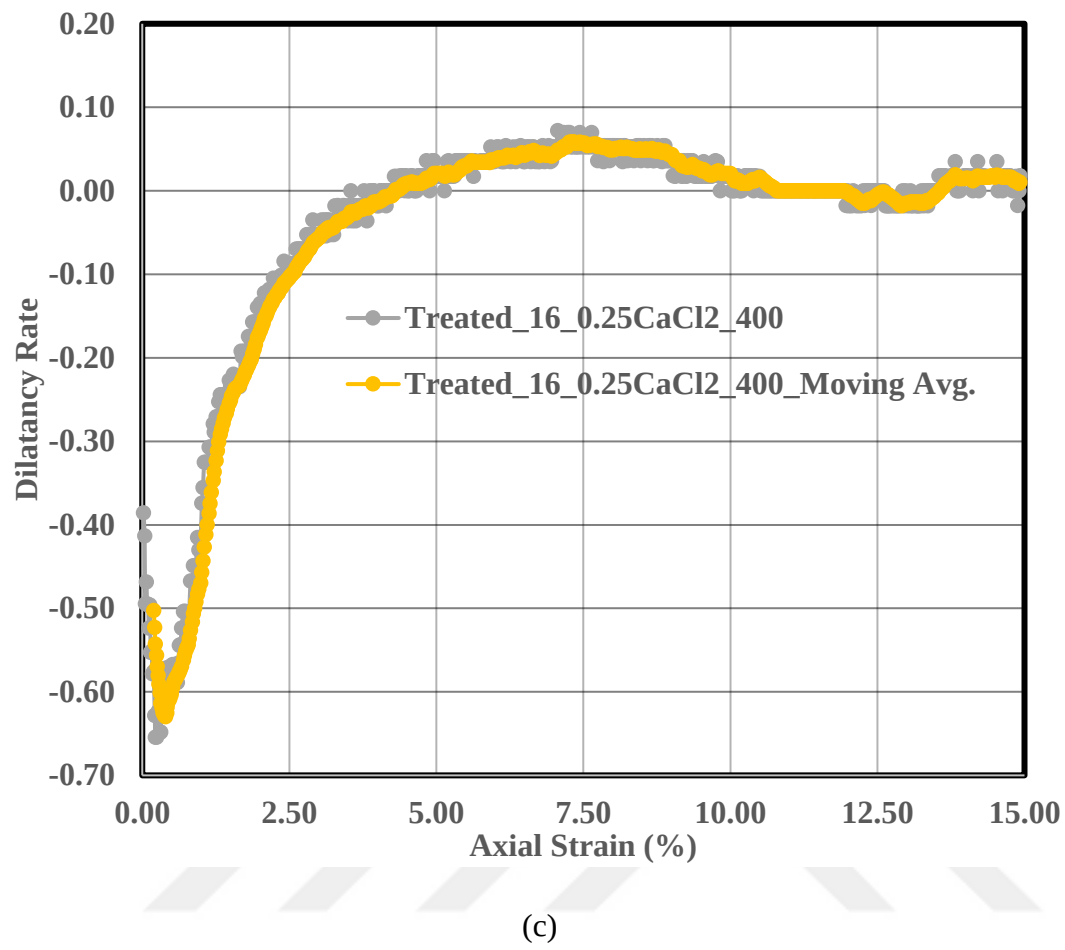
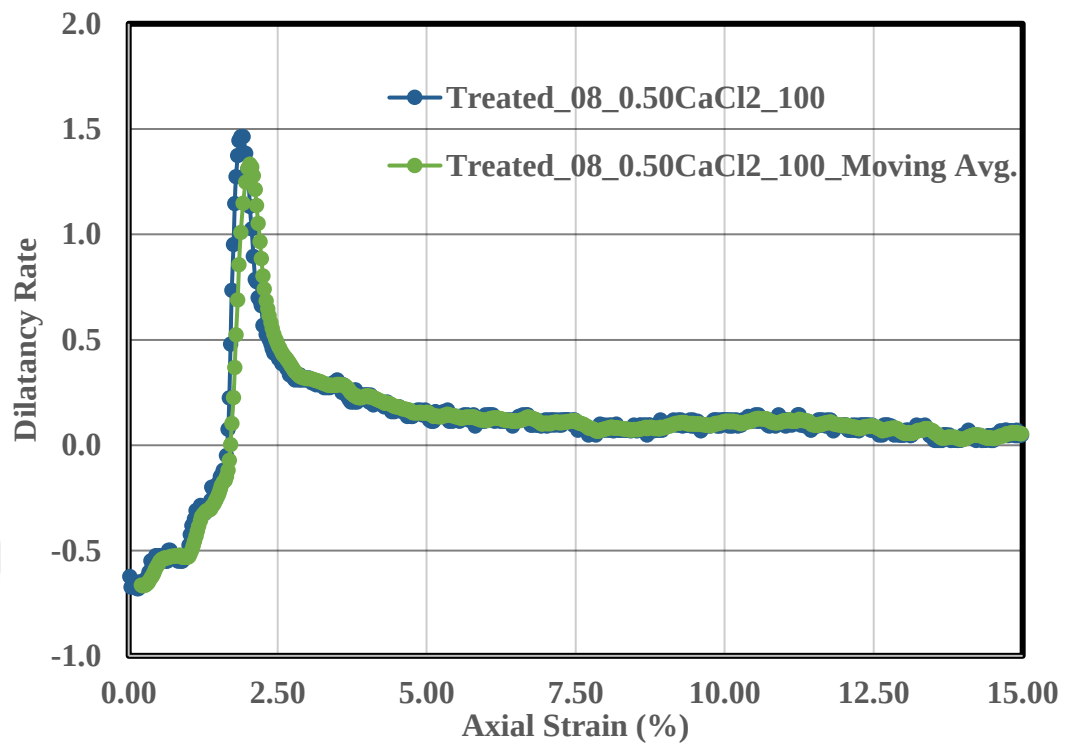
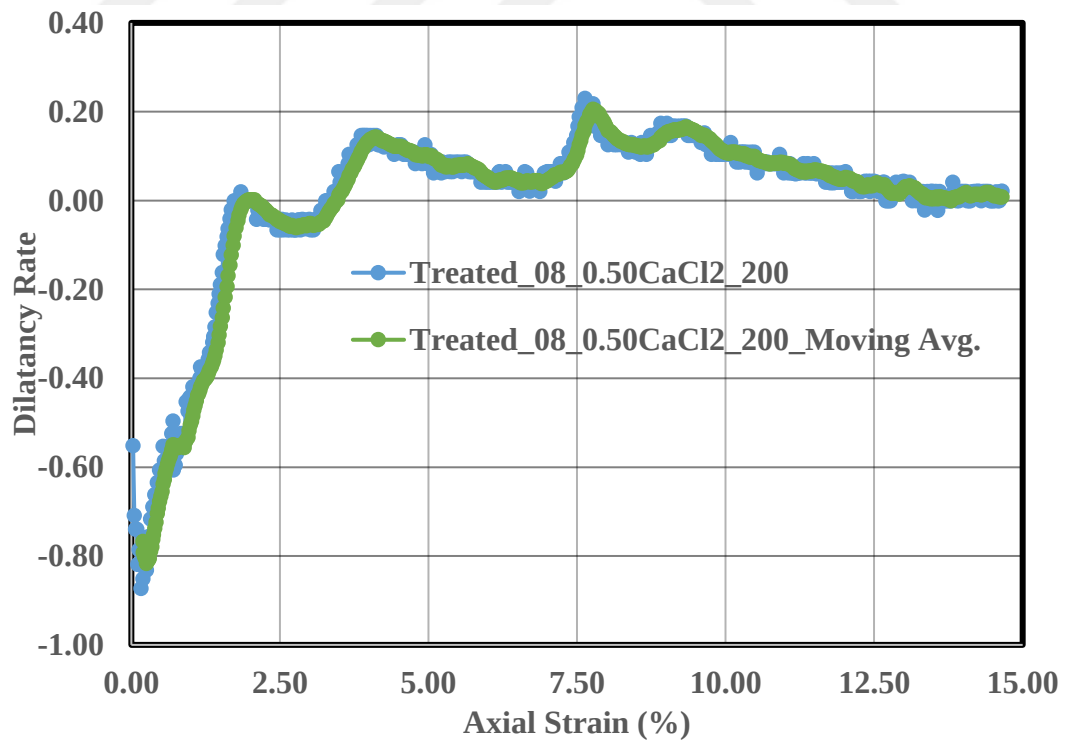


Figure 31 Dilatancy rate vs. axial strain (%) for 16 injections - 0.25 M CaCl_2 case
(a) 100 kPa (b) 200 kPa (c) 400 kPa



(a)



(b)

