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Biocementation process of soils: study of mechanical proprieties evolution of biocemented sand

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If it doesn’t challenge you it won’t change you

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Author

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Third Chapter: Results and discussion

Abstract

This master thesis explores the performance of small scale cemented soil columns produced using surface percolation with cement resulting from bacterially mediated reactions that precipitate calcium carbonate, a process often referred to as bio-cementation.

Bio-cementation has received considerable research attention over the last decade as it has the potential to complement existing ground improvement techniques and mitigate environmental concerns with currently used materials. Previous research has concentrated on pumping and injection techniques because of concerns that bacteria will be unable to survive the stresses associated with industrial mixing processes, however it has been difficult to create uniform bio-cemented soil masses.

This work was done as part of the end of study project. The objective of this work is to study the process of the biocementation method called MICP (Microbial induced carbonate precipitation), and to evaluate the mechanical properties of Biocemented sand (friction angle , cohesion) in laboratory, thus observations by micro-tomography X Ray and SEM to have a basic idea about microstructure behaviour of treated sand. We have proposed a model (an empirical law) depending on the evolution of soil elastic modulus (E) as a function of the percentage of calcite. This model can predict the value of the stiffness of biocemented sand by knowing the percentage of calcite available in the material without the need to perform tests in the laboratory

Key words: Microbial induced carbonate precipitation (MICP), *Sporosarcina pasteurii*, soil reinforcement, calcite precipitation, surface percolation.

ملخص

تستكشف هذه الأطروحة الرئيسية أداء أعمدة التربة الأسمنتية الصغيرة الحجم التي يتم إنتاجها باستخدام ترشيح السطح بالإسمنت الناتج عن تفاعلات بوساطة بكتيرية تترسب كربونات الكالسيوم ، وهي عملية يشار إليها غالبًا باسم التماسك الحيوي.

تلقى الأسمنت الحيوي اهتمامًا بحثيًا كبيرًا على مدار العقد الماضي لأنه يتمتع بإمكانية استكمال تقنيات تحسين الأرض الحالية وتخفيف المخاوف البيئية بالمواد المستخدمة حاليًا. لقد ركزت الأبحاث السابقة على تقنيات الضخ والحقن بسبب المخاوف من أن البكتيريا لن تكون قادرة على التغلب على الضغوط المرتبطة بعمليات الخلط الصناعية ، ومع ذلك فقد كان من الصعب إنشاء كتل تربة أسمنتية حيوية موحدة تم هذا العمل كجزء من نهاية مشروع الدراسة. الهدف من هذا العمل هو دراسة عملية طريقة التخليق (ترسيب كربونات المستحث الميكروبي) ، وتقييم الخواص الميكانيكية MICP الحيوي التي تسمى للرمل المستحضر الحيوي (زاوية الاحتكاك ، التماسك) في المختبر ، وبالتالي عمليات الرصد بالأشعة أن يكون لديك فكرة أساسية عن سلوك البنية المجهرية للرمال المعالجة. لقد SEM و X Ray المقطعية كدالة لنسبة الكالسيت. يمكن (E) اقترحنا نموذجًا (قانونًا تجريبيًا) يعتمد على تطور معامل مرونة التربة لهذا النموذج أن يتنبأ بقيمة تصلب الرمل الحيوي من خلال معرفة نسبة الكالسيت المتوفرة في المادة دون الحاجة إلى إجراء اختبارات في المختبر.

الكلمات المفتاحية : Sporosarcina pasteurii,تعزيز التربة – ترسيب الكالسيت - التقطيرالسطحي

Résumé

Cette thèse de master explore les performances de colonnes de sol cimentées à petite échelle produites en utilisant une percolation de surface avec du ciment résultant de réactions à médiation bactérienne qui précipitent le carbonate de calcium, processus souvent appelé biocementation.
La recherche sur la biocimentation a fait l'objet d'une attention considérable au cours des dix dernières années, car elle pourrait compléter les techniques d'amélioration des sols existantes et atténuer les préoccupations environnementales avec les matériaux actuellement utilisés. Des recherches antérieures se sont concentrées sur les techniques de pompage et d'injection, craignant que les bactéries ne puissent pas survivre aux stress liés aux processus de mélange industriels. Cependant, il a été difficile de créer des masses de sol uniformes biocimentées. L'objectif de ce travail est d'étudier le processus de la méthode de biocementation appelée MICP (microbialy induced carbonate precipitation), et d'évaluer les propriétés mécaniques du sable bio cimenté (angle de frottement, cohésion) en laboratoire. SEM doit avoir une idée de base du comportement de la microstructure du sable traité. Nous avons proposé un modèle (loi empirique) dépendant de l'évolution du module d'élasticité du sol (E) en fonction du pourcentage de calcite. Ce modèle permet de prédire la valeur de la rigidité du sable biocimenté en connaissant le pourcentage de calcite disponible dans le matériau sans qu'il soit nécessaire de réaliser des tests en laboratoire.

Mots-clés: Sporosarcina pasteurii, Précipitation de carbonate d'origine microbienne, renforcement des sols, précipitation de calcite, percolation de surface

General introduction

In many regions around the world, the mechanical properties of soils are insufficient for the desired land use: roads and railways underground settlement and require continuous maintenance. Dikes, dunes, and slopes can become unstable and slopes, coasts, and rivers can be subjected to erosion phenomenon. Earthquakes can cause liquefaction of loose sediments and consequently damage to constructions on top of it. Water and oil production wells in loosely cemented sediments often produce sand, of which removal is a costly process and in land reclamation projects the compaction of the recovered land is sometimes a major concern.

Stabilization of soil (ground improvement) can be desirable for these applications. Before and during construction, soil stabilization is often applied at or from the surface by using constructive approaches like compaction, installing nails, sheets, or piles, or mixing the soil with lime or cement (Karol, 2003). When stabilization of a soil mass is required deeper in the underground these surface techniques are insufficient and strengthening techniques, like deep mixing, cement or chemical grouting or ground freezing are being used.

Traditionally, geotechnical engineers assume soil and its behaviour are abiotic even though geologists and soil scientists have long recognized the influence microbiology has on the formation of soil, soil fabric, and soil properties. Recently geotechnical engineers acknowledged the presence of biological activity in the subsurface, and the potential effect it has on soil behaviour. This is in large part probably due to a research work published by Mitchell and Santamarina (2005) that outlined potential biological influences in the subsurface that could change soil properties and be utilized by engineers.

This new research field, focusing on harnessing biological activity to manipulate the local geochemistry and improve the mechanical properties of the soil, bio-mediated soil improvement research is at the convergence of microbiological, geochemical, and geotechnical engineering research. A fundamental understanding of microbiological and geochemical principals is essential to understand the governing mechanisms responsible for producing the desired engineering properties. The research presented in this dissertation has been conducted from a geotechnical engineering perspective, yet significant effort was spent understanding and manipulating the biogeochemical aspects of the treatment process to facilitate practical and reliable treatments improvement.

Calcite ($CaCO_3$) is one of the most common and widespread minerals on Earth, constituting 4% by weight of the Earth's crust. It is naturally found in extensive sedimentary rock masses, as limestone, marble and calcareous sandstones in marine, freshwater, and terrestrial environments (Hammes and Verstraete, 2002; Klein and Hurlbut, 1999).
Bacterial contribution to these extensive formations had been suspected for some time (Drew, 1910) but remained controversial until recent investigations involving the microbial pathways and the required precipitation conditions, indicated that bacteria have the potential to far exceed the abiotic contribution to calcium carbonate deposition in most environments on Earth (Castanier et al., 2000).
Microbial induced carbonate precipitation (MICP) technique has been widely used in various engineering applications, such as evolution of water resources and rehabilitation of old buildings. In addition, this method has been applied to ground improvement as well. For example, Zhou, Luo, and Wang (1997) found that the physical and chemical changes of organic matter and microorganisms induced by the environmental change would have an impact on the properties of geomaterials. Xu, Zhang, and Zhou (2009) extracted carbonate mineralization bacteria and polysaccharide of the adhesive bacteria from soils to improve the related engineering properties of silty soils. Al-Thawadi (2008) and Burbank et al. (2011) isolated the original ecological urease-producing bacteria that provide bacteria species for the application of MICP technique.
The bacteria were then used to improve the geotechnical properties of sandy soils successfully. In addition, the broad application of MICP technique and the potential benefits of the microbial reinforced soils attract more scholars to participate in this research area (Mujah, Shahin, and Cheng 2016).
In this study, review of ground improvement using MICP technique is performed. The mechanism of MICP-treated soils is systematically introduced, followed by the review of many aspects of MICP technique. As compared to the traditional ground improvement methods, cost analyses of the technique are then presented. The problems encountered in current theoretical and experimental studies, and the engineering applications of the technique are presented and discussed. Some recommendations are provided for future development as well.

On these papers, our work is devised into three chapters.
First chapter is a review of the work contributing from literature to the bio-mediated soil improvement field from the biological, geochemical, and geotechnical perspectives. The second chapter is the simulation of laboratory including soil characterisations essays and mechanical tests for the preparation to treat sand with MICP. The last chapter will contain results obtained from previous chapter's tests with some discussions and perspectives.

First chapter

Scope of Research

I. Introduction:

In geotechnical engineering, Soil reinforcement is necessary in lands where possibilities of erosion are high, it is a method concerned with the increase of strength properties of soil such the mechanical properties (cohesion, internal friction angle) by increasing micromechanical properties (contact surfaces and coordination number).
The Geosynthetics are most commonly used in soil reinforcement due to their cost-effective, more profitable, and highly adaptable. The physical properties like Stiffness, Compressibility, and Strength are some of the few important parameters to be considered of the many methods involved in improvement of soil properties.
Recently, a new revolutionary promising technique in geotechnical engineering gains a lot of attraction, called the Microbial Induced Calcite Precipitation (MICP).
This innovative soil improvement technique is capable of enhancing the strength and stiffness of soils, and controlling their hydraulic conductivity. These mechanical and physical properties of soils after MICP treatment are affected by many factors, such as $CaCO_3$ content, amount, and distribution in the soils.
Microbial-induced carbonate precipitation (MICP) is considered one of the more promising bio-mediated soil improvement techniques being investigated today (Martinez et al. 2013). MICP occurs through biologically driven urea hydrolysis, which primes soil conditions for calcium carbonate precipitation at particle-particle contacts by producing carbonate in the presence of calcium according to (Stocks-Fischer et al. 1999).

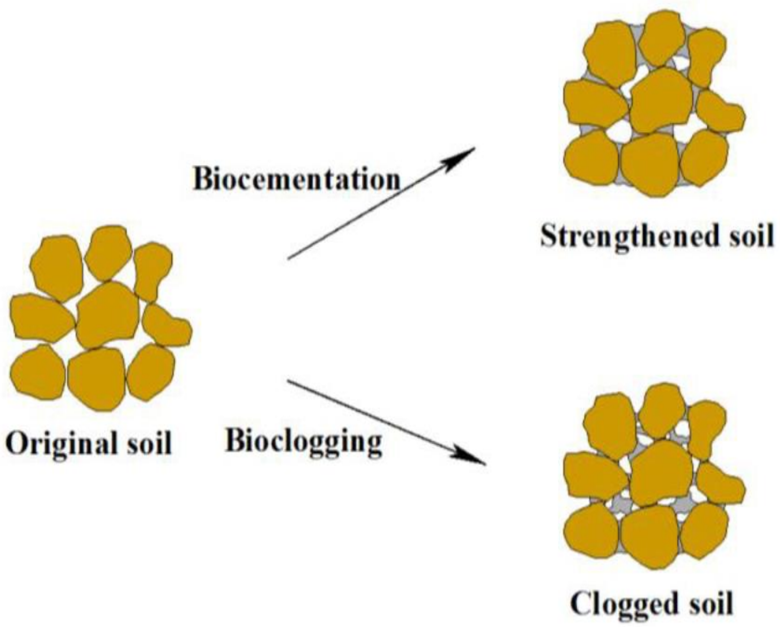

Figure 1: Schematic Illustration of bio cementation and bioclogging process by (Chu 2016)

I.1 Aspects of reinforced Soil:

Currently, the majority of soil improvement techniques require substantial energy for material production and/or installation (DeJong et al., 2010). Injecting synthetic man-made materials such as micro-fine cement, epoxy, acrylamide, phenoplasts, silicates and polyurethane (DeJong et al., 2010) is one of the most commonly used soil improvements methods in civil, geotechnical, and mining engineering applications.
Grouting can substantially increase the stability of soil with both enhanced shear and bearing strength properties by binding soil particles together. This is accomplished using a variety of compaction, jetting, and permeation grouting techniques (Warner, 2004; DeJong et al., 2010).

a) **Compaction grouting:**

A Compaction grouting is a soil injection with low workability cement paste that remains homogeneous without entering in the soil pore.
This method displaces the soil and is commonly used to remediate soil deficiencies under structures that have undergone settlement (Warner. 2004).

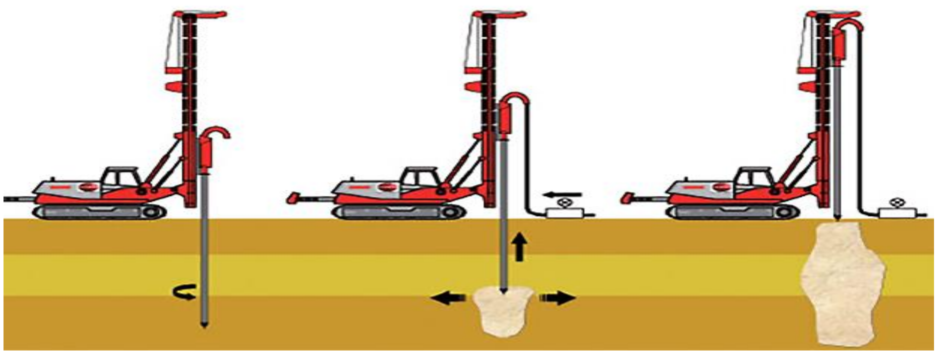

Figure2: compaction grouting

b) **Jet Grouting:**

This method consists of mixing grouting fluid with in-situ soils though turbulence caused by high-pressure jetting together with rotation of the nozzle (Warner, 2004).
High pressure fluid jets are used in jet grouting to erode and mix/replace soil with grout.

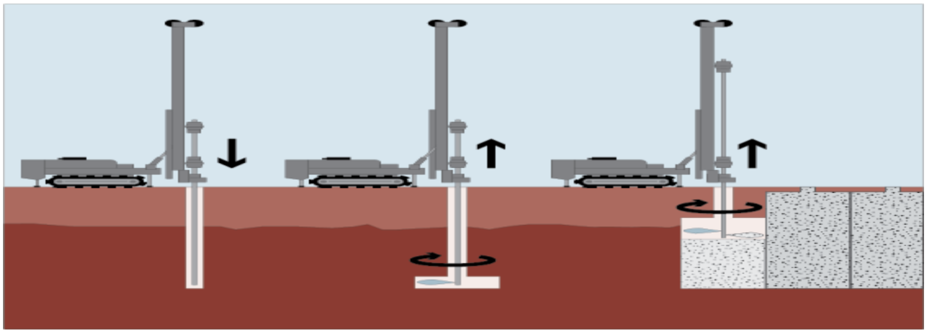

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Figure 3: jet grouting

c) **Permeation Grouting:**

This application consists of pumping and ejecting the grout from a nozzle at the end grout pipe to permeate the surrounding soil. The permeated grout fills up the pore voids between the soil grain interfaces (DeJong et al., 2010)
Apparently, permeation grouting helps to increase the soil strength while decreasing the permeability. This technique is most suitable for granular soils (i.e. sand or gravels) with high permeability due to the large pore spaces. The high permeability allows the grout to be ejected with lower pressure and penetrate to greater depth.

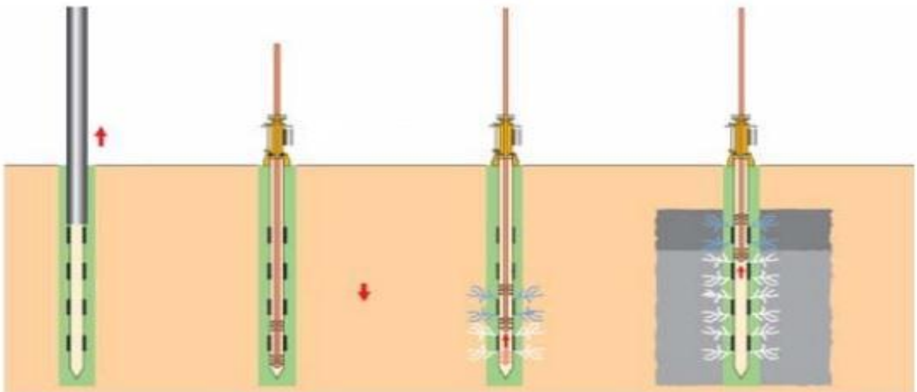


Figure 4: Diagram Showing Permeation Grouting

According to van Paassen (2009), traditional soil improvement methods have several limitations. The effective treatment distance of the grouting methods is up to 1-2 m from the injection point due to the limitation of the mixing equipment (DeJong et al., 2010). In general, grouting treatment methods require high pressure to introduce grout into soil. These techniques are time consuming, expensive, require heavy machinery, and are environmentally detrimental. In addition, commonly used cementitious grouts significantly reduce the permeability of the treated soil, which results in a limited injection distance.

I.2 Microbial-induced carbonate precipitation (MICP) process:

I.2.1 'Sporosarcina pasteurii bacteria' and urease:

Microbial-induced carbonate precipitation (MICP) has been the subject of research for several industrial applications. Several researchers have shown that MICP can be used to improve the mechanical properties of porous materials(Victoria S. Whiffin, van Paassen, and Harkes 2007). This technique based on the precipitation of calcite with an aid of certain bacteria called "Sporosarcina pasteurii." This last can decompose the urea $(NH_2)_2CO$ into ammonium ($NH4^+$) and carbonate (CO_3^{-2}) and by the injection of the ureasic bacteria and chemical reactants (Urea +calcium source) this chemical component can participate in the composition of the final product (calcite: CaCO3).
This phenomenon can be reproduced within soils, by the injection of the ureasic bacteria and chemical reactants (Urea + calcium source).

$$\mathbf{(NH_2)_2CO + 2H_2O + CaCl_2 \rightarrow CaCO_3 \downarrow +2NH_4Cl}$$

As (Whiffin 2004) mentioned in her thesis ,S. pasteurii has been described with the ability to constitutively express high levels of urease, so one of the roles of urease plays for it is to increase the external pH to 9.25 thus creating an environment conductive to growth , fig 3.

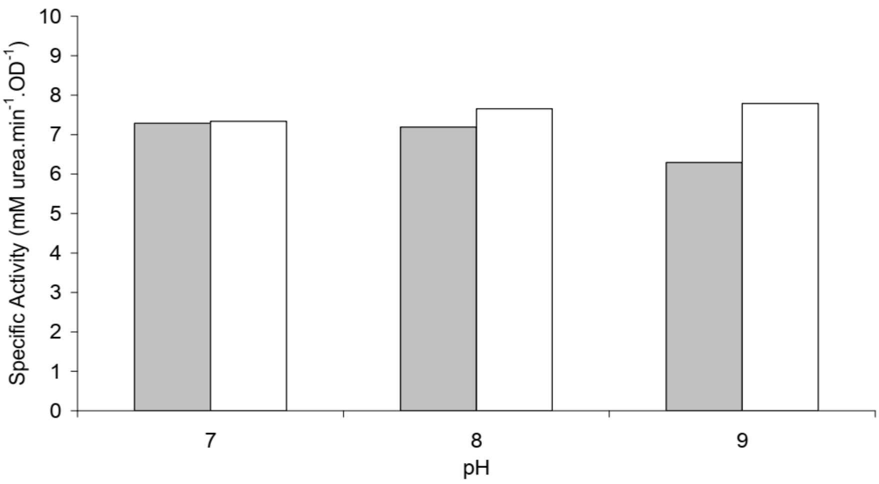


Figure 5: Initial specific urease activities immediately after inoculation into various pH media. Urease activity measured at each pH (□) compared with urease activity corrected for the biochemical pH effect (□).(Whiffin 2004)

I.2.2 Plants and urease:

Nowadays, the urease enzyme is widely used in different fields of industries such as medicinal, construction, agricultural, food, etc.

Different kinds of ureolitic bacteria and micro algae, soil urease and plant urease have applied for the above fields.

The use of plant-derived urease enzyme as mentioned (Hamdan N., Kavazanjian Jr. E. 2013) to induce calcium carbonate ($CaCO_3$) cementation has been demonstrated through laboratory tests. Benefits of the use of plant-derived urease over the use of microbially-generated urease to induce carbonate cementation include the small size of the enzyme, which permits penetration into finer grained soils and makes the process less sensitive to bioplugging, and the availability of 100% of the carbon in the substrate for conversion to $CaCO_3$.

Some families of common plants are very rich in urease, including some varieties of beans, melons and squash, and the pine family. It includes jack beans (Canavalia ensiformis), soybean (Glycine max) leaf, and seed, pigweed (Chenopodium album), and mulberry leaf (Morus Alba) and they help to catalyse the reaction of urea hydrolysis to form ammonium and carbonate ions. Many researches are investigating new plant species like leaves other than the seeds for urease activity.

Table2: Urease activity in some plant species (Dilrukshia and Kawasaki 2016)

Plant species	Urease activity	unit
Canavalia ensiformis (Jack bean)	2700-3500	µmol urea/min.mg
Glycine max (Soy bean)	650-800	µmol urea/min.mg
Cajanus cajan(pigeon pea)	3120	µmol urea/min.mg
Gossyplum hirsutum (Cotton seeds)	14.5	µmol urea/min.mg
Rumex japonicus Houtt	42.2	µg NH4-N/hour. g
Mirabilis jalapa L	83.8	µg NH4-N/hour. g
Phytolacca americana L	26.1	µg NH4-N/hour. g

I.3 Biocementation by MICP for soil stabilization:

I.3.1 Materials and methods:

I.3.1.1 Biocemented samples preparation

The manners of preparation the samples for MICP process are several according to the conditions and abilities of laboratories. So in this part we are aiming to make a general idea about these methods for samples preparation.
In order to evaluate MICP as a soil strengthening process and ensure optimal results, sand of particle sizes ranging from 90 to 300 μm was used for all experiments as proposed (Al Qabany, Soga, and Santamarina 2012),
In the study of (Dadda et al. 2017) the samples preparation was formed by the follow: Two solutions: the first was as a bacterial solution which contains one optical density of Sporosarcina Pasteurii provided under a dried form by Soletanche Bachy (Soletanche Bachy Entreprise, Rueil-Malmaison, France) with 3 g of NaCl dissolved in one litter of commercial water, in order to increase the potential attachment of bacteria to soil grains. Second formed as reactant solution (calcifying solution) which contains 1.4 moles of urea and the same amount of calcium chloride. Fontainebleau sand (NE34) was used. Eight sand columns with the same diameter of 68 mm and different heights (4 columns have a height of 560 mm and 4 columns of 300 mm) were prepared with a pluviation technique in the plastic tubes of the injection system for this experimental investigation of the biocementation procedure. Fig 6

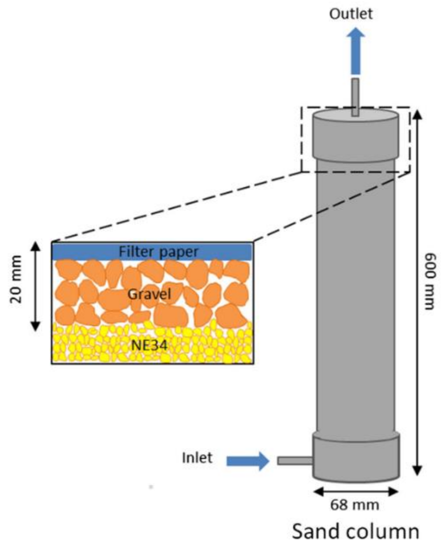


Figure 6: Injection protocol for biocementation process according to (Dadda et al.2017)

Other side like (Victoria S. Whiffin et al. 2007) proposed 5-meter-long PVC tube (internal diameter 66 mm) was positioned vertically and packed with 125–250 μm

Itterbeck sand (grain size characteristics: D_{10} = 110µm(10% of the grains have a diameter of this size or lower);
(D_{50} = 165 µm; D_{90} = 275µm) to a dry density of 1.65 g/cm^3 (porosity of 37.8%).
The column was positioned vertically with downward flow direction to avoid any settling of the packing material and generation of preferential flow paths that may occur if the column was positioned horizontally. Fig 5.

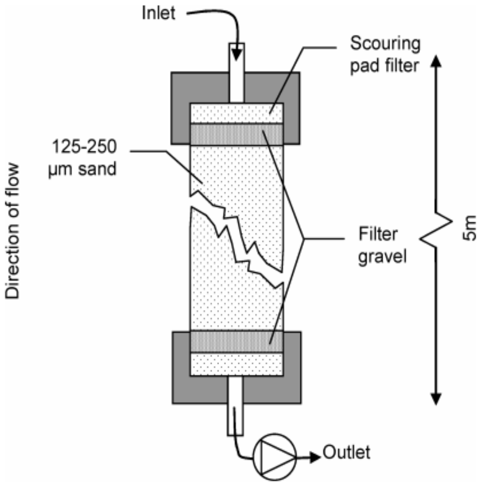


Figure 7: Schematic of column filter setup as (Whiffin et al.2007)

So generally samples preparation starts with packing the dry sand (fine and coarse) into kinds of columns with various amounts of water, were then flushed from top to bottom to provide the desired degree of saturation within the sand matrix.