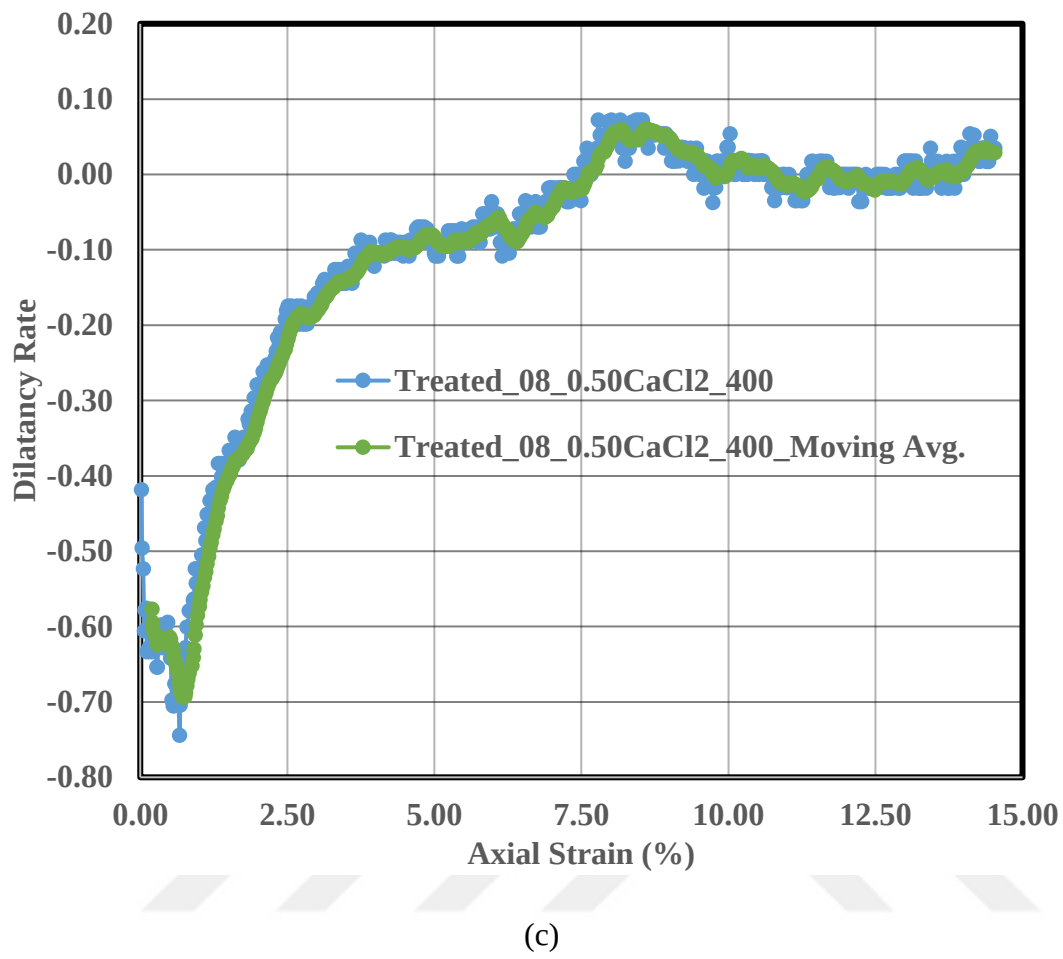
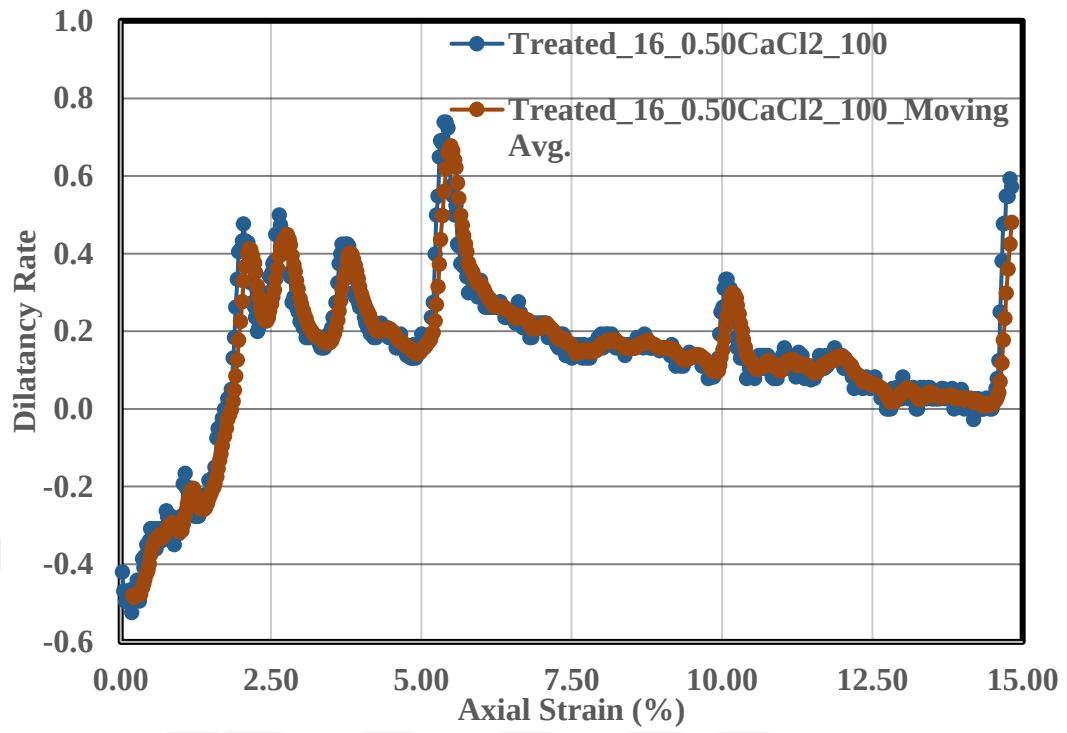
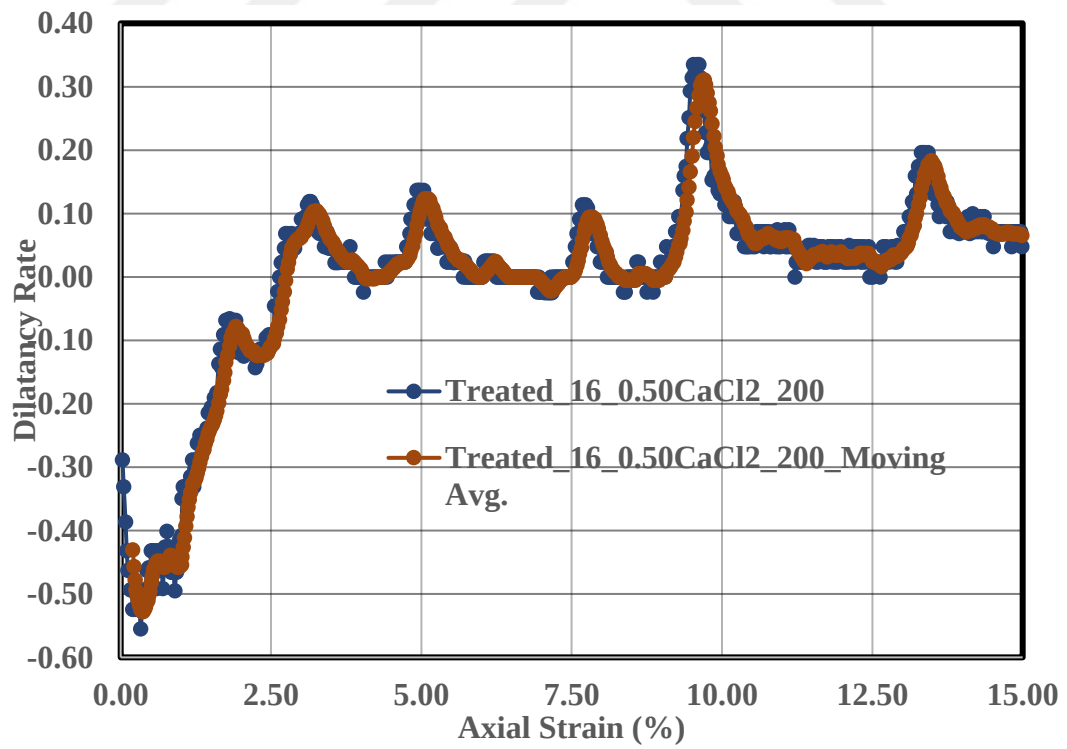


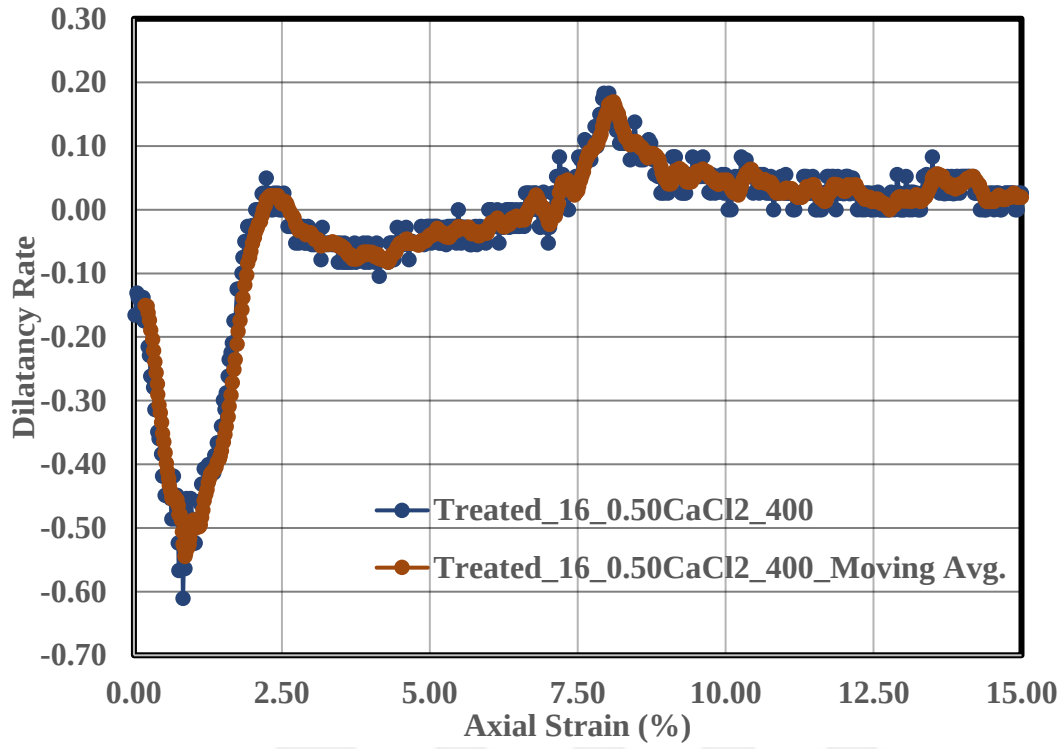
Figure 32 Dilatancy rate vs. axial strain (%) for 8 injections - 0.50 M CaCl_2 case
(a) 100 kPa (b) 200 kPa (c) 400 kPa



(a)



(b)



(c)

Figure 33 Dilatancy rate vs. axial strain (%) for 16 injections - 0.50 M CaCl₂ case
(a) 100 kPa (b) 200 kPa (c) 400 kPa

4.2 Initial Tangent Modulus (E_i)

Initial tangent modulus was obtained from the stress-strain curves. The slope of the initial tangent line was determined as the modulus. Janbu (1963) defines the initial tangent modulus as a function of confining pressure as seen in (4).

$$E_i = K P_a \left(\frac{\sigma_3}{P_a} \right)^n \quad (4)$$

Where

K = Janbu modulus (MPa)

P_a = Atmospheric pressure

n = An exponent

The E_i vs. σ_3 graph was drawn on a log-log scale and is shown in Figure 34. Initial modulus, K , and n values are described in Table 10 and Figure 35.

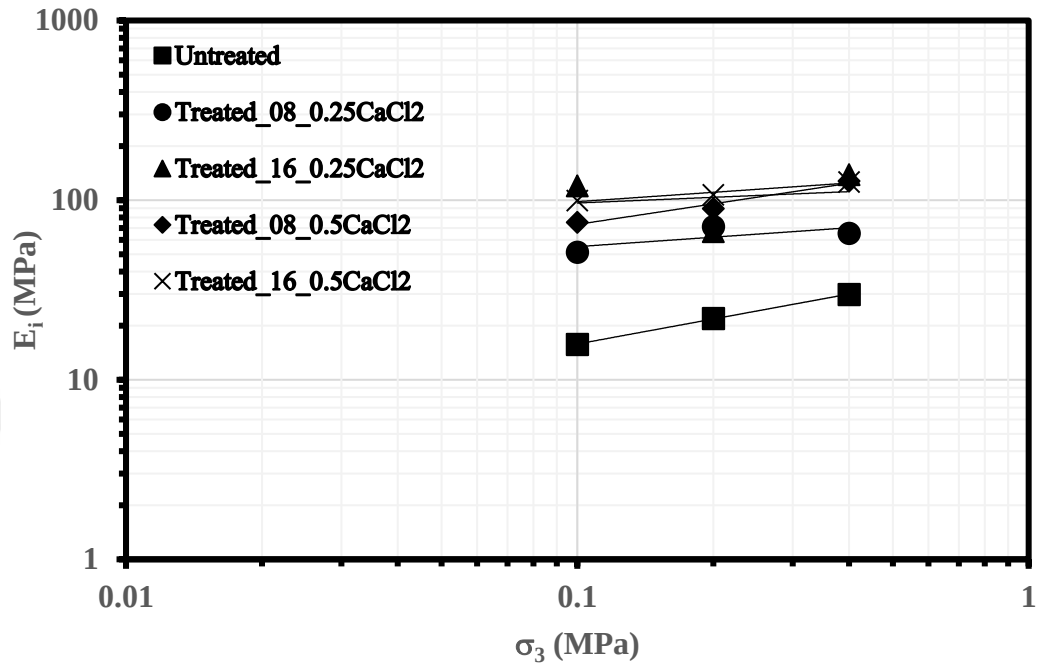


Figure 34 Initial tangent modulus of treated and untreated sands under different confinement pressures

Table 10 Initial tangent modulus for each test and Janbu parameters

Specimen	σ'_3 (MPa)	E_i (MPa)	n	K (MPa)
Untreated	0.1	16	0.46	157
	0.2	22		
	0.4	30		
0.25M CaCl ₂ - 8 Injections	0.1	51	0.17	545
	0.2	71		
	0.4	65		
0.25M CaCl ₂ - 16 Injections	0.1	120	0.11	952
	0.2	67		
	0.4	139		
0.50M CaCl ₂ - 8 Injections	0.1	75	0.38	726
	0.2	90		
	0.4	128		
0.50M CaCl ₂ - 16 Injections	0.1	99	0.17	970
	0.2	107		
	0.4	126		

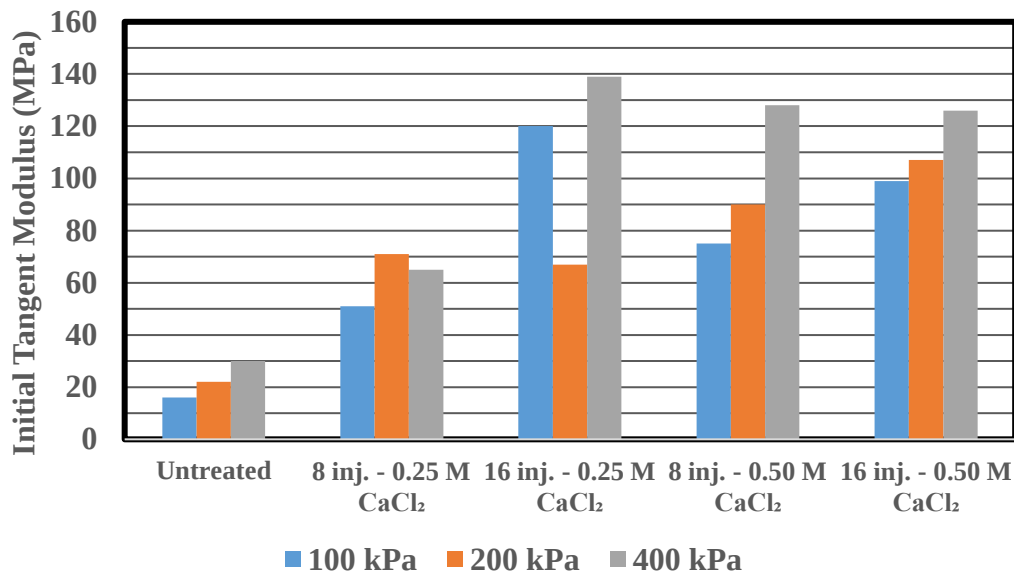


Figure 35 Initial tangent modulus for each case at each confining pressure

The fitted curves show a common trend that MICP treatment decreases the n value and increases K value, which agrees with other studies (Lin et al. 2015; Feng & Montoya 2015; Terzis & Laloui 2018) in the literature. A lower n value means less

dependency of initial tangent modulus to confinement pressure. Additionally, MICP treatment significantly increases the initial tangent modulus in all cases. A higher stiffness led to a faster peak shear mobilization, in which all tests peak shear reached at lower axial strains compared to untreated sand.