I.3.1.2 Injection methods :

For Dadda et al. (2017) injection of one pore volume of the bacterial solution was performed from the bottom to the top with a flow rate of 0.2 mm/s. After one hour, two injections of calcifying solution of one pore volume were carried out with a flow rate of 0.14 mm/s and with a time offset of 10 hours between these two injections. After the second treatment, two pore volumes of flushing water were injected inside columns with a flow rate of 0.14 mm/s, in order to expel all the process residues.
This injection procedure usually leads to columns with a mass fraction of calcite of about 5–6%. This injection was repeated twice on eight columns in order to reach higher mass fractions of calcite, typically between 10 and 12%. The protocol of injection chowed on figure below. Fig 8

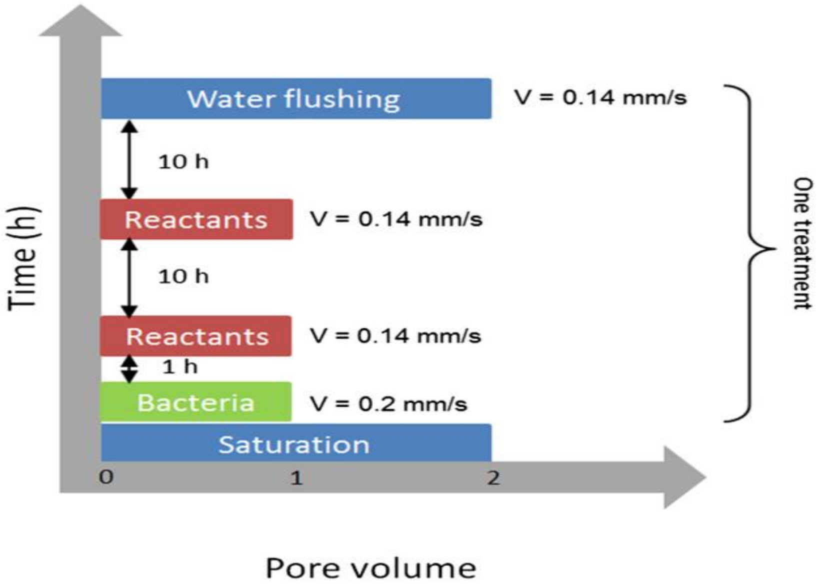


Figure 8: Injection protocol for biocementation process (Dadda et al. 2017)

For Whiffin et al. (2007) they made another way to inject, using five water pressure transducers were fitted to monitor water pressure inside the column at 0, 0.5, 1, 2 and 3 m from the top, of the column. In addition to these, the column was fitted with 10 pore fluid sampling ports (0.25, 0.5 and thereafter at 0.5 m intervals until reaching 4.5 m). Fluid reservoirs containing the injected fluids (water, bacteria, $CaCl_2$, Urea etc.) were connected at the top of the column.

A pump was installed at the bottom of the column to regulate the outflow rate and hydraulic head between free gravity flow of 1 L/h at a hydraulic head of 5 m when the pump was fully open and zero when the pump was fully closed. During the experiments the flow rate was kept constant at approximately 0.35 L/h. the table below shows details for this injection method.

Table3. Summer of column injections (Whiffin et al. 2007)

Phase	Description	Duration (h)	Flow rate (L/h)	Volume (L)	Details
Rinse	water flush	30.7	0.35	10.75	Tap water
Placement	Bacterial injection	18.1	0.35	6.34	OD_{600}: 1.583 Act: 0.23 mS/min
	$CaCl_2$ injection	17.1	0.35	5.99	0.05 M $CaCl_2$,
Cementation	Reaction fluid injection	24.9	0.35	8.72	1.1 M Urea and $CaCl_2$
	No flow—reaction	102	0	0	—
Rinse	water flush	23.7	0.35	8.30	Tap water

I.3.2 Calcium carbonate content :

$CaCO_3$ (calcite) amount may be achieved by many different processes. (Whiffin et al. 2007) Calcium carbonate content of the consolidated samples was determined by adding 2 mL of 2 M HCl solution into a 1–2 g dry sample and then measuring the volume of CO_2 gas with a U-tube manometer under standard conditions (25 ◦C, 1 atm). (Dadda et al. 2017) used the Bernard calcimeter basing on the dissolution of the calcite with HCl acid and the measurement of the released CO_2, which has the same molar quantity as $CaCO_3$. The measurement shall be repeated many times to assure a good currency of the results obtained.

I.3.3 Process monitoring:

Dejong et al. (2010) declare Control and management of bio-mediated soil improvement processes require real-time, non-destructive monitoring of chemical, biological, and geotechnical components.
Process monitoring of select chemical, biological, and geotechnical parameters is necessary to develop a full understanding of a given bio-mediated process.

I.3.4 Permeability measurements:

Permeability is a primary factor that controls the behaviour of porous materials under saturated conditions and thus dictates the suitability of a specific material for certain applications as (Shahin et al. 2011).
Laboratory determination of the permeability of the untreated and bio-cemented sands was conducted using a constant head permeability test with a rigid side wall device in accordance with the Australian standard. All specimens were saturated prior to the permeability test by flushing through 2 L tap water under 15 kPa back pressure (hydraulic head of about 150 cm) to remove most of the remaining pore air.
To compare the permeability of the MICP-improved soil with conventional soil improvement using chemical additives, (Cheng, Cord-Ruwisch, and Shahin 2013) prepared a series of mixtures of fine sand with various proportions of Portland cement were prepared and tested for their strength and permeability. The details of the Portland cement samples are listed in table 4. However, inhomogeneity along the sand column samples can still be attributed to the localized clogging. It is thereby recommended that a low concentration solution should be used if less permeability reduction is desired, to ensure a uniform consistency of CaCO3 precipitation.
(Mujah, Shahin, and Cheng 2017) proposed a solution with low concentration may produce more uniform precipitation pattern and stronger samples for a given amount of CaCO3 precipitation.

Tab4.Mix proportions of Portland cement samples (Cheng et al. 2013)

Mix ID	Cement (g)	Sand (g)	Water (mL)	Density (g/cm^3)
1	40	580	124	1.93±0.01
2	56	580	124	1.93±0.01
3	72	580	124	1.93±0.01
4	84	580	124	1.93±0.01

I.3.5 Triaxial compression tests:

A triaxial compression test for was carried out by (Cheng et al. 2013) to provide verification for the MICP as a soil stabilization technique. This test is considered to be the most reliable one to measure the shear strength parameters of soils.

I.4 Scanning electron microscope (SEM):

Fractions of cemented samples, taken from different parts of sand column, were prepared and examined by scanning electron microscope.

To characterize the shapes and locations of the precipitated $CaCO_3$ and to investigate the bonding behaviour between the grain hosts and cement agent, microscopy analysis was conducted on the cemented soil samples, which were taken from the centre of the cemented sand columns. Before conducting the microscopy investigation, all samples were flushed with tap water and dried at 60 °C for 24 h. The microscopy investigation was carried out scanning electron microscopy (SEM) using a PHILIPS XL20 scanning electron microscope (Eindhoven, the Netherlands).(Cheng et al. 2013).

Other way, (Al Qabany et al. 2012) used a JEOL JSM-5800LV scanning electron microscope (SEM) where backscattered imaging was applied on these samples. Some samples were also sputter-coated with platinum or carbon using an Emitech K550 sputter coater to determine the most suit- able imaging method for detection of precipitation pattern. It was found that the backscatter detection technique was the most appropriate for the purpose of the imaging.

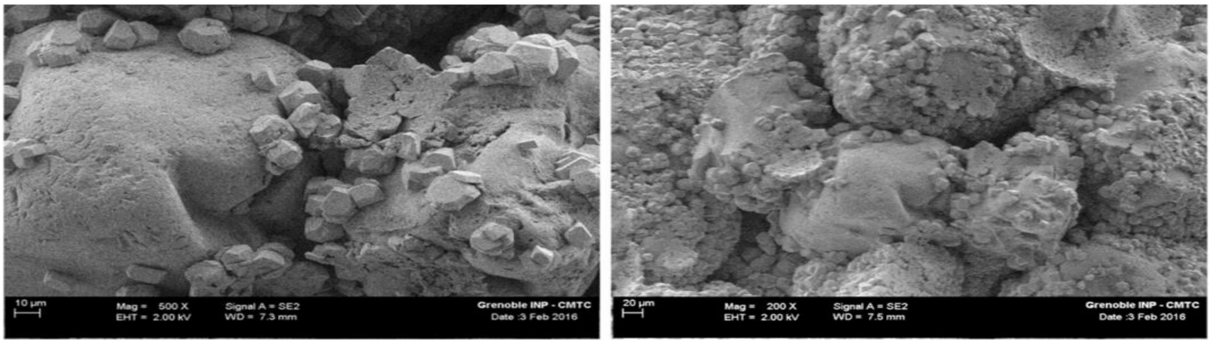


Figure 9: SEM observation of the biocemented sand by (Dadda et al. 2017)

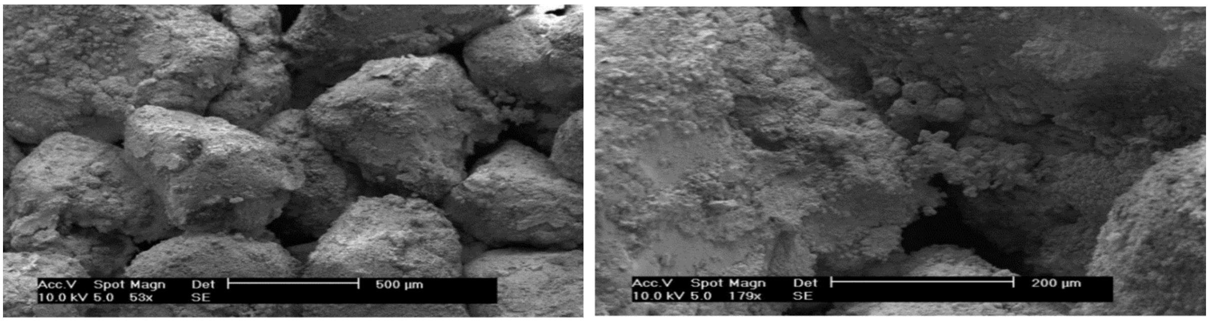


Fig. 10:Formation of $CaCO_3$ crystals for samples treated at 100% saturation (Cheng et al. 2013)

I.5 Review of some results from literature:

I.5.1 Factors affecting the performance of MICP:

I.5.1.1 Treatment formula and injection rate:

The rate of the calcium carbonate precipitation as Mortensen et al. (2011) reported must be controlled to achieve uniform cementation. Precipitation that occurs too quickly will result in localized cementation around the injection source, which may lead to plugging. Higher rates of calcite precipitation have been observed to lead to plugging at the injection source and larger gradient of mass of calcite along the injection path.

A slower rate of precipitation allows for the nutrient delivery to more distant locations along the flow path and a more uniform delivery of the chemicals. The rate of precipitation is dependent on the delivery rate of chemicals and the rate of pH rise to trigger precipitation. Chemical delivery rate in situ is largely dependent on the flow rate from the injection source.

At slow flow rates, the rates of urea consumption and calcium carbonate precipitation are larger than the flow rate, resulting in cementation immediately adjacent to the

injection source.The urea and aqueous calcium are consumed before they reach biological communities farther along the flow path.
Conversely, increasing the flow rate to exceed the rate of urea consumption and calcium carbonate precipitation allows for a more uniform distribution of chemicals along the entire flow path length.

I.5.1.2 Temperature

The microbial activity and growth are less sensitive to the temperature within the range of 20 to 30 °C. The rate of urea hydrolysis is marginally higher in 30 °C, as compare to 20 °C. Increment in temperature after 30 °C does not promote the decomposition rate any further (Nemati, Greene, and Voordouw 2005). It is, however, impractical to alter or control the soil temperature while the MICP treatment is performed on soil specimen or in situ. It is suggested to select a calcite forming bacteria that live optimum in soil temperature. With (Ng, Lee, and Hii 2012). The soil temperature varies with latitude, altitude, incident solar radiation, moisture content, conduction, type of soil, depth of soil and etc.

I.5.1.3 PH:

The calcite precipitation commences when urea is decomposed by urease enzyme. The urease enzyme is produced by microbial metabolic activities and as a result, urea hydrolysis is preferable around the cell.
(Stocks-fischer, Galinat, and Bang 1999) found that the urease activity increased rapidly from pH 6.0 to 8.0. Urease activity reached its peak at pH 8.0 and decreased gradually at higher ph. Nevertheless, promising level of urease activity is still available at pH 9.0. The pH of reactant medium will increase gradually during the MICP process. In addition a preliminary study carried out by Cheng et al. (2014) pointed out that the relationship between the initial soil pH and formation of $CaCO_3$ crystals is a function of the $CaCO_3$ solubility variation generated as a result of the different initial pH values. Until a proper SEM image is made to examine the $CaCO_3$ crystals precipitation patterns under the effect of super saturation condition (i.e. the change in pH value), debates regarding this issue will continue.

I.5.2 Effect of Cementation on Engineering Parameters:

MICP can result in the improvement of a variety of soil properties including permeability, stiffness, compressibility, shear strength, and volumetric behaviour.
(Whiffin et al. 2007) show that lower concentrations of calcium carbonate (below 60 kg/m^3 or 3.5% w/w) had no significant effect on strength or stiffness properties

relative to untreated sand. At calcium carbonate contents above this value, a clear improvement was evident that was proportional to the amount of precipitate present. After the initial strength measurement, the residual strength after failure was also determined and in all samples this value approximated the strength of untreated sand. This indicated that any strength improvement given by the treatment was lost after failure and thus the material was more characteristic of rock than soil.

In future experiments it would be useful to extend the upper range of calcium carbonate precipitated, to give a broader understanding of the relationship between strength/stiffness and calcium carbonate content. It provides also that Cementation of particles together by calcite precipitation increases soil strength. Furthermore (Dejong et al. 2010) mentioned that the cementation increases the initial stiffness of soil at small strains and the maximum deviatoric stress and assure that bio-mediated calcite precipitation can effectively be captured throughout treatment using bender elements.

I.5.2.1 Effect of Cementation on Porosity:

Porosity is the amount of voids in a material. (Qian, Pan, and Wang 2010) characterized the effectiveness of cementation in terms of the porosity of cemented sand samples and its reduction, and found that the porosity was reduced to 25% after MICP treatment, although the porosity value was reduced, the $CaCO_3$ precipitates were found to fill the soil pores of sand grains. It should be noted that the porosity governs the effectiveness of MICP treated samples by means of controlling the replacement of the pore content of sand grains by $CaCO_3$ (Rong, Qian, and Li 2012). As the degree of cementation increases, the amount of precipitated $CaCO_3$ increase and higher amount of $CaCO_3$ crystals replaces the pore content of the inner structure of the soil matrix, leading to higher strength by (Mujah et al. 2017)

(Whiffin et al. 2007) determined porosity from the wet and dry densities during strength testing. The presence of calcium carbonate had a clear effect on porosity of the material and a reasonably linear relationship between the two parameters was observed. At the maximum calcium carbonate content (105 kg/m^3 $CaCO_3$) the column porosity was decreased to 90% of the untreated material (Figure10)

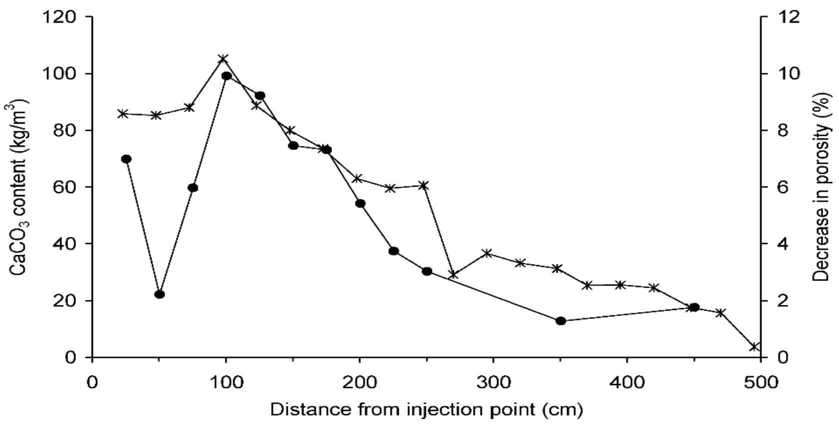


Figure 11: Relative decrease in porosity (•) versus calcium carbonate content (∗) over the length of the column by (Victoria S. Whiffin et al. 2007)

I.5.2.2 Shear Strength Improvement:

Shear strength is the magnitude of shear stress that a soil can sustain and depends strictly on the shear strength parameters of soil including the cohesion (c) and friction angle. It was found by (Mujah et al. 2017) that the shear strength of biocemented soil was strictly affected by the increase in soil cohesion resulting from the increase in the cement content, while the friction angle was not greatly affected by the cementation process. In addition (Lee Min Lee et al. 2012) show in the figure below that shear strength improvements of the bio-mediated soils under various concentrations of cementation reagent.

The unconfined compressive strength of the original soil, c_u was 33.1 kPa.

The soil treated with cementation reagent only (control specimen) showed a marginal improvement in shear strength (38.3 kPa) implying that natural urease-producing bacteria exist in the residual soil at an insignificant amount. For the soil treated with 0.25 M of cementation reagent, the shear strength improved significantly to 60.2 kPa, yielding an improvement of approximately 82%. The shear strength of soil was further improved to 64.8 kPa with increased concentration of the reagent up to 0.5 M. However, the improvement of the bio-mediated soil was retarded when using the 1.0 M cementation reagent. The shear strength of the treated soil was almost identical to that of control specimen. These results are in good agreements with the findings reported by De Muynck et al, who found that higher concentration of urea and calcium chloride would increase the amount of composited calcite. However, at high salinity (i.e. 1 M), inhibitory effect was observed in the microbial activities, and hence retarded the calcite precipitation.

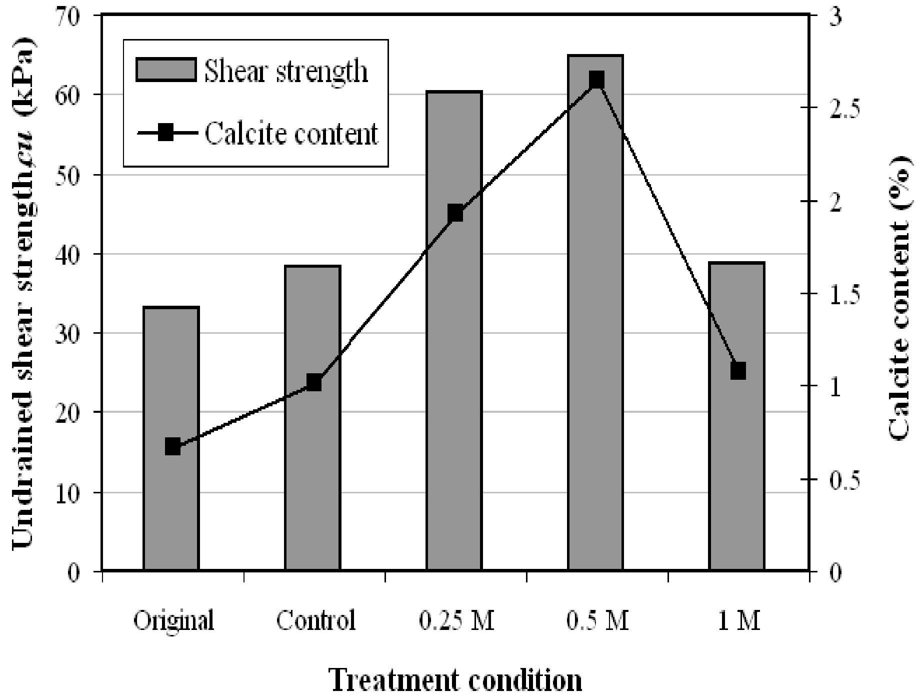


Figure 12. Shear strength and calcite content (Lee Min Lee et al .2012)

I.5.2.3 Effect of MICP-treated sand on permeability:

Most previous reported studies on MICP soil treatment have proven successful in improving the engineering properties of sand, it can be seen that a reduction in permeability was encountered for all bio-cemented sand samples. In contrast to the phenomenon reported by Whiffin et al. (2007), the permeability decreased with an increase in $CaCO_3$ content for both fine and coarse sands, irrespective ofthe saturation degree. Results suggest that it is preferable to conduct the MICP process under lower saturation conditions, as it enabled improved mechanical behavior at the same time as maintaining relatively high residual permeability. Figure 11 shows the results of comparison between sand samples treated with Portland cement and bio-cement obtained by (Cheng et al. 2013). It can be seen that the bio-cemented samples have higher strength in the range of lower cement agents content (<0.1 g/g sand) compared to the Portland cement samples after 7 days of curing. However, this comparison would differ depending on the applied curing time of the Portland cement samples.

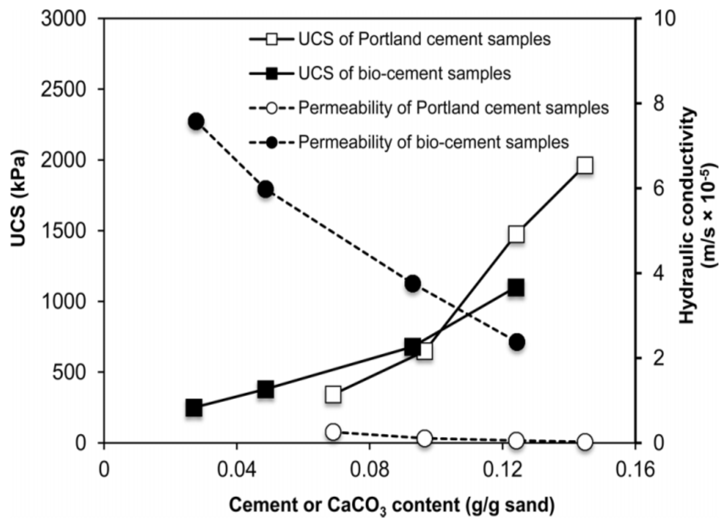


Figure 12: permeability of sand samples cemented with bio- cement $CaCO_3$ (100% saturation) and Portland cement (Cheng et al. 2013)

(Dadda et al. 2017) found that the evolution of the permeability ratio of the same sub-sample with and without calcite versus the volume fraction of calcite. A nonlinear decrease in permeability of the cemented sand was found with the increase in the cementation level (Fig. 13). This reduction, which is about 70% for a volume fraction of calcite of 14%, is mainly due to the reduction of the porosity of the porous media and the change in the microstructural properties with the calcite deposition. As mentioned in figure below.

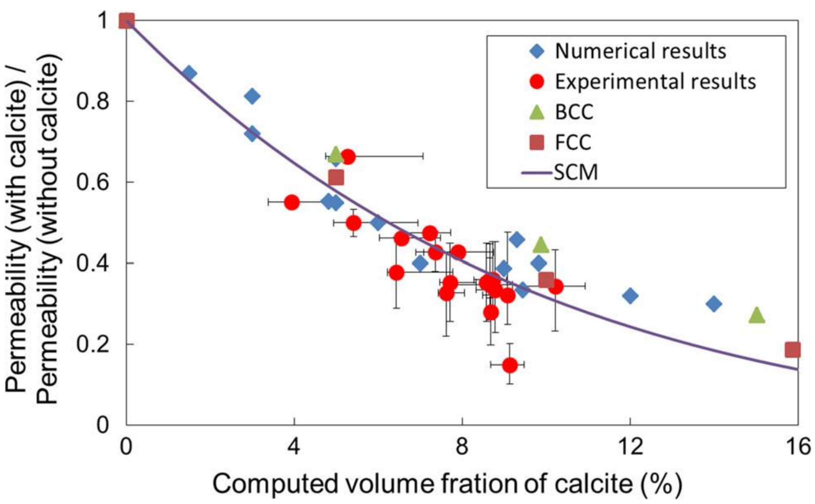


Figure 13. Evolution of the permeability ratio of the same sub-sample with and without calcite versus the volume fraction of calcite computed from 3D images (Dadda et al. 2017)

I.5.2.4 Stiffness:

Soil stiffness, commonly known as soil elastic modulus (E), is the ratio of stress over strain. Soil stiffness is closely related to the bonding strength between loose soil grains. Cheng et al. (2013) compared the elastic modulus of biocemented sand with other types of geomaterials such as concrete, gravel, and soft rock, and found that the bio treated sand is the most flexible among the materials tested. In earthquake prone areas, less stiff soil can provide an extra time for evacuation due to its ability to maintain significant residual strength even after failure.
(Min Lee Lee, Wei Soon Ng 2013) performed MICP on residual soil and found that the stiffness behaviour of biocemented residual soil is similar to that of biocemented natural sand. It was found also that strength and stiffness of cemented materials increase with the increase of the amount of cementing material in the soil matrix; although the amount of cementing material required to produce a certain cementing effect may vary.