4.3 Effect of Treatment Number on Drained Response

Stress-strain behavior was profoundly affected by the number of the treatment cycle, as the higher peak shear strengths obtained at the 16 injection cases, compared to 8 injections. The number of injections directly related to cementation content, but higher cementation does not refer to higher shear strength. Shear strength heavily depends on effective calcium carbonate crystals (Cheng et al. 2013), i.e., grain bonding the crystals. The higher number of treatment cycles possibly led to more number of bonding calcium carbonate crystals. As Wang et al. (2019) explained in microfluid chip experiments, bacterial aggregates produce initially unstable calcite crystals (vaterite). Then, unstable ones were dissolved. Meantime, stable ones (calcite) were produced on bacterial aggregates. In that study, two cementation solution injections were performed, and crystal growth was observed after the second injection. It is possible that 16 injections produced larger crystal agglomerates compared to 8 injections case, and enhanced the shear strength. Additionally, higher injection numbers led to higher initial stiffness, as seen in Table 10.

At 100 kPa confining pressure, all of the specimens exhibited strain hardening, followed by strain-softening, except 8 injections, 0.25M CaCl₂ case. A visible failure plane was observed in 8 injections, 0.50M CaCl₂ (Figure 44), as the volumetric behavior suddenly transformed to dilative behavior from contractive behavior. It may be due to an increase in localized porosity at the specimen, i.e., increased void ratio at the shear plane. However, in its counterpart specimen (Treated_16_0.5CaCl₂_100), no visible shear plane was observed. The specimen deformed at the bottom, as the top part remained intact (Figure 47). The deformed shape of the specimen clearly shows the inhomogeneity. Also, in 16 injections – 0.5

M CaCl₂ case, peak deviatoric stress was highest at the 100 kPa confining pressure, as it refers to the inhomogeneity of the specimen again. But the volumetric behavior was still suppressed as the confining pressure increased. A similar phenomenon was observed in 16 injections – 0.25M CaCl₂ case, in which peak deviatoric stresses are almost the same at each confinement level, meanwhile dilative behavior was suppressed. The specimens were again deformed at the bottom. Martinez et al. (2013) investigated microbe density along the half-meter sand column, and the density has decreased from top to bottom, as the injection was made in the same direction. This could possibly explain the inhomogeneity of the specimen (Feng & Montoya 2016), as in all tests, the bottom part deformed the most. However, in 8 injections case, peak deviatoric stresses were increased with the increasing confining stresses. 16 injections case probably led to a more nonhomogeneous specimen.

The initial tangent modulus was highly affected by the number of treatment cycles. The higher number of treatment cases showed greater initial tangent modulus at each confining pressure. For the 0.25 M CaCl₂ case, the initial tangent modulus almost doubled for 16 injections case compared to 8 injections one. The increase in the 0.50 M CaCl₂ case is not evident, as in the case of 0.25 M CaCl₂.

Volumetric behavior is also dependent on the treatment number. 16 injection case was showed more dilative behavior compared to 8 injection case at 100 kPa confining pressure. 8 injection case did not affect volumetric behavior profoundly compared to untreated sand at 400 kPa confining pressure. On the contrary, 16 injection case the specimens started to dilate at lower axial strains. The distinction with volumetric behavior of treated and untreated sands was disappeared at the 8 injection case, yet 16 injections case was showed a faster dilation mobilization.



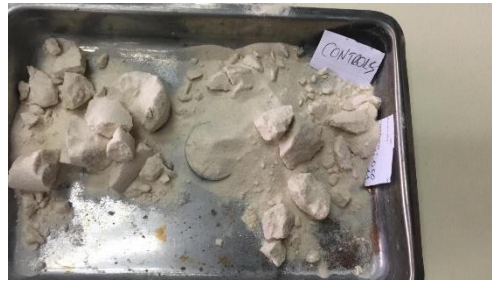
(a)



(b)



(c)

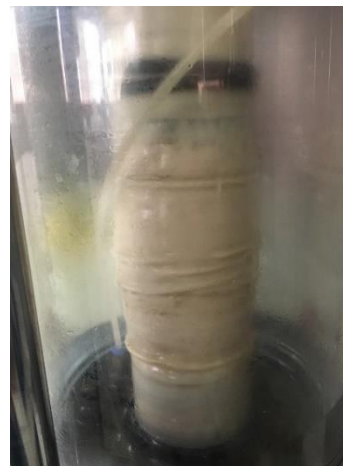


(d)

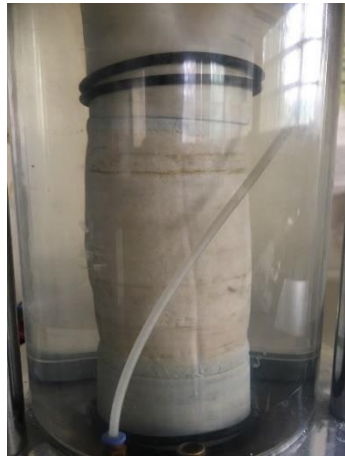
Figure 36 8 injections - 0.50 M CaCl_2 case control tests (a) after completing injections (b) while transferring the oven (c) after oven dried (d) while transferring the pedestal



(a)



(b)



(c)

Figure 37 Deformed shape of untreated sand (Left is during the test, right is the post-failure shape) (a) 100 kPa (b) 200 kPa (c) 400 kPa

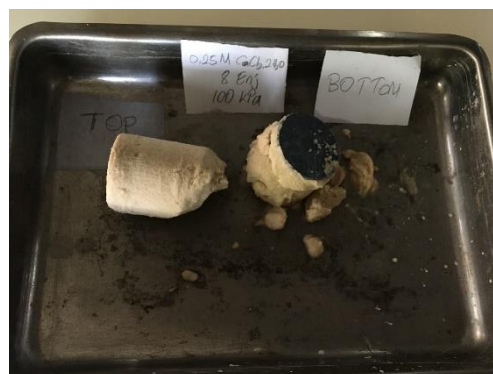


Figure 38 Test Treated_08_0.25CaCl2_100, deformed shape (during and post-failure) and collected specimen

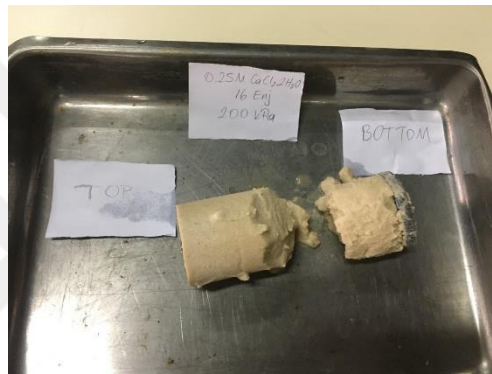
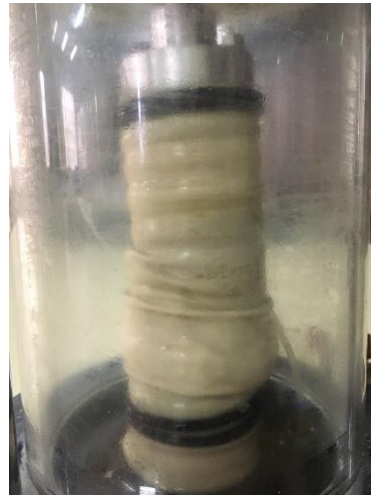


Figure 39 Treated_08_0.25CaCl2_200, deformed shape (during and post-failure) and collected specimen





Figure 40 Treated_08_0.25CaCl₂_400, deformed shape (during and post-failure) and collected specimen

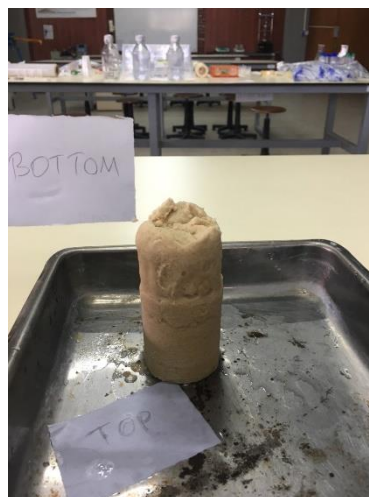


Figure 41 Treated_16_0.25CaCl₂_100, deformed shape (during and post-failure) and collected specimen

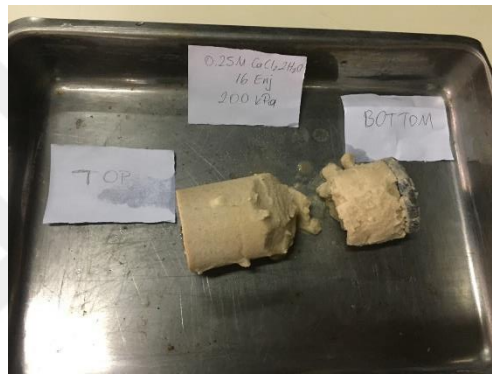
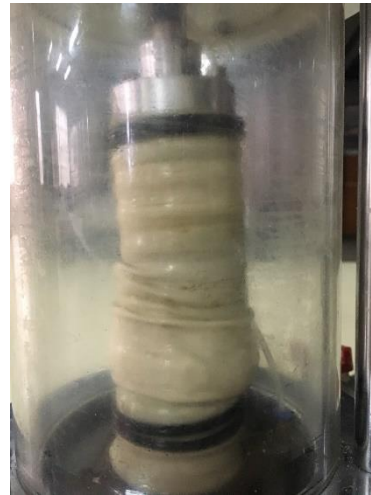
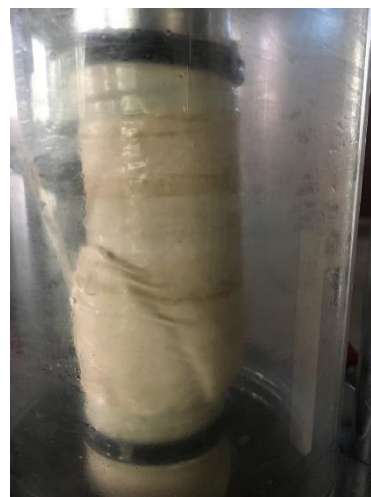


Figure 42 Treated_16_0.25CaCl2_200, deformed shape (during and post-failure) and collected specimen



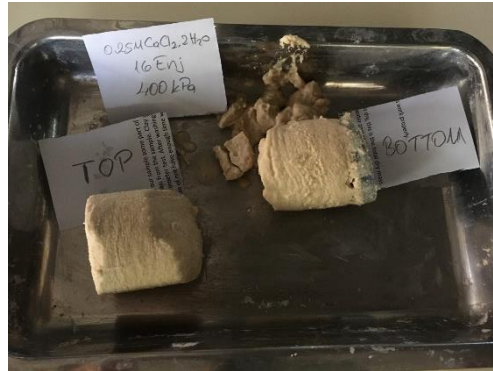


Figure 43 Treated_16_0.25CaCl2_400, deformed shape (during and post-failure) and collected specimen



Figure 44 Treated_08_0.50CaCl2_100, deformed shape (during and post-failure) and collected specimen (specimen couldn't be collected at two pieces)





Figure 45 Treated_08_0.50CaCl2_200, deformed shape (during and post-failure) and collected specimen

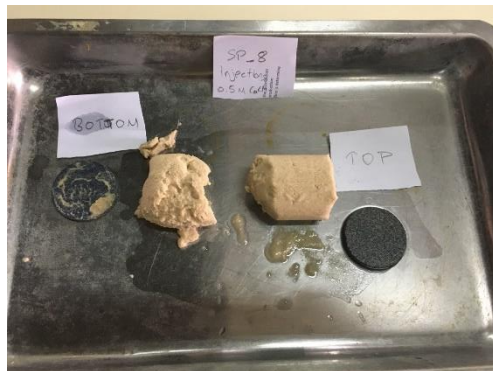


Figure 46 Treated_08_0.50CaCl2_400, deformed shape (during and post-failure) and collected specimen