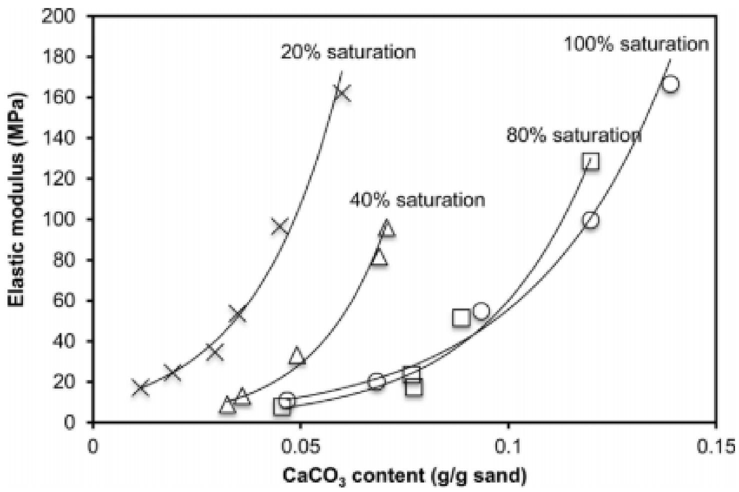


Figure 14: Variation of stiffness with CaCO3 content and different saturation conditions for coarse sand (Cheng et al. 2013)

I.6 Advantages of MICP for soil biocementation:

As opposed to other soil improvement techniques involving the use of cementation agents, MICP is currently relatively costly to be implemented in the field. The difference in the cost of various cementing agents toward soil improvement applications is compiled in Table 5.
Although the initial cost of MICP installation is relatively more expensive than other cementing agents, Whiffin et al. (2007) stated that MICP is cost-saving because the

bacterial enzyme can be reused in subsequent (two to three) applications of treatment using the same cementation solution. This means that MICP offers cheaper treatment in the long run. Similar observations were reported by (Al-Thawadi 2013).
(Dejong et al. 2010) also cited several advantages, he mentioned that the development of bio-mediated processes for soil improvement has several characteristics that may prove advantageous relative to industry standard soil improvement techniques. These include:

- Reduced costs
- The use of natural materials, reduced treatment injections, etc.
- Reduced impact to the environment
- The use of natural materials that do not permanently alter subsurface conditions
- Improved treatment uniformity
- Biological processes have potential to enhance spatial uniformity
- Optimal treatment concentration
- Degree of treatment can be controlled and monitored
- Adaptable duration treatments can be removed if only temporary support needed (e.g. by reversal of chemical processes)
- Hydraulic and mechanical control
- Degree of treatment can be adjusted
- Flexible implementation
- Methods can be used in new and retrofit construction
- Penetration into soils w/ fines

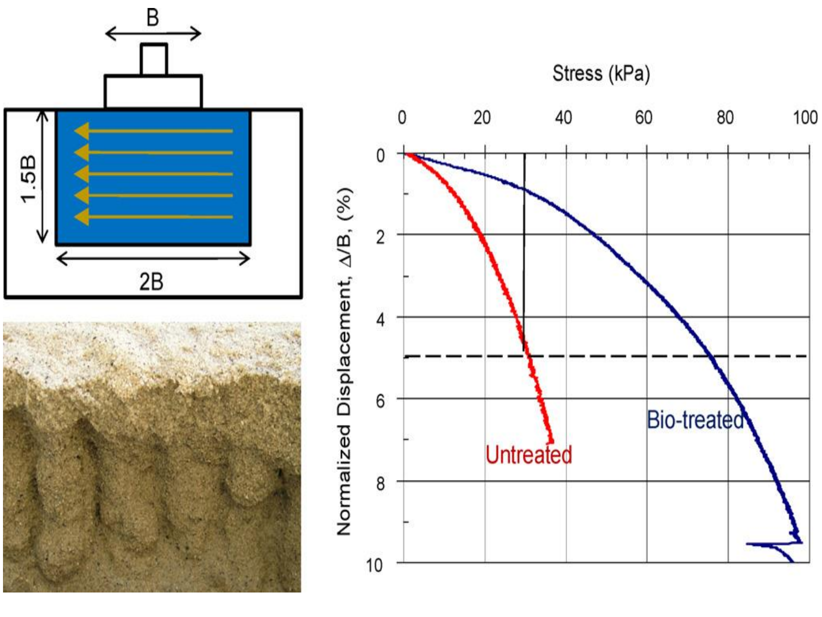
I.7 Applications of soil biocementation:

Once the MICP process has been fully optimized experimentally, further field applications can be realized. Although field applications of soil biocementation are still in their early stage of conception, more research is being tailored to examine the upscale effect of MICP process in longer soil columns and larger improved area. Thus, the envisioned applications by
(Mujah et al. 2017) in Table 5 of soil biocementation are important so as to open up more alternatives to the present research dealing with MICP.

Table 5. Envisioned applications for soil bio-cementation

Envisioned applications	Possible mechanism	References
Self-healing of soils	A portion of bio-cementation bonds degrade when loaded beyond its yield strength. The degraded MICP bonds can be healed by re-initiating the bio-geochemical process, returning the cemented sand properties to pre-shearing levels	Harbottle et al. (2014); Montoya and Dejong (2013)
Slope stabilization	The bio-cemented bonds help to strengthen the failure plane surface to provide additional stability needed to prevent slope failures	DeJong et al. (2010, 2013)
Settlement reduction	The bearing capacity of bio-cemented soils is increased; hence, settlement of foundation is reduced	DeJong et al. (2010, 2011); van Paassen et al. (2010)
Erosion control	MICP increases the bio-cemented soil resistance to the erosive forces of water flow along the sea shores and river banks	CheNg et al. (2014); DeJong et al. (2006)
Liquefaction prevention	Similar to the concept of self-healing, the post-shearing loads could re-initiate the MICP process; hence, preventing further liquefaction damages	Montoya and DeJong (2015); Montoya et al. (2013)

Furthermore a simple example of the improved application performance that can be realized is shown in Fig. 14a. A zone of soil beneath a model footing was treated with bio-mediated calcite precipitation. The settlement induced by loading of the footing was decreased by five times at a footing stress of about 30kPa. Details of the model shallow foundation test are presented in Martinez and DeJong on figure 14b.



(a) (b)

Figure 15. Experimental results from bio-mediated improvement of soil support a shallow footing foundation (Dejong et al. 2010)

I.8 Conclusion:

This chapter presents a review of new process called MICP as soil improvement technique. Precipitation of calcium carbonate by microbial methods made a significant improvement in soil strength without a major reduction in permeability. For ground improvement requirements, it is desirable to achieve this result at low injection pressures, which are acquired with relatively low flow rates (<10 meters per day). This study was conducted under such conditions and successful soil strengthening was achieved. In addition a clear critical aspect of this process has been identified. Balancing the rate of urea hydrolysis in the column with the delivery of reactants via the flow rate is essential to precipitate calcium carbonate at locations where strengthening is desired. When these two parameters are out of balance, a non-homogeneous result will be attained with higher strengths near the injection point. This work demonstrated that microbial carbonate precipitation can be applied for large-scale soil improvement work and further development of the technique for this application area is warranted.

MICP is a complex biochemical process that utilizes the urea hydrolysis that takes place between the sand particles for improvement of soil engineering properties. There is an increasing need for a ground development method, and one of the methods is to improve the strength of soil particles by utilizing the cementation technique. Even though there are various chemical methods available that are currently in practice, many of them have adverse environmental effects.

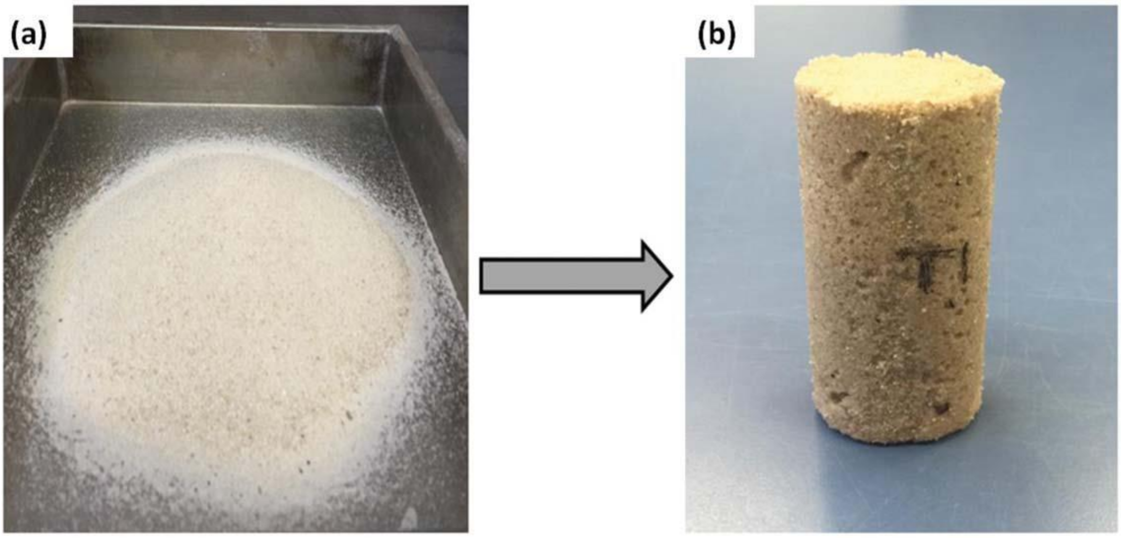


Figure 16. Sand metamorphosis: (a) natural sand; and (b) biocemented sand (bio sandstone) (Mujah et al. 2017)

Second chapter

Experimental program

II. Introduction:

We will present in this section the characteristics of the studied sand to trait with the MICP process and the experimental tests used. We will also briefly describe the test procedures performed as part of this work, as well as the experimental protocols developed in this study in addition some that will help us make a comparison between untreated sand and treated one with microbial induced carbonate precipitation process.
The objective of the present chapter is adopting material and methodology to trait our sand with MICP process.

II.1 Description of the studied sand

In this experimental study we chase El Hadjeb sand (Biskra/Algeria) with $D_{50} \simeq 240$ µm because it is a fine sand with narrow particle size distribution and almost like Fontainebleau sand (NE34) ($D_{50} \simeq 210 \mu$m) that been used in many research studies . This kind of sand is a potential target of the biocementation technique as a mitigation measure for liquefaction problems, it represents optimal sand for the biocementation process in terms of geometric compatibility between the pore, and the bacteria cell sizes.

II.2 Grain size analysis (NF P 94-056)

The test consists of dividing and separating a material into several grain size classifications of decreasing sizes by means of a series of sieves. The aperture sizes and the number of sieves are selected in accordance with the nature of the sample and the accuracy required. Snap the sieves used on top of each other in increasing order by a full-bottomed recipient (to collect the final items) and the top on puts a cover to prevent dust dispersion. On arrival at sieving pouring the dry material. We will recover a refusal sieve (part that remains through the sieve) and a portion of the sieve will be retained and a sieve which will let the elements under 0.08 mm pass. If the sieving is manual on the different sieves to pass the part to diameter from the sieve, the results are plotted on a semi logarithmic graph where they construct a Grain-Size Distribution curve. In the context of this work we have limited ourselves to carrying out only the size analysis by sieving, since the quantity of fine particles smaller than 80 µm was very small. The uniformity coefficient Cu is defined as the ratio of D_{60} by D_{10}. So when Cu is greater than 4 to 6, it is understood as a well graded soil and when the Cu is less than 4, they are considered to be poorly graded or uniformly graded. Uniformly graded in the sense, the soils have got identical size of the particles.

→ $C_U = D_{60}/D_{10} = 0.28/ 0.130 = 2.153$

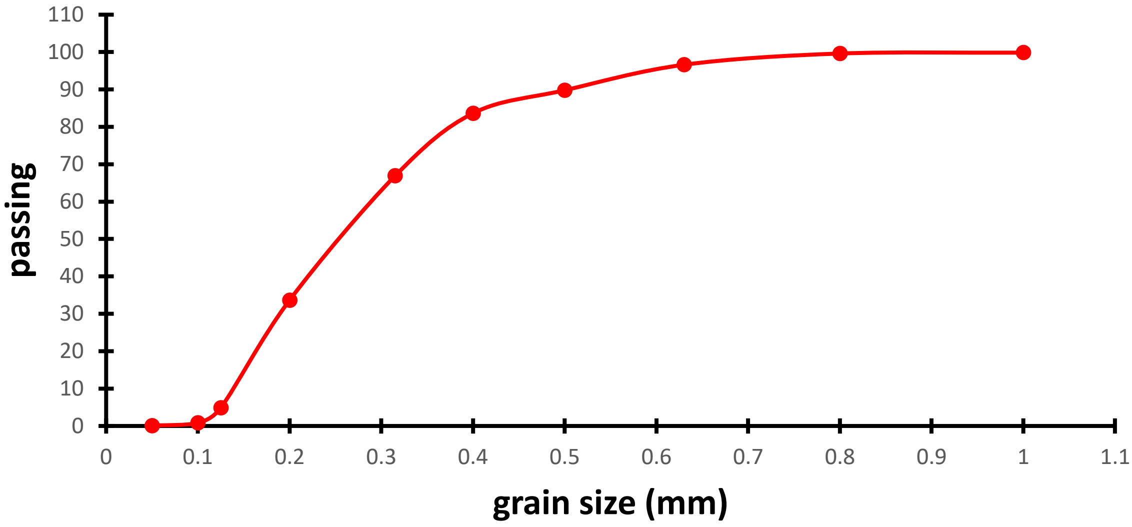


Figure 17: particle size distribution curve

II.3 Maximum and minimum void ratio

The maximum and minimum void ratio for granular soils depends on several factors such as:

- Grain size
- Grain shape
- Particle fine contents

II.3.1 Maximum void ratio emax:

e_{max} is the void ratio of soil in loosest state.
We take the paper funnel, and put it at the bottom of the mold. We fill the funnel with sand, then slowly raise the funnel making sure that the funnel remains full of sample until the end of the experiment, we weigh the sample in the scale and extract the value (emax) through the formula below.

$$e_{max} = \left(\rho_s \times \frac{Vr}{ms}\right) - 1$$

With:

- ρ_s: weight of sand grains = 2.65 g/cm^3
- V_r: recipe volume (cm^3)
- m_s= soil mass (g)

Figure 18: maximum voids ratio measurement

II.3.2 Minimum voids ratio e_{min}

e_{min} is the void ratio of soil in densest state

We carefully put a layer of sand in the mold with a spoon and shake the mold with a small hammer (about 10 kicks for the four directions). We repeat the process until the mold is filled and with a ruler we remove the excess quantity on the mold. We weigh the sample in the balance and extract the value (e_{min}) through the law:

$$e_{min} = \left(\rho_s \times \frac{Vr}{ms}\right) - 1$$

Figure 19: maximum voids ratio measurement

With:

- ρ_s: weight of sand grains = 2.65 g/cm^3
- V_r: recipe volume (cm^3)
- m_s= soil mass (g)

The purpose of soil classification systems is to store soils in families with the same geotechnical characteristics or very similar characteristics. They thus make it possible to group the very numerous samples collected during a survey campaign and to establish geotechnical sections of the ground.

These cuts are precious for the engineer. They complement the geological data, which do not involve those of geotechnical; soils of the same geological origin can have very different geotechnical properties, and vice versa.

The characteristics of this sand are listed in the table below:

Table 6: El Hadjeb sand characteristics

Sand	Mean diameter D_{50} (mm)	Uniformity Coefficient C_u	Minimum Void ratio e_{min}	Maximum void ratio e_{max}	Weight of sand grains ρ_s(g/cm^3)
El Hadjeb sand	0.24	2.15	0.49	0.78	2.65

II.4 Direct shear test (NF P94-071)

In many engineering problems such as design of foundation, retaining walls, slab bridges, pipes, sheet piling, the value of the angle of internal friction and cohesion of the soil involved are required for the design. Direct shear test is used to predict these parameters quickly. The laboratory report cover the laboratory procedures for determining these values for cohesion less soils.
A normal load is applied to the specimen and the specimen is sheared across the pre-determined horizontal plane between the two halves of the shear box. Measurements of shear load, shear displacement, and normal displacement are recorded. The test is repeated foe two or more identical specimens under different normal loads. From the results, the shear strength parameters can be determined. The strength of a soil depends of its resistance to shearing stresses. It is made up of basically the components:

1. Frictional – due to friction between individual particles
2. Cohesive - due to adhesion between the soil particles

The two components are combined in Coulomb's shear strength equation.

$$\tau_f = c + \sigma_f \tan \phi$$

Where τ_f = shearing resistance of soil at failure

- c = apparent cohesion of soil
- σ_f = total normal stress on failure plane
- ø = angle of shearing resistance of soil (angle of internal friction)

II.4.1 Procedure:

1. Assemble the shear box
2. Carefully transfer the sample into shear box after compacting soil sample to optimum moisture condition

Figure 20: transfer sample into shear box

3. Place the loading plate on top of the upper porous plate. After recording the weight of the loading carrier place it is on the loading cap
4. Position all dial gauges and set the readings to zero. Remove the alignment screws which hold two halves of the shear box together.
5. Tighten the remaining, two diagonally opposite screws, until there is a small gap between upper and lower boxes to reduce the frictional force
6. Apply the desired normal load. If there is any vertical displacement, wait till the dial gauges indicate a constant reading and then reset the dial gauge to zero

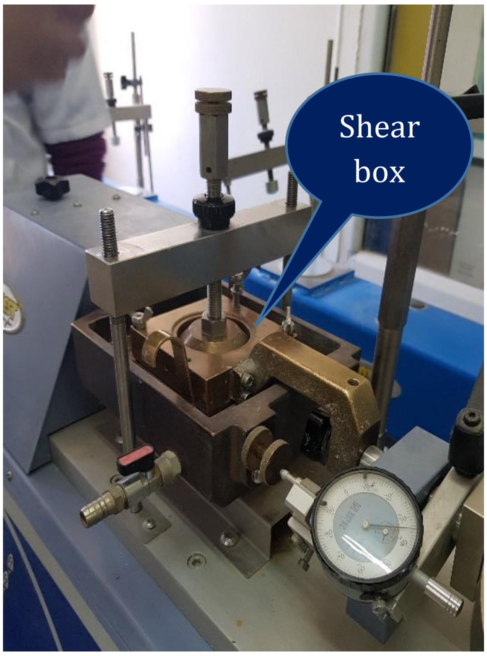


Figure 21: position of shear box

7. Check that screws have been removed and then start the motor to produce the desired constant rate of shearing
8. Take readings of
   a) Shear load from the proving ring
   b) Shear displacement (i.e. Horizontal displacement)
   c) Vertical displacement at every 25 division increment in horizontal dial gauge
9. Stop the test when the shear load starts to reduce or remains constant for at least three readings
10. Remove the soil and repeat the procedure with different normal loads at least for another two samples

Figure 22: direct shear test instrument (LNHC-Batna)

The test has several advantages:

- Large samples can be tested in large shear boxes, as small samples can give misleading results due to imperfections such as fractures and fissures, or may not be truly representative
- Samples can be sheared along predetermined planes, when the shear strength along fissures or other selected planes are needed.
- Easy, fast.

The disadvantages of the test include:

- The shear box cannot give reliable undrained strengths because it is impossible to prevent localised drainage away from the shear plane.
- There is no provision for measuring pore water pressure in the shear box and so it is not possible to determine effective stresses from undrained tests.
- The failure plane is always horizontal in the test, and this may not be the weakest plane in the sample. Failure of the soil occurs progressively from the edges towards the centre of the sample

II.5 Permeability measurement (Mariotte's bottle)

Permeability is a measure of the ease in which water can flow through a soil volume. It is one of the most important geotechnical parameters. However, it is probably the most difficult parameter to determine. In large part, it controls the strength and deformation behaviour of soils

It directly affects the following:

- quantity of water that will flow toward an excavation
- design of cut-offs beneath dams on permeable foundations
- design of the clay layer for a landfill liner

II.5.1 Application

- Estimation of quantity of underground seepage water under various hydraulic condition
- Quantification of water during pumping for underground construction
- Stability analysis of slopes, earth dams, and earth retaining structures

II.5.2 Procedure

The constant load permeability measuring device is based on a Mariotte bottle.

The Mariotte bottle (also called a Mariotte siphon) is a device that allows one to deliver a liquid at a constant (adjustable) pressure. As long as the liquid remains above the bottom of the tube that determines the exit pressure (vide infra), the pressure remains constant regardless of the level of the liquid in the delivery vessel. This ingenious device was invented by Edmé Mariotte, a 17th-century French physicist.

In the device shown in Figure below, the metal plate makes a hermetic seal at the top of the plastic cylinder via a greased rubber gasket, and the inlet tube passes through the plate through a compression fitting that seals with a seal. The pressure at the bottom of the inlet tube is always the atmospheric pressure, but the pressure inside the outlet hole exceeds the atmospheric pressure. As soon as the water starts to come out of the outlet hole, the pressure inside the container decreases, causing a drop in the pressure at the bottom of the inlet tube below atmospheric pressure. This causes air to enter the tube, maintaining the pressure at the bottom of the tube at atmospheric pressure.

So, as long as the water level inside the cylinder is above the bottom of the inlet tube, this causes air to enter the tube, maintaining the pressure at the bottom of the tube at atmospheric pressure. So, as long as the water level inside the cylinder is above the bottom of the inlet tube.

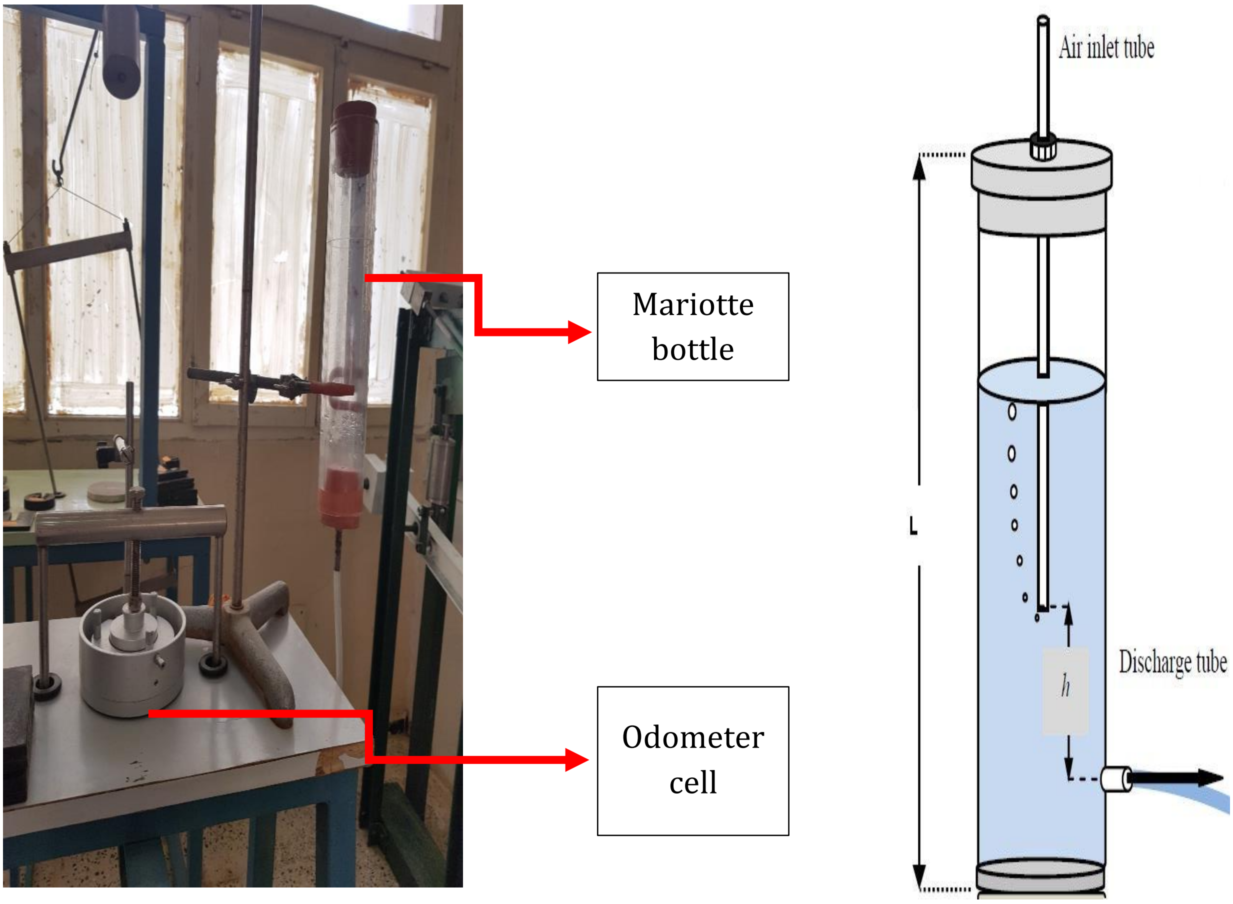


Figure 23: permeability measurement process

II.6 MICP procedure

Three treatment strategies were used in the bio-cementation process in laboratory studies (injection method, surface percolation method, and mixing method).
The current test was performed with surface percolation method, this last consists of spraying the bacterial solution and the calcifying solution which contains reagents on the soil surface. Bacterial and calcific solutions will diffuse under the effect of gravity in the soil (Mujah et al., 2017). The advantage of this method is the minimum energy required for carrying out the bio-cementation operation compared with the other methods like injecting and soil mixing. This method does not make it possible to reinforce soils at great depths, especially soils whose low permeability can hinder the diffusion of the bacteria and the calcifying solution more deeply.
Cheng and Cord-Ruwisch (2014) showed that this treatment strategy successfully calcified, in a nearly uniform manner. In fact, this method has limitations for fine sands (<3 mm).

Precipitation of calcite does not exceed 1 m with great heterogeneity (clogging at the surface). This method is not suitable for large structures of large dimensions (thicknesses) and which consist of fine soils such as earth dams. Moreover, this treatment strategy can be a very effective solution for strengthening of the roadbed, for dust suppression and soil stabilization against external erosion (Cheng et al., 2016).