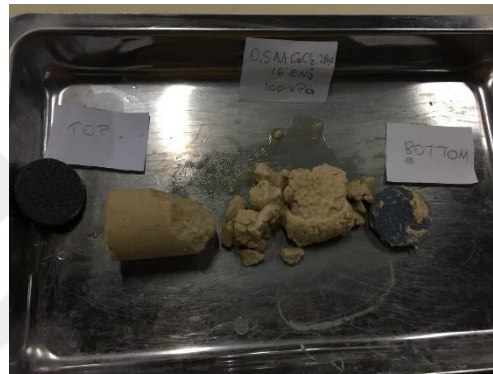
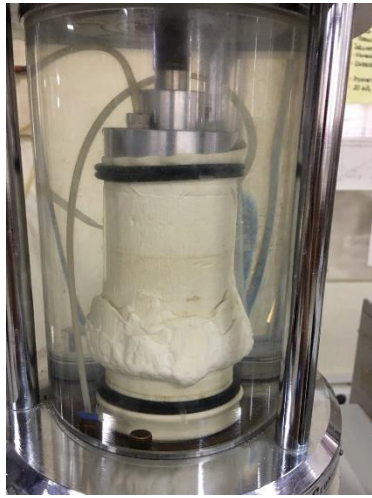


Figure 47 Treated₁₆-0.50CaCl₂-100, deformed shape (during and post-failure) and collected specimen



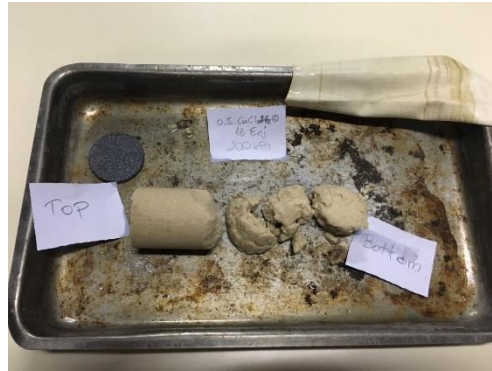


Figure 48 Treated_16_0.50CaCl2_200, deformed shape (during and post-failure) and collected specimen



Figure 49 Treated_16_0.50CaCl2_400, deformed shape (during and post-failure) and collected specimen

4.4 Effect of $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ Concentration on Drained Response

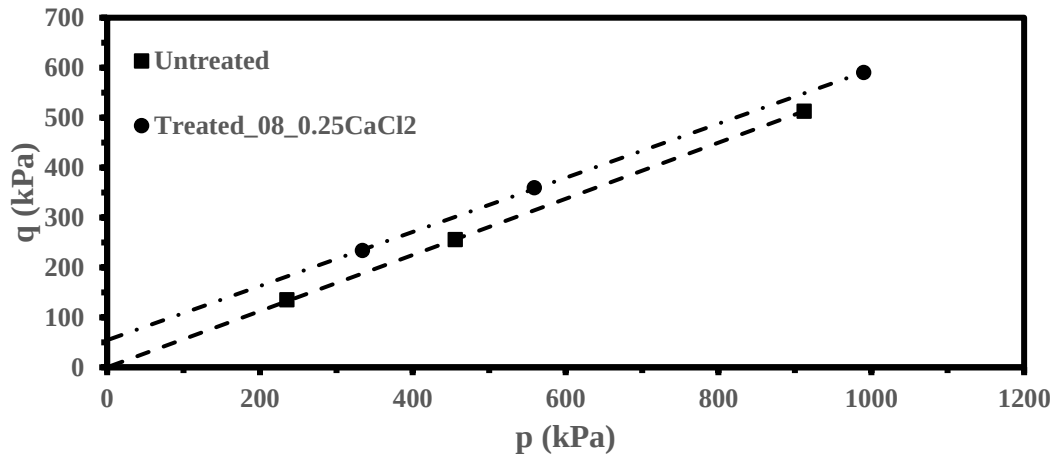
Two different $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ concentrations (0.25M and 0.50M) were investigated as a treatment solution by keeping the urea to calcium ratio constant. There is an apparent trend in all experiments that 0.50M cases produced higher peak shear resistance for the same injection number. This could be explained by the difference in crystal size for both cases. Al-Thawadi & Cord-Ruwisch (2012) studied variance in the crystal size by changing the concentration of CaCl_2 , with equimolar urea- CaCl_2 . Increasing CaCl_2 concentration resulted in larger calcium carbonate crystals and a greater number of effective crystals. 8 injection – 0.25 M CaCl_2 case produced the least peak shear strength, and the behavior did not transform from ductile to brittle at 100 kPa effective confining stress. The specimen exhibited only strain hardening behavior like the untreated case. 8 injection – 0.50 M CaCl_2 case produced higher peak shear strength compared to 16 injection – 0.25 M CaCl_2 . On the contrary, 16 injection – 0.25 M CaCl_2 cases exhibited higher residual strength. It could possibly be due to increased frictional resistance or residual cohesion. 0.25 M case would produce a higher number of crystals. As the cementation degrades with shearing, broken crystals fill the void space, provide newly generated contact points with the sand grain and coated calcium carbonate crystals. The higher number of rhombohedral shaped crystals provides better interlocking, i.e., higher frictional resistance. But also, residual cohesion could lead to the higher strength, as in the all deformed shapes, the top part is intact, can stand with its self-weight. Feng & Montoya (2016) justified the increase in strength by higher frictional resistance for moderately and lightly cemented sands. But the calcium carbonate content in this study is greater than the specimens in Feng & Montoya (2015). Additionally, the same phenomenon was also observed at the 200 kPa confining pressure, even though the peak shear resistances were the same for both cases. This notion has disappeared at 400 kPa confining pressure, likely due to the nonhomogeneous distribution of CaCO_3 in 16 injection – 0.25 M CaCl_2 case, where the residual strength is almost the same at 200 and 400 kPa effective confining pressure. On the other hand, residual

strength was almost increased by 50% at the same effective confining pressure range for 8 injection – 0.50 M CaCl₂ case.

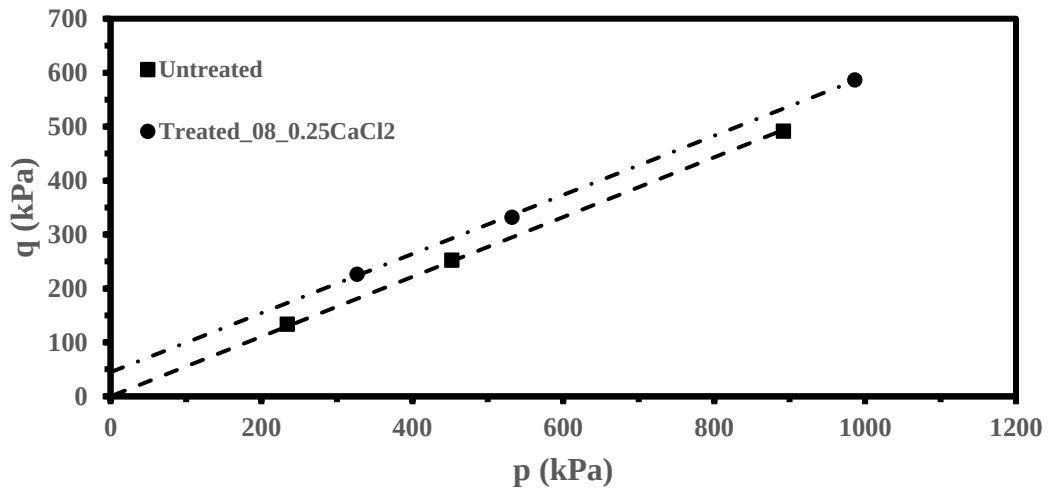
There is not a clear trend between the volumetric behavior of 0.25 M CaCl₂ and 0.50 M CaCl₂ cases. Nevertheless, a distinction can still be seen. For the 16 injections case, dilation was mobilized earlier axial strains at 0.25 M CaCl₂ case compared to 0.50 M CaCl₂ case under 100 kPa effective confining pressure. Yet, 0.50 M CaCl₂ case was showed a higher rate of dilatancy, which led to a higher positive volumetric strain at 10% axial strain. For the 8 injections case, the same phenomenon has occurred, where dilation started at lower axial strains in the 0.25 M CaCl₂ case. However, 0.50 M CaCl₂ case was exhibited a greater rate of dilatancy, which is likely due to the occurrence of the shear band (see Figure 44). The dilative behavior was suppressed at 200 and 400 kPa effective confining stresses for both cases, in which the specimens were behaved contractive or dilative with a very low rate of dilatancy.

4.5 Shear Strength Parameters

Mohr-Coulomb modified failure surface was assessed for the stress range 100 – 400 kPa and is shown in Figure 50. It is only assessed to untreated sand, and 8 injections – 0.25 M CaCl₂ case since the other cases showed significant nonhomogeneous behavior, in which specimens under 100 kPa confining pressure showed greater peak shear strength compared to specimens under higher confining pressures. The envelopes are represented in p-q space, which p and q are defined as $\frac{(\sigma_1 + \sigma_3)}{2}$ and $\frac{(\sigma_1 - \sigma_3)}{2}$, respectively.



(a)



(b)

Figure 50 Modified failure envelopes for (a) peak shear strength (b) residual strength

The peak and residual frictional angles for the untreated case are 32° and 31° , respectively. The MICP treated sand showed a similar peak and residual angle of friction, 31° , and 31° . However, it exhibited a greater cohesion at peak strength (60 kPa). It clearly shows that MICP treatments did not affect the friction angle, yet the effect on cohesion is evident. The residual cohesion is similar to peak strength cohesion, which is 50 kPa. The similarity is expected since 8 injections – 0.25 M

CaCl₂ case specimens do not exhibit any strain softening. A possible reduction or degradation in cohesion is possible for other cases since they tend to have strain-softening behavior. However, strength parameters cannot be defined for these cases due to the nonhomogeneous distribution of CaCO₃.

4.6 CaCO₃ Contents

Many researchers investigated the amount of precipitated calcium carbonate content (Lin et al. 2015; Montoya & DeJong 2015; Feng & Montaya 2016) and related it to improved strength characteristics of sand (Cui et al. 2017). It is sensible to follow this approach in a single type of sand and cementation solution. However, different treatment concentrations and grain sizes might change precipitation patterns and crystal sizes. Yet, determining CaCO₃ content is somewhat insightful, and it will be discussed next.

Table 11 shows the calcium carbonate contents at the top, bottom, and rest of the specimens. Unfortunately, specimens were not taken at once piece after the triaxial testing (excluding the repeat tests). Therefore, the contents were determined for the top, bottom, and the rest. In all of the injection schemes, the bottom part has considerably less calcium carbonate content than the top part. But the two-point approach might not reflect the overall distribution.

Table 11 reflects the effect of CaCO₃ content on the peak deviatoric stress. An increase in cementation content enhanced the peak stress. This phenomenon was also observed in Clough et al. (1981) for naturally cemented sands. In all tests, peak stress was increased, moving from 200 kPa to 400 kPa, except the 16 injections – 0.50 M CaCl₂ case. But, it is possibly due to the lesser content of CaCO₃, since the specimen has 10.3% CaCO₃, yet other specimens (100 and 200 kPa) has 14.5 and 13.2%.

Table 11 CaCO₃ contents (%) at the top, bottom, and rest of the specimens

Specimen	σ'_3 (kPa)	CaCO ₃ at the top (%)	CaCO ₃ at the bottom (%)	Whole specimen (%)	Peak deviatoric stress (kPa)
0.25 M CaCl ₂ - 8 Injections	100	10.4	3.8	5.7	469.5
	200	10.4	4.5	5.4	721.9
	400	11.6	4.0	5.6	1180.1
0.25 M CaCl ₂ - 16 Injections	100	13.8	9.1	9.1	1412.9
	200	13.6	8.9	8.7	1432.0
	400	12.9	6.4	7.6	1384.0
0.50 M CaCl ₂ - 8 Injections	100	11.7	5.6	8.2	1596.4
	100 Repeat 1	-	-	8.7	1288.2
	100 Repeat 2	-	-	7.7	1140.9
	200	10.5	6.0	8.2	1421.4
	200 Repeat	-	-	7.8	1309.9
	400	10.6	6.9	7.5	1721.5
	400 Repeat	-	-	9.3	1845.9
0.50 M CaCl ₂ - 16 Injections	100	17.5	9.9	14.5	2614.7
	200	16.9	10.6	13.2	2216.4
	400	16.4	8.7	10.3	2358.3

Repeat test data are available in Appendix A.