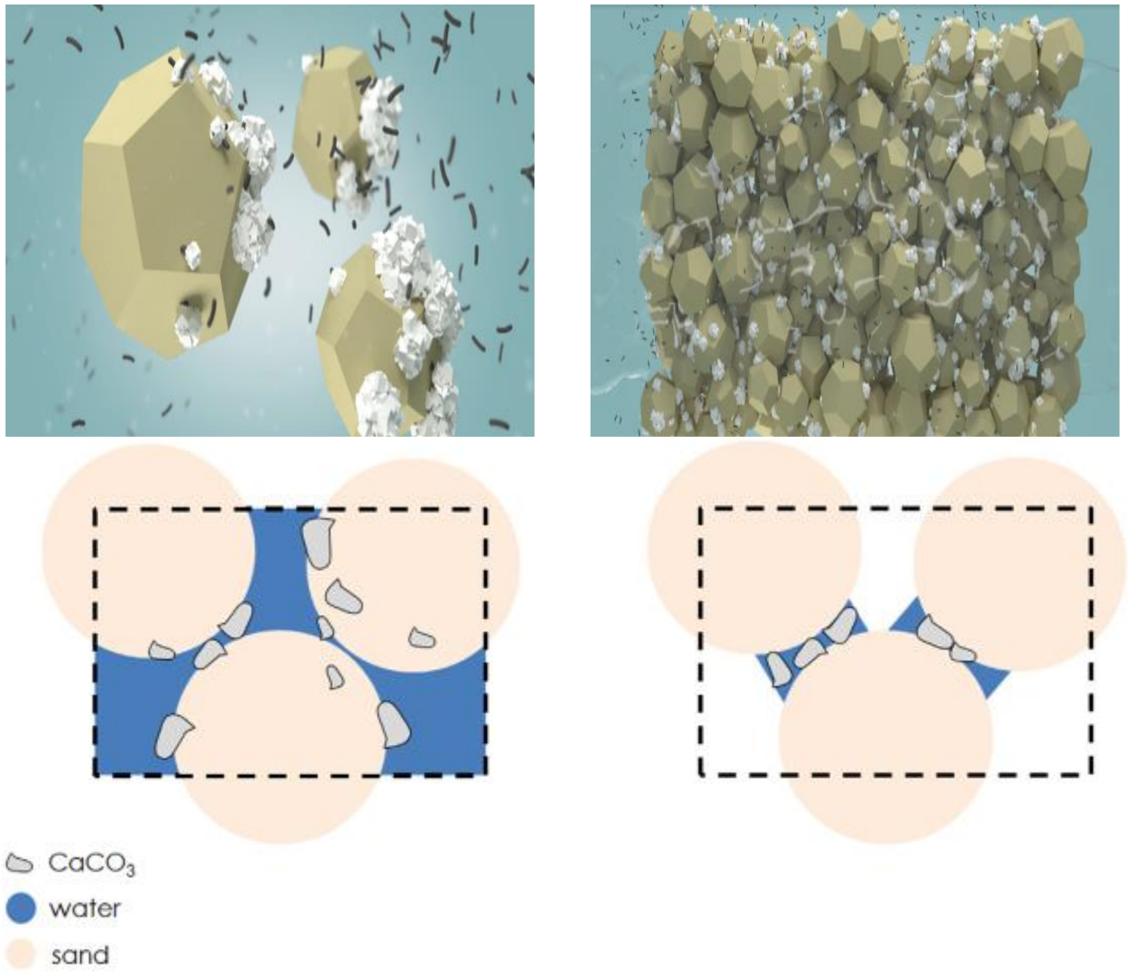


Figure 24: Schematic representation of *S. pasteurii* cells attaching on soil grains (Terzis and Laloui 2019)

II.6.1 Materials and methods

After preparing several mold to treat the cemented sand later through permeability and direct shear test (2 mold for the permeability test and 5 mold for direct shear test), first two ones mentioned before have 20 mm of height and the others have 30 mm of height. These mold were filled with untreated sand with different relative density of (80%, 70%, and 90% etc.).

Plastic glass penetrated with thick needle on the bottom of it to pattern the percolation form of the treating system.
In order to homogenize the water flow injection. A saturation phase of each column was adopted before starting the treatment by surface percolation. After this phase, an injection of one pore volume, the injection solution was divided into two parts:
The first one named the bacterial solution which contains an optical density of specific bacteria called '*sporosarcina pasteurii*' this last was imported from Soletanche Bachy (Soletanche Bachy Entreprise, Rueil-Malmaison, France) under a dried form and saved under temperature between 0 and 7 °C.
The bacteria was dissolved with amount of NaCl in commercial water in connection to increase the potential attachment of bacteria to soil grains, the amount of NaCl added to samples is different from one to another according to the relative density provided.
The second part of injection solution is the calcifying solution, from sample to another it contains percentages of urea and the same of calcium chloride ($CaCl_2$).
As the pictures below show it starts with saturation phase with a carful and strict manner thus it is important to not make the percolation much high to eliminate any possibility to lose the density of the samples due to the flew the percolated water.
Then starting with the bacterial solution in first, we fill the plastic glass with it and percolate it carefully into the sand sample; we left the bacterial solution about 1 hour to make bacteria stabilise among sand particles then we add the first calcifying solution on the sample.
The test doesn't finished yet, after 10 hours we add the second calcifying solution on it. Typically reinforcing untreated sand samples by microbial induced carbonate precipitation through surface percolation contains two calcifying solutions and one bacterial solution; firstly the bacterial solution then the first calcifying solution after one hour and the second one ten hours later.

Figure 24: soil treatment by surface percolation method

II.7 Conclusion:

The method of soil improvement by biocementation trough precipitation of calcite is a promising technique in the field of securing hydraulic structures, potentially subject to internal erosion and liquefaction. More advanced work on biological geotechnical applications is possible through a variety of research, including bio-cementing.
However, serious problems remain unresolved, preventing the development of such large-scale processes.
The main challenge in this process is the heterogeneity of treatment; this phenomenon is related to several factors (dosage of calcifying solution and enzymatic solution, viscosity and density of the injected solution, size of the enzyme relative to the size of the pores of the soil, existence of a flow of water, etc.). Before a large-scale application of the process, fundamental research is still needed to define the conditions of applicability of this process, in order to validate its efficiency in terms of mechanical behaviour. Specific tests will be conducted for each issue to further investigate changes in microstructural properties and to determine the possible links between microstructural changes and mechanical behaviour. Most of the research to date has focused on the implementation of biogeochemical processes of soil properties improvement. Furthermore, performing an in situ treatment is only the first step in the development of the technique, whether it is the mechanical or biogeochemical durability over time.

Third chapter

Results and discussions

III. Introduction

In this chapter, the experimental results will be presented on the studied sand. In addition this chapter will show some results of behavior changed of the treated sand by microbial induced carbonate precipitation especially on the mechanical side. Still, the third chapter will include some comments on the experimental results adopted for applications of soils reinforcement in laboratory by MICP process.

III.1 Improvement of mechanical properties of biocemented soil

III.1.1 Shear strength

Shear strength is the magnitude of shear stress that a soil can sustain and depends strictly on the shear strength parameters of soil including the cohesion (c) and friction angle (φ). It was found that the shear strength of biocemented soil was strictly affected by the increase in soil cohesion resulting from the increase in the cement content, while the friction angle was not greatly affected by the cementation process.

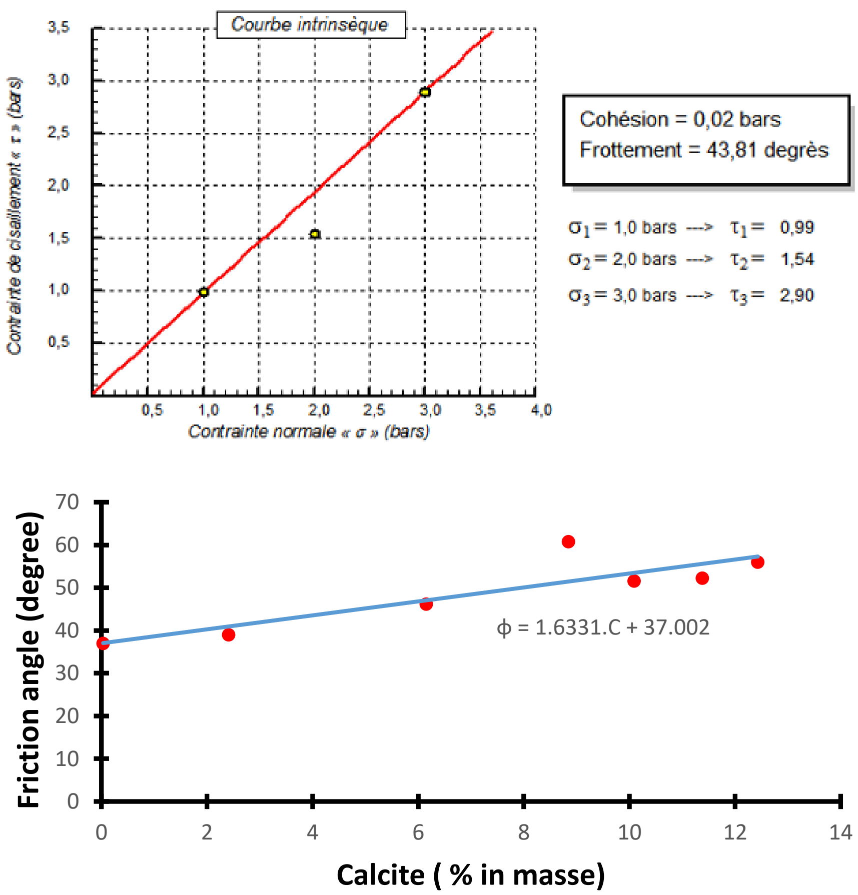


Figure 25: shear strength improvement: untreated (up), treated (down)

In accordance with literature, (Chou et al. 2011) reported a large increase in soil friction angle but a small increase in soil cohesion was detected for almost all treated samples using MICP.

It was also found that the peak shear strength of biocemented soil was higher compared to untreated specimens, and generally higher in the growing cell treatment than that of other treatment methods. Ng et al. (2012) applied MICP using bacteria to treat a residual soil and found that the shear strength ratio of treated to untreated soils was increased at values ranging from 1.40 to 2.64. Montoya and DeJong (2015) observed that the shear strength of MICP treated sand was dramatically improved with the increase in MICP cementation. With increasing cementation level, the peak shear strength increased leading to a transition in the stress–strain behaviour from strain hardening to strain softening. Cheng et al. (2013) also discussed the cohesion and friction angle of biocemented soil samples treated under different degrees of saturation and showed that at lower saturation degree, the precipitated $CaCO_3$ crystals contributed more to improving the soil cohesion than the friction angle. On the other hand, regardless of the saturation degree, both the cohesion and friction angle increased at higher $CaCO_3$ content due to the filling effect of the calcite crystals in the soil pore spaces.

III.2 Stiffness

Soil stiffness, commonly known as soil elastic modulus (E), is the ratio of stress over strain. Soil stiffness is closely related to the bonding strength between loose soil grains. For sand treated by MICP it is clearly showed an augmentation of soil elastic, with the amount of calcite in the highly cemented sand this modulus has exceeded 250 MPa. This result confirms other ones from previous studies, Cheng et al. (2013) compared the elastic modulus of biocemented sand with other types of geomaterials such as concrete, gravel and soft rock, and found that the biotreated sand is the most flexible among the materials tested.

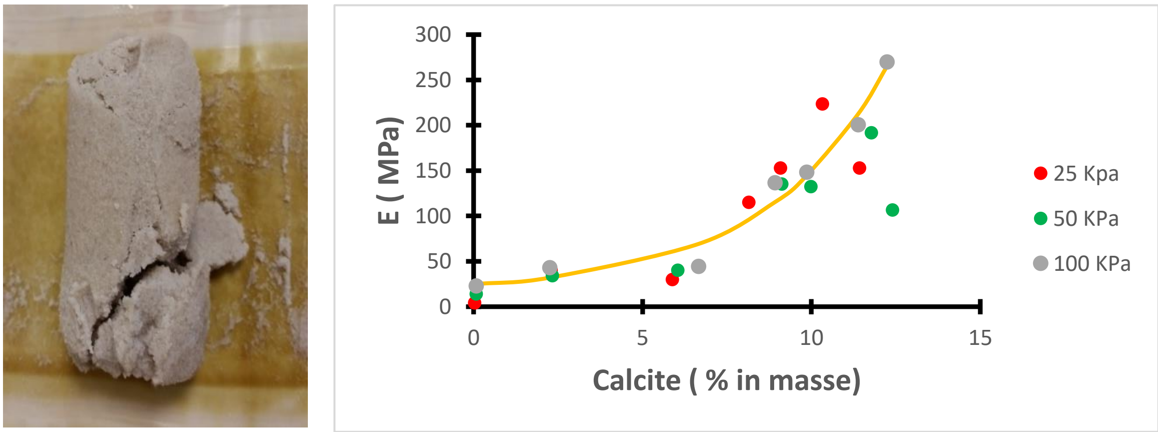


Figure 26: evolution of soil elastic modulus (data obtained from Dadda 2017)

In earthquake prone areas, less stiff soil can provide an extra time for evacuation due to its ability to maintain significant residual strength even after failure. Lee et al. (2013) performed MICP on residual soil and found that the stiffness behavior of biocemented residual soil is similar to that of biocemented natural sand. Previously, researchers studied the effects of cementation on strength and stiffness of granular soils using a variety of different cementing agents namely the Portland cement, gypsum and sodium silicate.

It was found that the strength and stiffness of cemented materials increase with the increase of the amount of cementing material in the soil matrix; although the amount of cementing material required to produce a certain cementing effect may vary.

Based on this fact, Montoya and DeJong (2015) studied the effect of biocementation on stress–strain behavior of biotreated sand and found that the stiffness was dramatically improved with the increase of MICP cementation (i.e. $CaCO_3$ content). It is worth noting that the effective stress path as well as the drainage condition influence the MICP treated soils in a way that it can reduce the rate of stiffness due to the degradation of cementation prior to failure. The stress paths of a given soil depend on the initial, in situ and final state of soil sample.

From the results of the evolution of soil elastic modulus as a function of masse of calcite obtained by Dadda et al. (2017) we would like to obtain a model (an empirical law) that would link these two parameters without the need to perform tests. To do this we adopted a calculation procedure using the options available on Excel. The evolution of the soil elastic modulus (E) as a function of the percentage of calcite can follow the following mathematical function:

$$f(x) = a\ e^{bx} + h$$

With the identification of the previous parameters we will have the equation below.

$$E(c) = a\ e^{b.C} + h$$

Using the least squares method and a solver available on Excel, minimizing the error between the value of E given by Dadda et al. (2017) and that given by the proposed expression, we could determine the expression of the parameters a and b and h expressed before:

a=8.24
b=0.27
h=16.62

The expression of soil elastic modulus (E) as a function of percentage of calcite C thus becomes the equation:

$$E(C) = 8.24\ e^{0.27.C} + 16.62$$

In order to verify the good coherence between the result of the proposed expression and that obtained by Dadda et al. (2017), we plot the two results on the same graph.

III.3 Biocemented sand using 3D x-ray micro tomography

Nowadays, X-ray micro-tomography represents one of the most efficient techniques to explore the 3D microstructural properties of a porous media in qualitative and quantitative way. The accuracy of the results depends to the resolution and the contrast of the objects in the 3D images.
3D images of some biocemented sand sub-samples were performed using X-ray synchrotron microtomography on the ID19 beamline at the ESRF in Grenoble.
To obtain such 3D images, the sub-sample is fixed between the parallel beam line and the detector (FReLoN CCD) which is characterized by fast data saving and a low noise. A resolution of (0.65 µm3/voxel) was chosen in order to visualize precisely the calcite crystals, which have a typical size of 15 micrometres. The field of view is (3250 x 3250 x 2000 voxels), i.e. (2.11 x 2.11 x 1.3 mm^3) to obtain 3D images large enough to be representative of the material. The transmitted rays were recorded for 1800 projections. Later, these images were collected to reconstruct numerically the internal microstructure of the sample using a filter back projection algorithm.
Figure 34 shows some 2D views of three sub-samples after reconstruction. We can distinguish the three phases: air (dark phase), sand grain (grey phase) and calcite (light grey phase). The chosen approach allows increasing the contrast between both phases with a slight brightness of calcite, which is coating sand grains (Figure 4.4). In the case of pure absorption images, it would not have been possible to distinguish between calcite and sand, because of the similar absorption coefficients of the two phases which is related to their densities (ρs = 2650 kg/m^3, ρc = 2710 kg/m^3).
To resume, in the future work, we propose to determine and to quantify the evolution of the contact properties (contact surfaces, coordination number, contacts orientation, type of contact, etc.) of biocemented sand as function of the cementation level using 3D images obtained by X-ray microtomography.

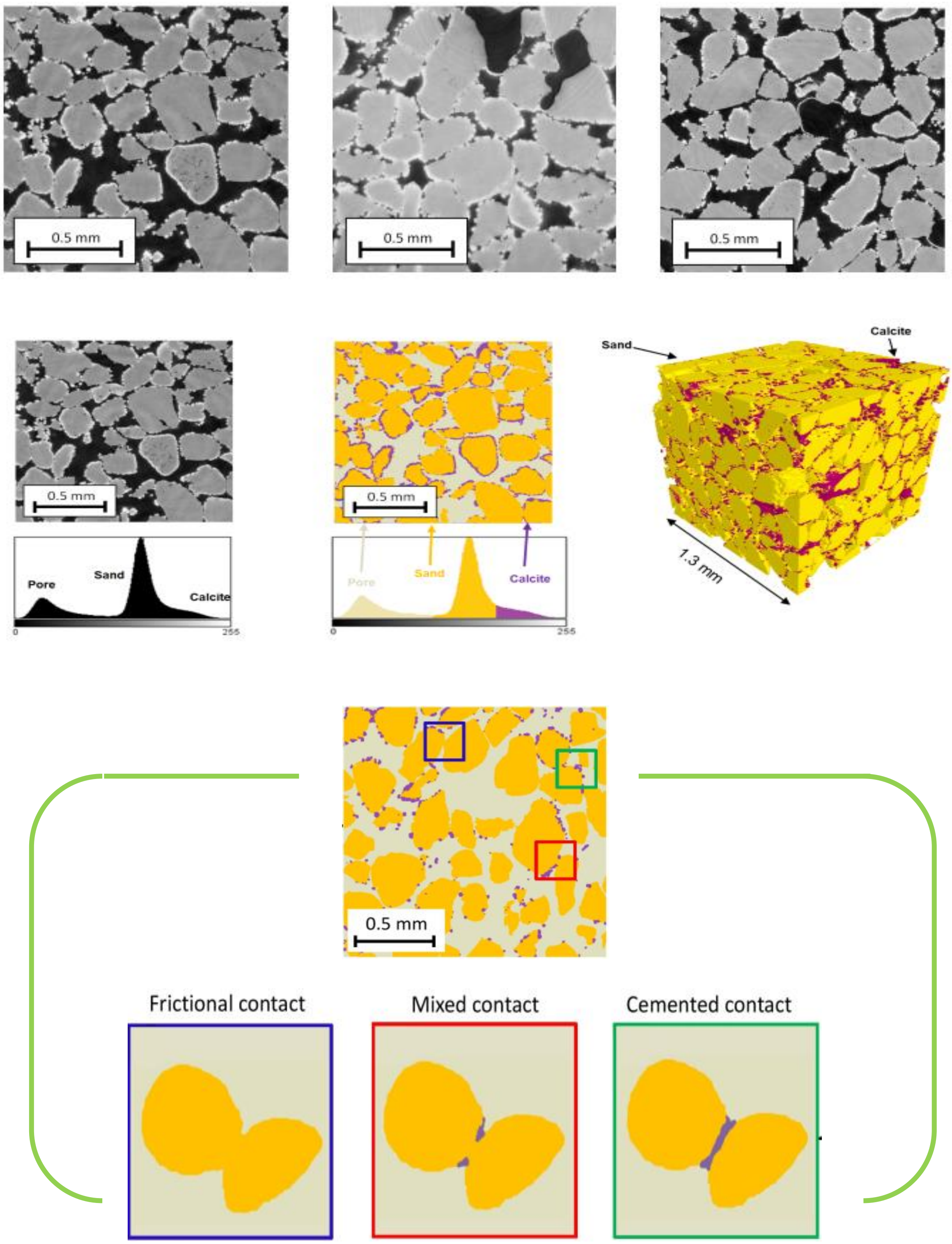


Figure 27: image treatment by 3D x-ray micro tomography (Dadda (2017) data)

III.4 Contact surface area

A cloud of voxels with a complex 3D shape represents each contact surface between two grains (see figure below). A direct computation of the voxels number can be used as a first estimation of the contact surface area. However, this method usually underestimates the real value. For that reason, different methods (marching cube, voxel-based area estimation, Crofton method, etc.) have been proposed in order to compute the surface area of a cloud of voxels (Legland et al. 2011).

In this work, all the contact surface areas have been estimated by using the Matlab function "geometric measures in 2D/3D images" developed by Legland et al. (2011). The surface area is measured using a discretization of the Crofton formula. For regular 3D objects (cubes, spheres and plane surfaces), a discretization along the three main orthogonal directions is sufficient to get a good accuracy of the surface area. When the surface is irregular with a complex shape, it has been shown that 13 directions of discretization are sufficient to estimate the surface area with accuracy (Legland et al., 2011).

In order to evaluate the errors induced by the method, we have computed the surface area of well know surfaces. The surfaces under consideration are planar square surfaces of different sizes (the number of voxels varies between 100 and 10000, i.e. the area ranges between 50 μm^2 and 5000 μm^2), and inclined with an angle of 45 ∘ with respect to both planes (XY) and (XZ). An error has been found ranging from 3% for the smaller surfaces to 15% for the larger ones.

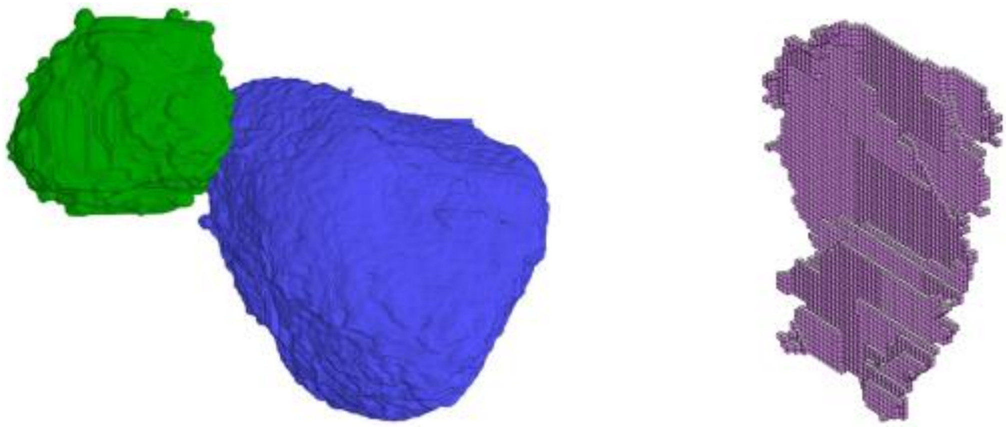


Figure 28: Geometry of the grain to grain contacts
Example of two grains in contact (left),
contact surface between the two grains in contact (right)

III.5 Future research

The field of biocementation involves a multidisciplinary research at the confluence of geotechnical engineering, micro- biology, ecology and chemical engineering. Despite the fact that several researches from the above fields have developed main sets of data and interpretations, currently no study has yet attempted to determine the optimum MICP process in terms of the cost and factors involved, for potential commercial implementation. Once the factors affecting the MICP process have been optimized in the laboratory at the micro level (i.e. at the particle to particle contact points) and macro level (i.e. soil columns set up), further research in terms of upscaling to field applications at the mega level can then be executed and predicted in the complex true natural environment. The complexity of the coupling effects among the flow, mixing and reaction contributes to the limited progress in MICP upscaling. Specific challenges ahead with respect to MICP upscaling include controlling the flow and transport through heterogeneous media, durability of treatment, permanence of the mixing technique and mapping of the subsurface stratigraphy at the particle level.

Future research should also enlarge the scope of MICP applications, not just in terms of strengthening and improving soils but also harnessing the soil ability to self-heal using the premixed microorganisms in the soil matrix. Bacteria can be reactivated upon loading and undergo the same microbial reaction inside the soil, provided that ample cementation solution is supplied. By doing so, MICP treatment would be able to heal the degraded $CaCO_3$ bonds post-shearing (Harbottle et al. 2014).

Montoya and DeJong (2013) was the first to observe the improved behavior of MICP treatment after the healing process. The healing ability of MICP can be used to prevent additional settlement and damage to structures or soils during earthquakes and aftershocks, for example. Also, the plausibility of using seawater which already has calcium ions that provides calcium chloride source naturally deposited in the solution as a potential substitute for the pre- purchased manufactured calcium chloride should not be forgotten.

A preliminary study carried out by Cheng et al. (2014) has shown the potential use of seawater as a replacement for the calcium source in the $CaCO_3$ precipitation during MICP process. It was found that the UCS of biocemented sand samples achieved two times higher strength (with the same amount of crystals produced) than that of MICP treatment by highly concentrated calcium and urea solution retaining up to 30% permeability which signifies a good drainage potential.

III.6 Conclusion

This work presents a new application for MICP as a consolidation technique for ground by using an easily applicable surface percolation method.
An experimental study has been performed to investigate the biocementation effects on the mechanical strength of treated sand.
The experimental results point out that the strength of the biocemented sand is strongly improved compared to .that of initial untreated material. We made observations by SEM and micro-X-ray tomography to actually look at this phenomenon and to check the distribution of calcite crystals in the soil and the morphologist of these crystals.
The evolution of physical properties is directly related to changes in the microstructure of bio-cemented sand. However, a clear relationship between the change in microstructure and these mechanical properties.

General conclusion

In this work we presented an experimental study via a new technique of stabilization and soil reinforcement. This technique bases it on biochemical procedures by the use of bacteria or enzymes to catalyse chemical reactions, thus integrating chemical and biological processes and applying them to geotechnical engineering to obtain a biogeochemical process called bio-cementation.

Bio-cementation is a modern method of strengthening and improving the physical and mechanical properties of soil, environmentally friendly and economical, which has attracted attention in the twenty-first century because of its great advantages over traditional reinforcement. This process is carried out by a catalyst (urease enzyme) and other chemical solutions as a source of calcium and carbon (urea-$CaCl_2$).

This technique is done in three main stages; (1) The urease enzyme catalyses the hydrolysis of urea ($CO(NH_2)_2$) to form ammonium and carbonate ions, (2) The carbonate ions produced react with calcium ions and precipitate in the form of carbonate crystals of calcium, (3) the grains of sand are bound together by the crystals of calcium carbonate and forming a crystalline solid material. These reactions cause the precipitation of calcium carbonate as solid crystals that increase the cohesion between the soil seed and increase the rigidity and mechanical strength of the soil. Some important factors may influence this process, such as the concentration of bacteria or urease enzyme, the concentration of chemical solutions, temperature, pH, and other factors must be monitored during this process. A significant change is observed in all the properties of the soil treated via the bio-cementation technique that change directly linked by a modification on the microstructural properties such as the decrease of porosity and the increase of contacts between the seeds in the treated soil.

The main work in this memory is the evaluation of the physical properties of sand dune El Hadjeb Biskra defined as a loosely loose soil cohesion and given the worst case. We used in this work several data obtained from PhD thesis of Dr.Dadda (2017). The most important results obtained in this work are:

- A significant increase in compressive strength, shear strength, and stiffness of bio-cemented soil.
- Increasing the calcite rate in bio-cemented soil also leads to a rapid and almost linear increase in other mechanical properties such as cohesion and friction angle.
- Microstructural observations by SEM and X-ray micro-tomography can be given an image with good precision to better understand this phenomenon at the microscopic scale.

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