Figure 51 shows the exponential relation ($y = ae^{bx}$) between peak deviatoric stress and calcium carbonate content for each stress level. A better fit would be obtained by having more data given that the lowest calcium carbonate content starts from 5.4%. The disadvantage of not having data at this cementation range is that the curve should start flat at the lower contents instead of steep since lightly cemented sands have similar peak deviatoric stress compared to untreated sand (Feng & Montoya 2016). Additionally, exponential relation was also observed in van Paassen (2009), Cui et al. (2017), and Terzis & Laloui (2018).

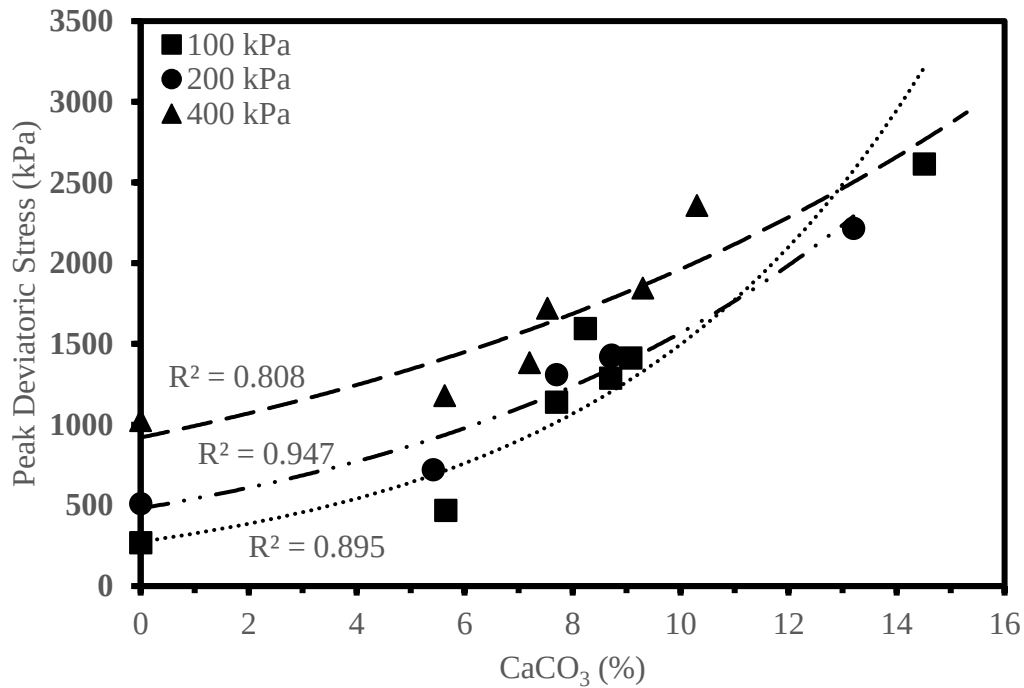


Figure 51 Peak deviatoric stress vs. CaCO_3 content at each effective confining pressure

Figure 52 indicates the nonhomogeneous distribution of calcium carbonate within the specimens. The least precipitation occurred at the bottom for all specimens. Previously, deformed shapes were linked with the nonhomogeneous distribution, but the acid washing technique showed the phenomenon quantitatively, even though the distribution profiles are available for repeat tests of 8 injections – 0.50 M CaCl_2 case.

Nonhomogeneous distribution affects the mechanical response of treated sands, as seen in Figure 53. Repeat 1 and Repeat 2 tests have similar CaCO_3 contents, 8.7 and 7.7%, respectively. Yet their response is somewhat different. Repeat 1 test shows a stiffer response compared to Repeat test 2 and reaches peak deviatoric stress at the earlier axial strain. Therefore, the behavior transforms from contractive to dilative faster for the Repeat 1 test. This might be due to the fact that Repeat 2 test specimen has 5.0% calcium carbonate at section 4, which shows that it is weaker at the bottom.

However, the content at section 4 is 7.2% for Repeat 1 tests. The Repeat 2 test has less peak deviatoric stress, naturally. But at the higher strains, the behavior is similar.

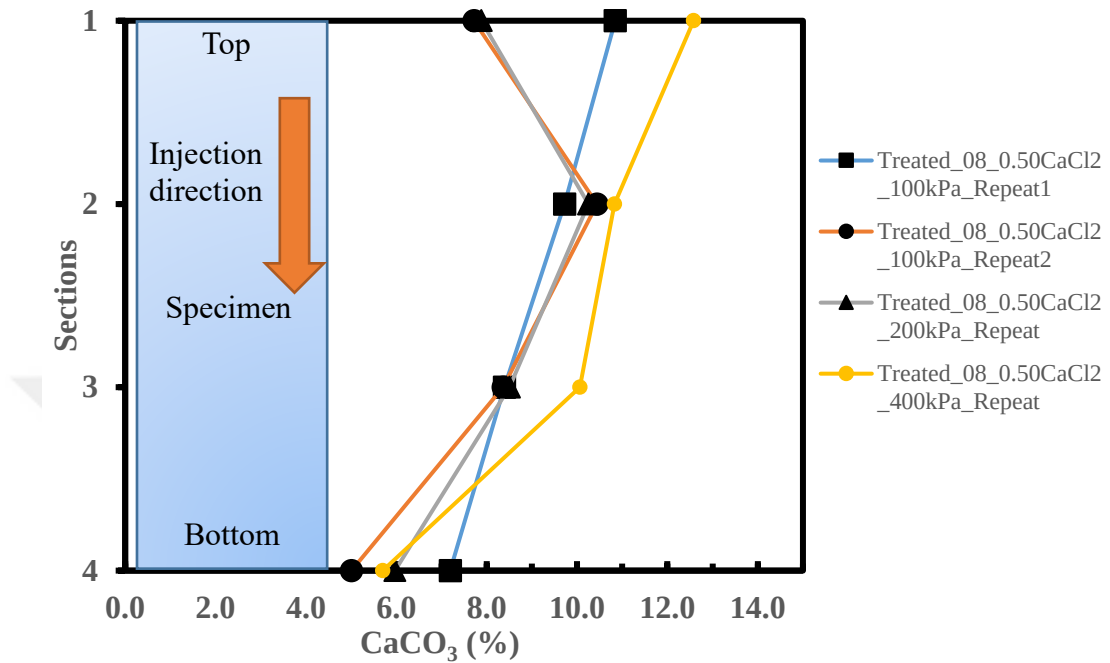
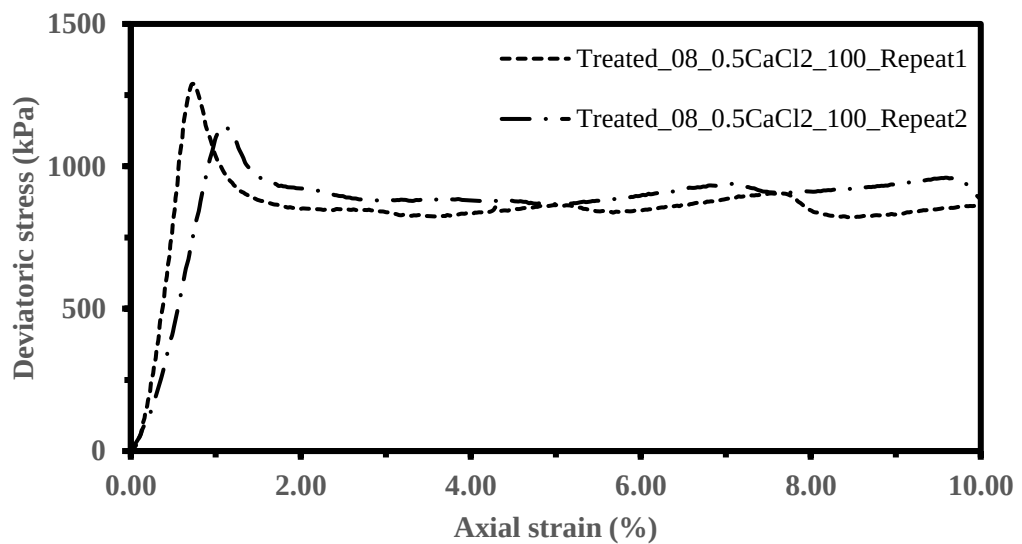
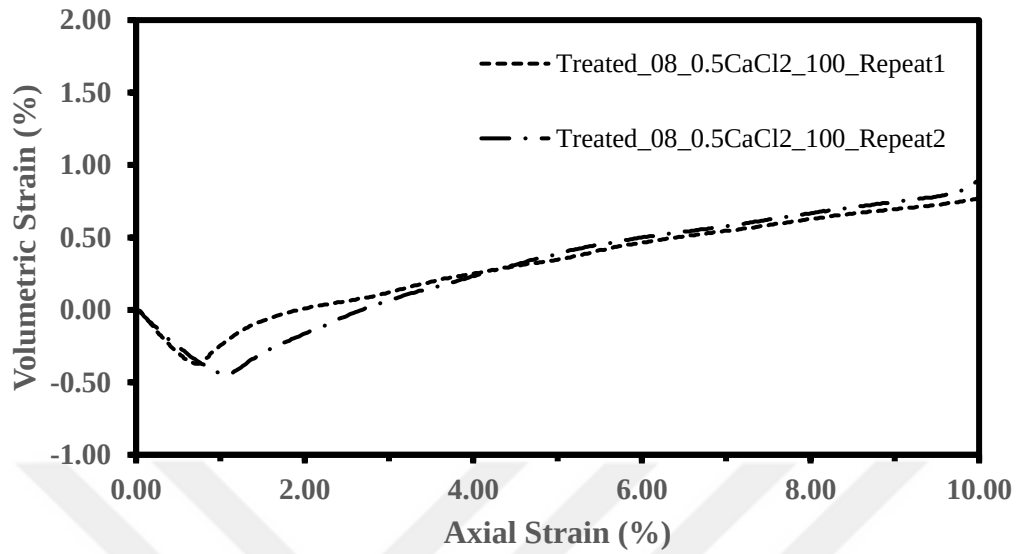


Figure 52 CaCO_3 (%) profiles of 8 injection - 0.50 M CaCl_2 case for repeat tests



(a)



(b)

Figure 53 8 injection - 0.50 M CaCl_2 at 100 kPa effective confining pressure (a) Deviatoric stress vs. axial strain (b) volumetric strain vs. axial strain

4.7 Scanning Electron Microscopy (SEM) Images

SEM images were taken at METU Central Laboratory. The oven-dried samples were taken from the intact part of specimens and placed on the conductive adhesive tabs, as seen in Figure 54. Then Au-Pb coating was done. EDS analyses were also performed during the imaging.



Figure 54 SEM samples placed on the conductive adhesive tab

Figure 55 shows the SEM images from the untreated specimen. The images clearly show the grain coating (Figure 56, Figure 57, Figure 58, and Figure 59) and effective grain binding mechanisms (Figure 57 and Figure 59). Precipitated crystals are mostly in rhombohedral shape, which indicates that crystals are in a stable form, i.e., calcite. EDS analyses proved the precipitation of calcium carbonate within the soil specimen. Several spherical crystals are also formed and shown in SEM images (Figure 57). Spherical crystals might be an unstable form of calcium carbonate called vaterite. Wang et al. (2019) investigated the precipitation pattern in microfluid chips and showed that spherical crystal precipitates first and dissolves after time passes. Yet, spherical crystals can be seen in SEM images. Van Paassen (2009) revealed spherical crystals in the SEM images, with their size up to 50 μm . In Figure 57, spherical crystals marked with rectangles have a diameter of 65 and 80 μm . The apparent bowl shape (marked in a rectangle) is seen in Figure 58, which possibly indicates the debonding of a sand grain.

EDS analysis from untreated sand (Figure 60) reveals the silica and oxygen peaks as expected since the sand used in this study is quartz. The analyses from treated cases

(Figure 61 and Figure 62) show a high percentage of calcium, which indicates the precipitation of calcium carbonate. However, a small peak of calcium was also observed. This might be due to the residuals of the cementation solution within the specimen, even though distilled water was flushed after completing the injections.

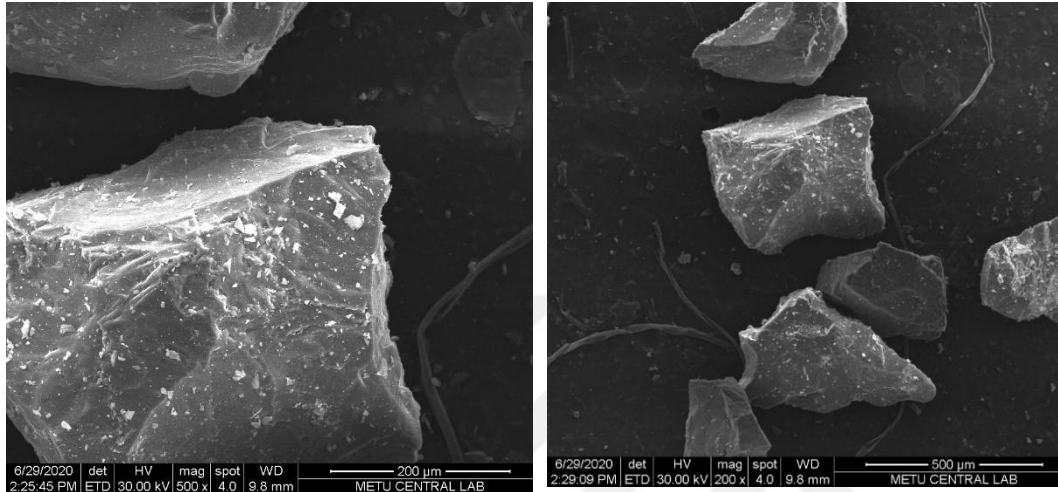


Figure 55 SEM images from the untreated specimen

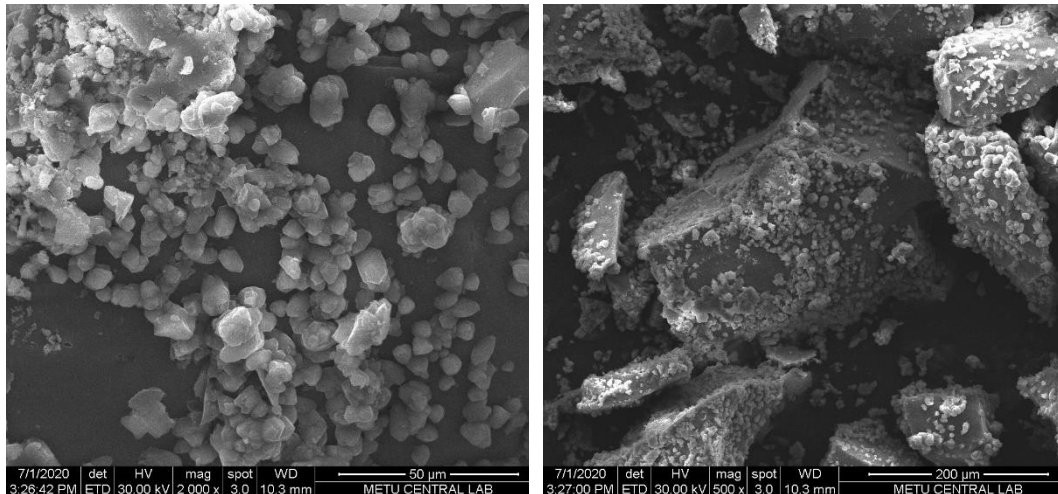


Figure 56 SEM images from Treated_08_0.25CaCl₂_100kPa specimen

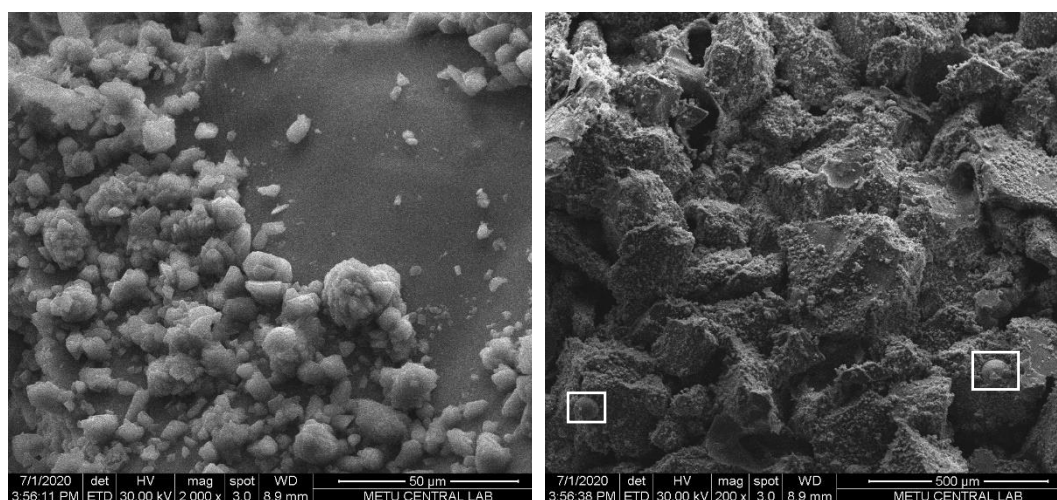


Figure 57 SEM images from Treated_08_0.50CaCl₂_100kPa specimen

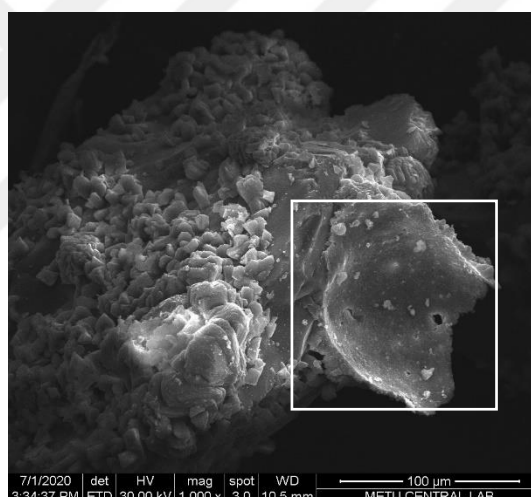


Figure 58 SEM image from Treated_16_0.25CaCl₂_100kPa specimen

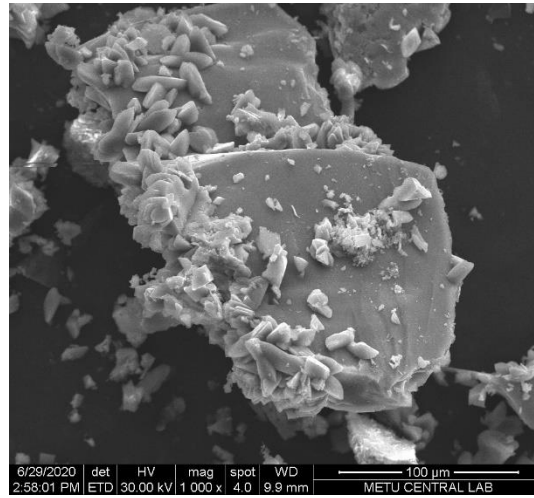


Figure 59 SEM image from Treated_16_0.50CaCl₂_100kPa specimen

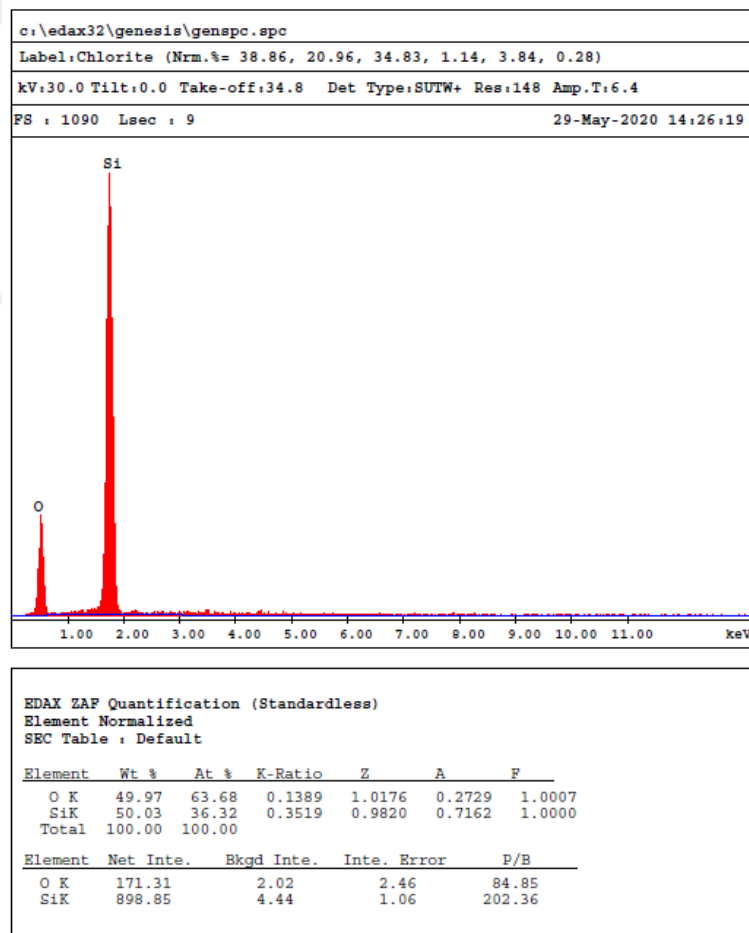


Figure 60 EDS analysis result for the untreated sand

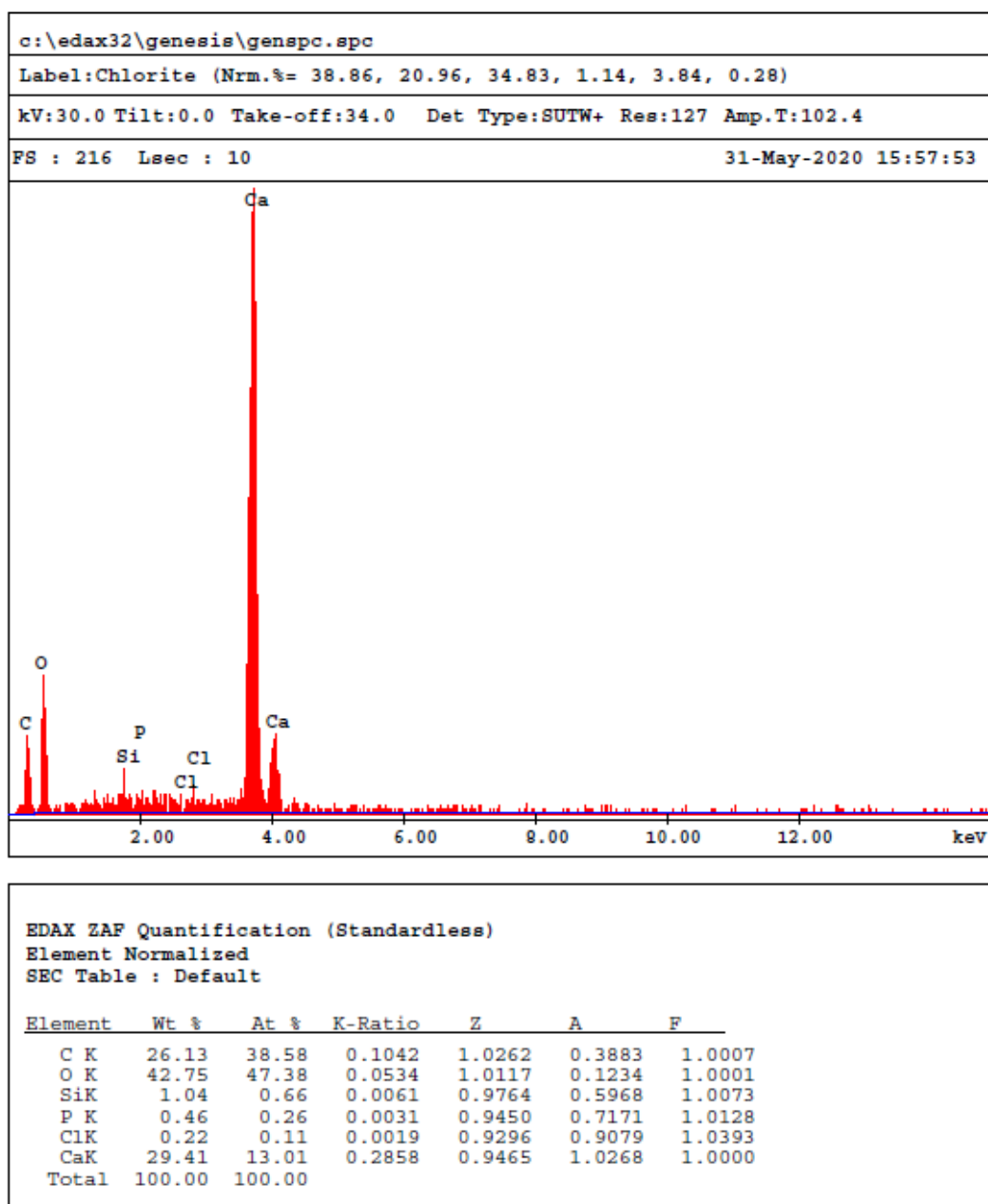


Figure 61 EDS analysis result from Treated_08_0.50CaCl2_100kPa specimen

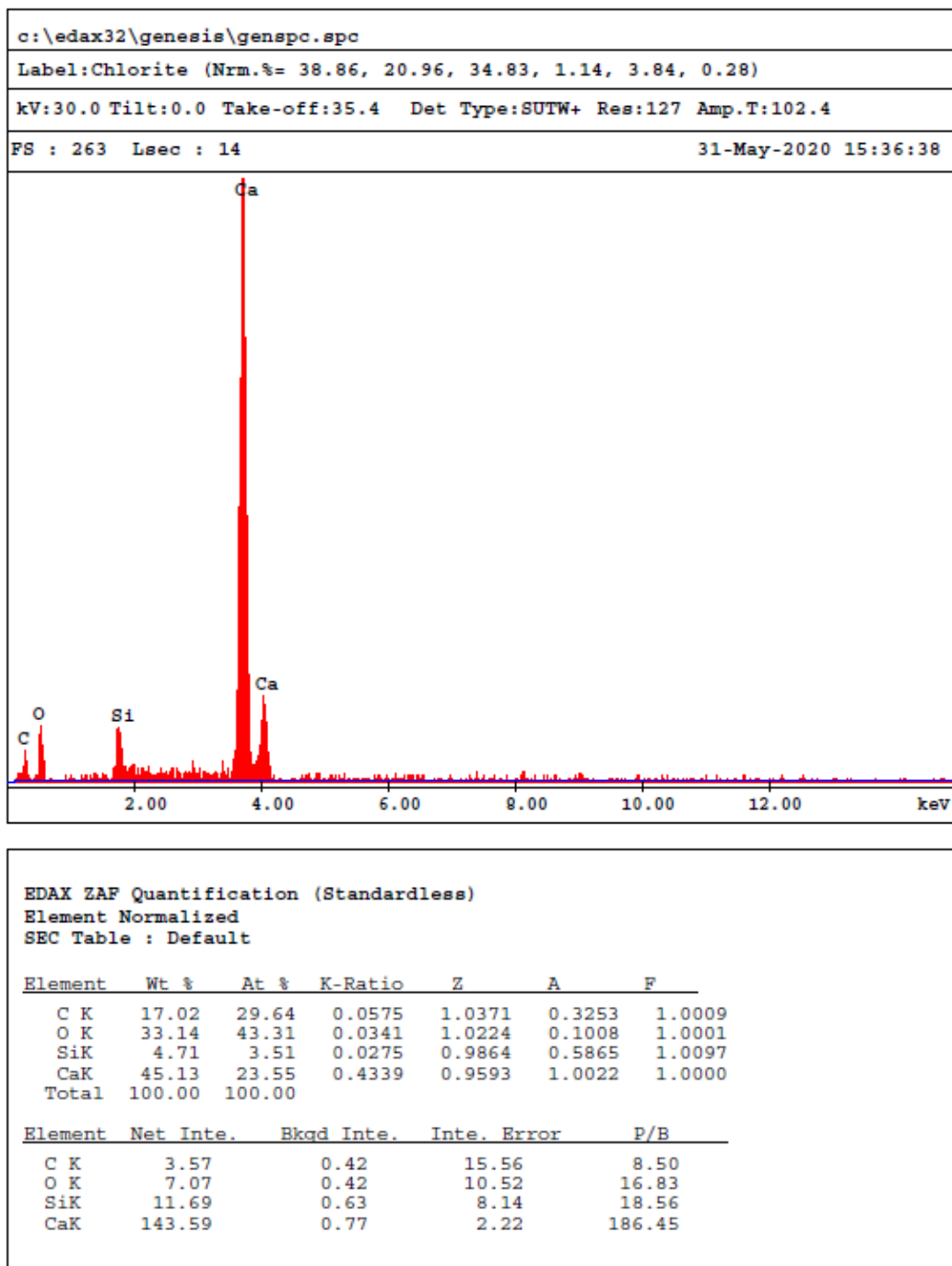


Figure 62 EDS analysis result from Treated_16_0.25CaCl₂_100kPa specimen

CHAPTER 5

CONCLUSIONS AND FUTURE WORKS

The work presented herein discusses the various specimen preparation method for MICP treated sands with different treatment numbers and concentrations and investigates the mechanical behavior of these sands by conducting conventional drained triaxial tests. Concluding remarks and future works will be shared in this chapter.

5.1 Conclusions

Preparing MICP treated sand specimens and conducting triaxial tests were challenging and rewarding processes. This study enables us to understand the mechanical behavior of MICP treated sands and shows how the treatment number and concentration affect the behavior itself. The conclusion drawn from this study are:

- MICP treated sands exhibited a higher peak deviatoric stress, stiffness, and dilative volumetric strain than untreated sand even for the treatment with the least number and concentration.
- The behavior is confinement dependent, as the increase in deviatoric stress and dilative behavior are suppressed with increasing effective confining pressures. At the lower confinements (i.e., 100 kPa), specimens exhibit significant strain-softening behavior.
- An increase in injection number leads to higher initial stiffness and peak deviatoric stress at the same chemical concentration. The enhancement of peak deviatoric stress was also achieved by increasing the chemical concentration by keeping the treatment number constant.

- Treated sand exhibit a higher peak and residual cohesion for the least improved case, yet the effect on friction angle is negligible.
- Mohr-Coulomb failure envelope only assessed for 8 injections – 0.25 M CaCl_2 case, since other cases exhibited significant inhomogeneity. A better injection method should be adopted to improve the distribution of calcium carbonate.

5.2 Future Works

This study does not extensively deal with the alternative injection schemes or various concepts within the MICP literature due to the limited time frame.

Possible improvement in this work could be made by:

- proposing alternative injection schemes to obtain a homogeneous distribution of calcium carbonate
- obtaining an optimum injection scheme that provides sufficient improvement within an economic framework
- working at lower calcium carbonate contents
- utilizing biostimulation of native bacteria instead of bioaugmentation
- making treatments under confining pressure to stimulate the real conditions
- investigating the effect of different grain sizes on MICP
- investigating the fine content on possible MICP treatments

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APPENDICES

A. REPEAT TESTS

The repeat tests are presented here in this section.

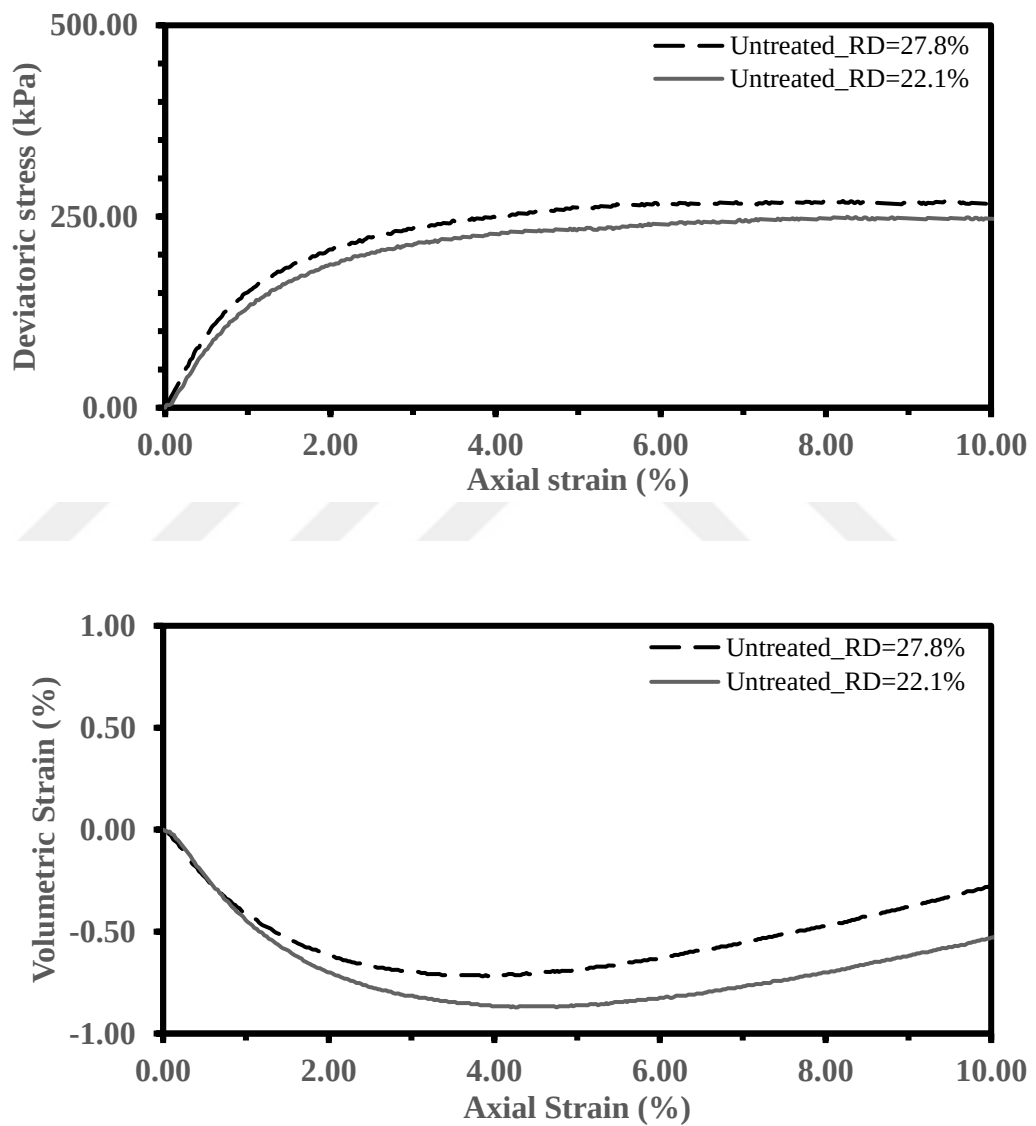


Figure A.1 Stress vs. strain and volumetric strain vs. axial strain curves for untreated sands with a relative density of 22.1 and 27.8% at 100 kPa effective stress

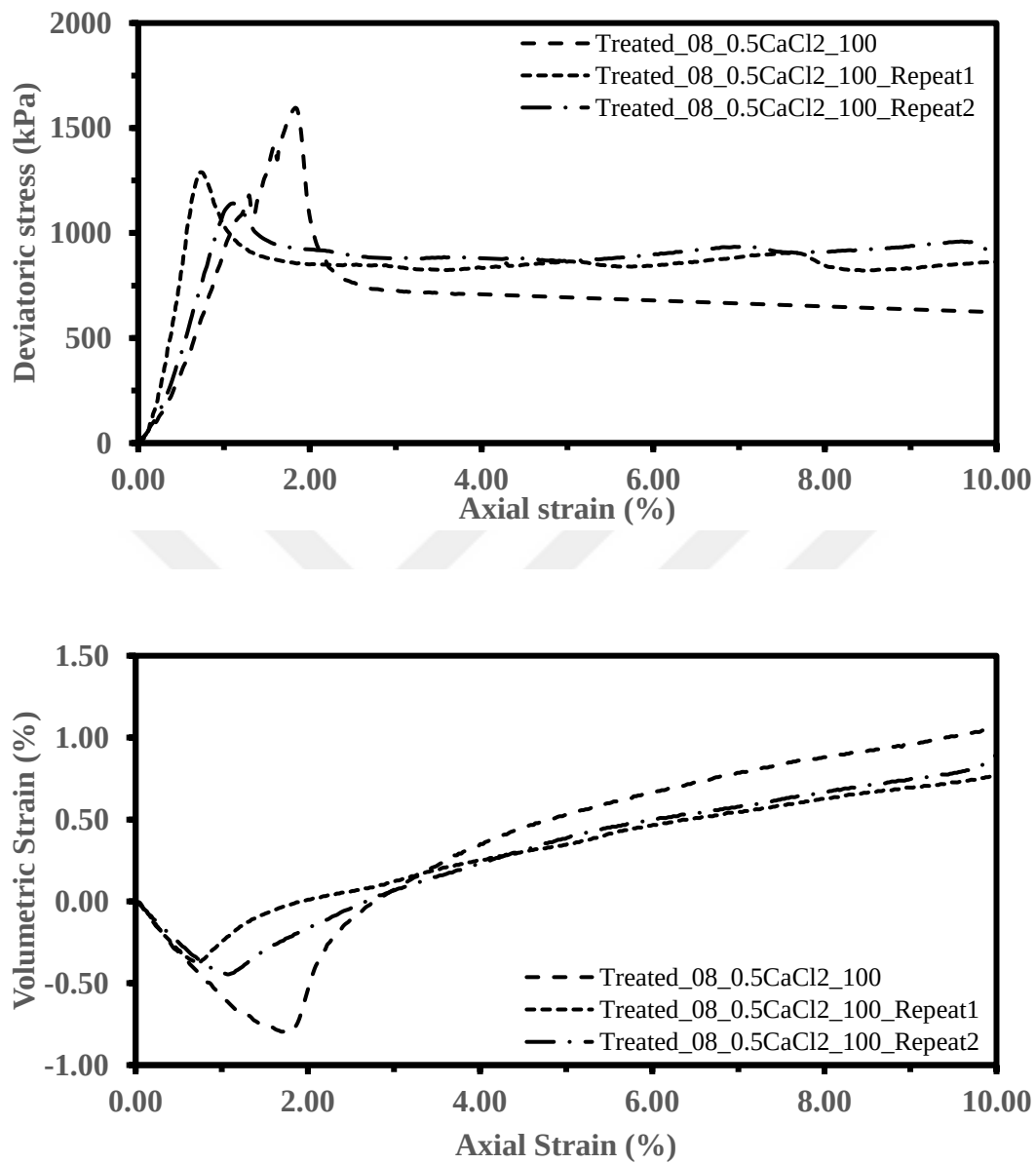


Figure A.2 Stress vs. strain and volumetric strain vs. axial strain curves for 8 injections – 0.50 M case at 100 kPa effective stress

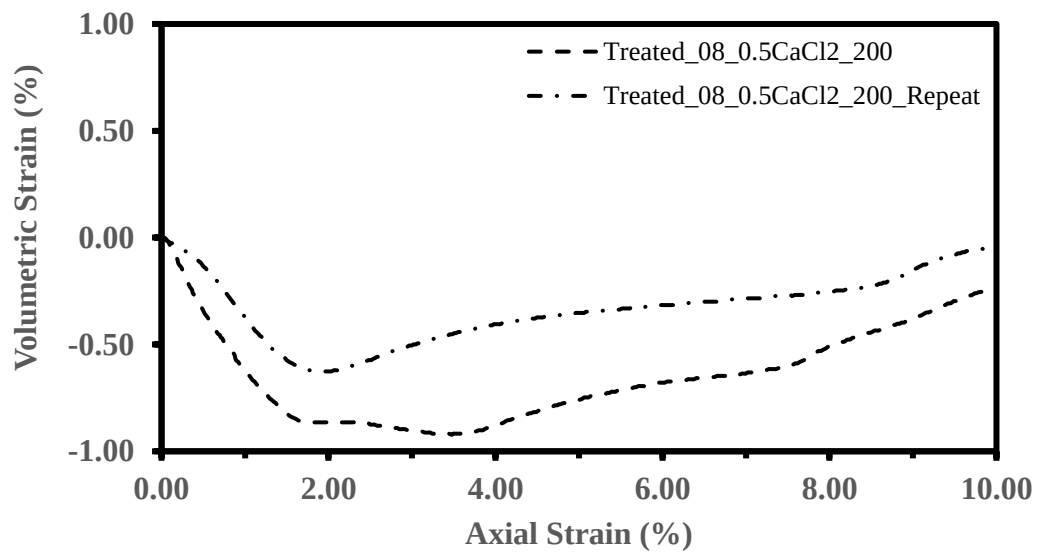
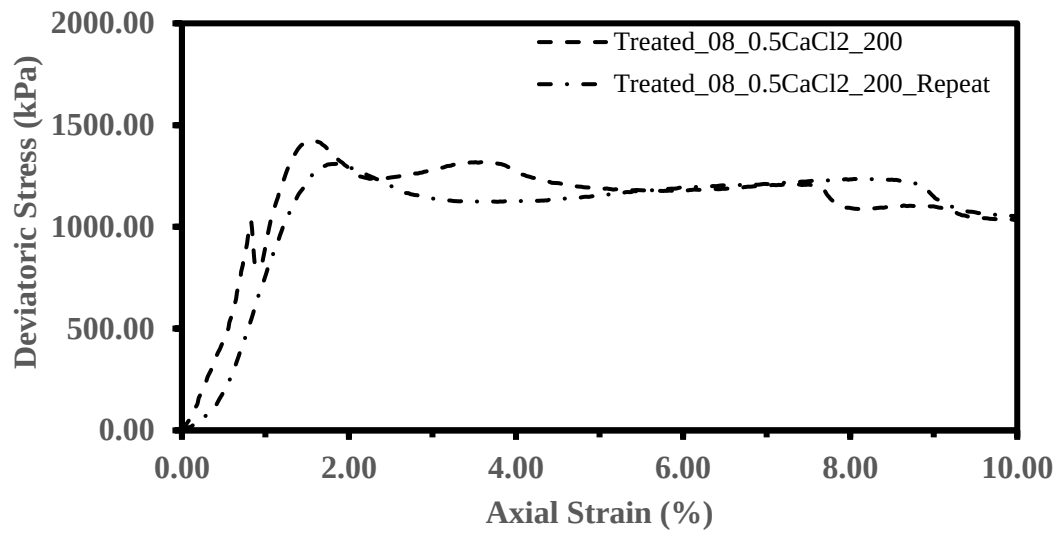


Figure A.3 Stress vs. strain and volumetric strain vs. axial strain curves for 8 injections – 0.50 M case at 200 kPa effective stress

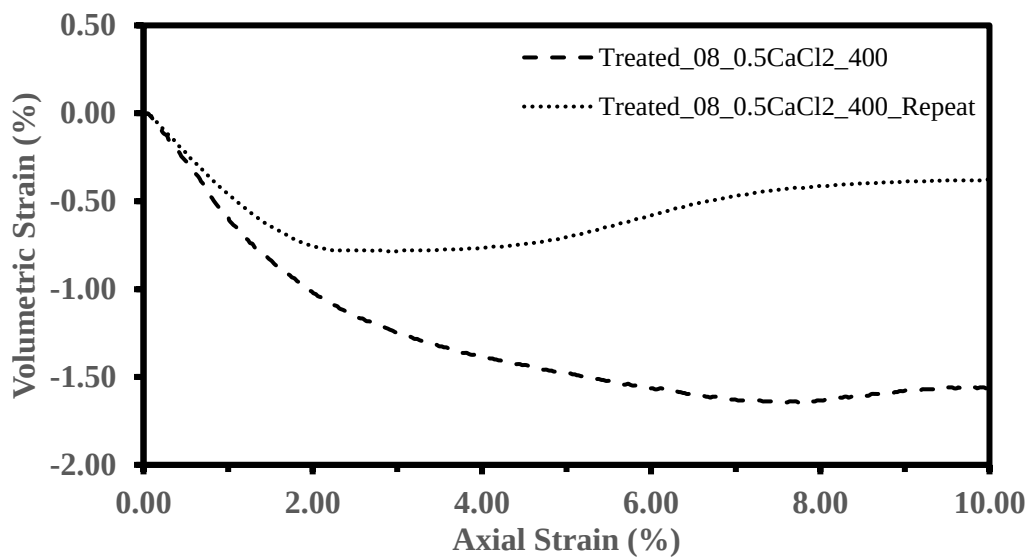
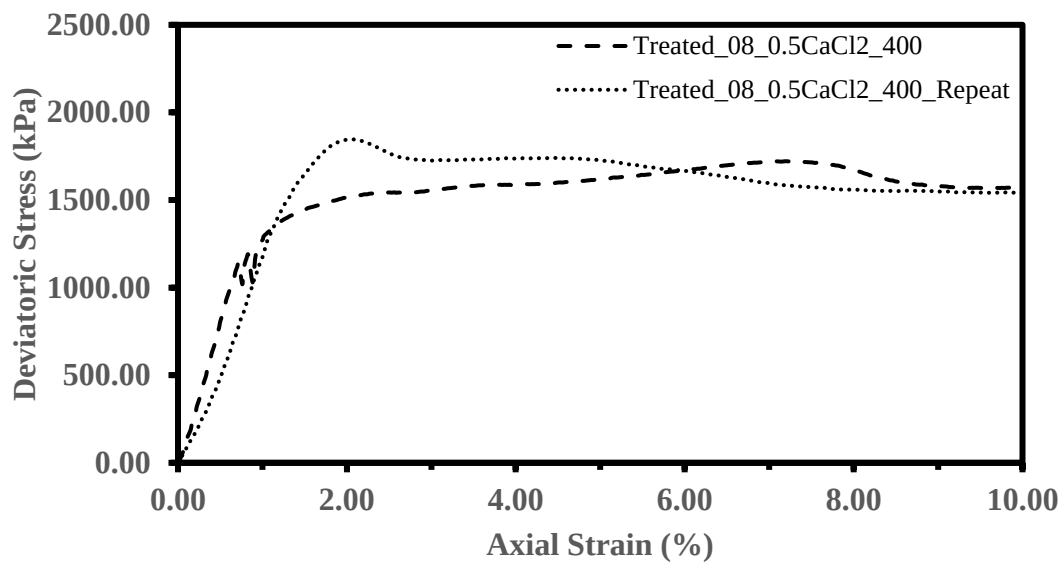


Figure A.4 Stress vs. strain and volumetric strain vs. axial strain curves for 8 injections – 0.50 M case at 400 kPa effective stress

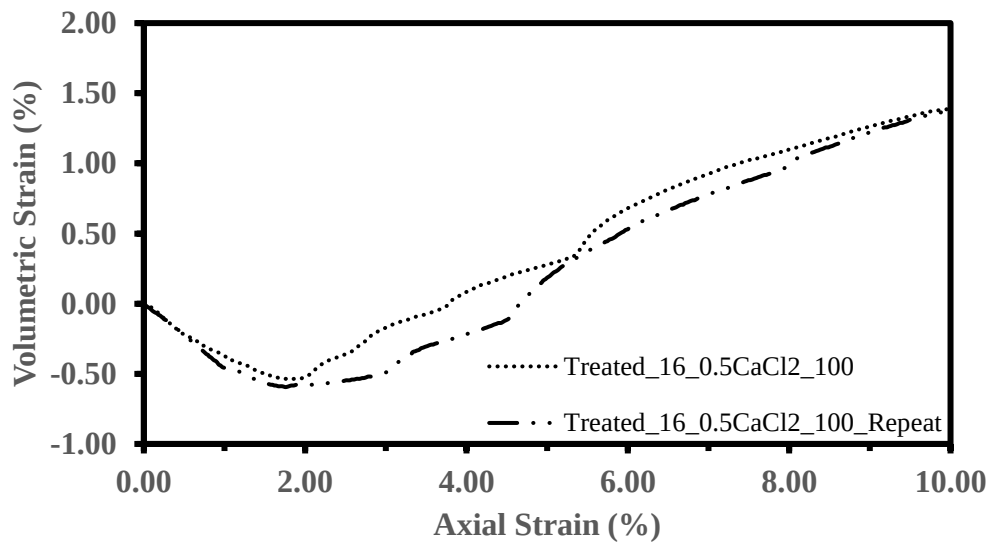
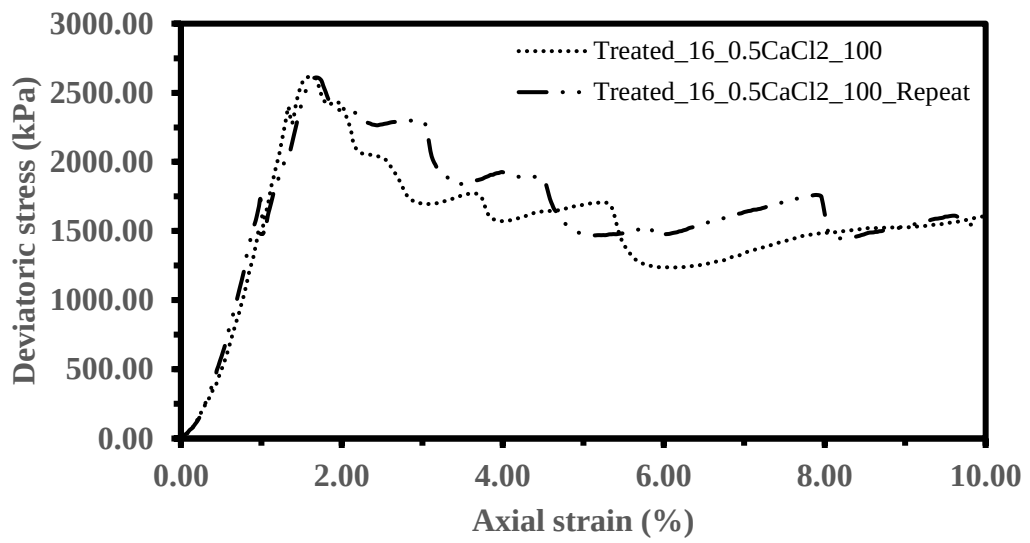


Figure A.5 Stress vs. strain and volumetric strain vs. axial strain curves for 16 injections – 0.25 M case at 100 kPa effective stress