Bio-cementation in sandy soils: effect of treatment duration on compressibility and the amount of bio-cement produced

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Abstract

Concrete is the second most-consumed product in construction after water. Its production causes more than 5% of anthropogenic CO_2 emissions and therefore, due to the increasing awareness of the climate impact, there is a large interest in reducing its carbon footprint. Microbial induced calcium carbonate precipitation technique (MICP) was introduced at the end of the 20th century as an alternative to the use of cement for soil improvement. Microbial-induced calcium carbonate precipitation (MICP) is a biologically driven calcium carbonate precipitation technology that uses urease producing bacteria fed with a calcium source. With this treatment, the precipitation of calcium carbonate in the soil pores occurs, reducing soil permeability and increasing stiffness and strength as a function of the amount precipitated. This work aims to understand how the treatment duration would influence the mechanical behaviour of the soil by analysing compressibility and the amount of bio cement produced and, indirectly, durability. The ultimate purpose of this work was to present conclusions that could help develop this topic in further investigations that could culminate in more cases of upscaling from the laboratory to the field.

KEYWORDS: Bio-cementation, MICP, calcium carbonate, SEM, MIP

1. Introduction

Bio-cementation, or microbial induced calcite precipitation (MICP), is a green and sustainable soil improvement technique that explores a soil natural biochemical process to improve its engineering properties.

In this process (1), specific urease producing bacteria hydrolyses urea ($CO(NH_2)_2$), which results in the formation of carbonate ions (${CO_3}^{2-}$) and ammonium (NH_4^+).

$$CO(NH_2)_2 + 2\,H_2O \rightarrow {CO_3}^{2-} + 2\,NH_4^+ \quad (1)$$

Local pH rises during this process and increases the tendency for bacteria itself to serve as a nucleation site for calcite crystal. Calcite is then precipitated through the combination of carbonate ions from the hydrolysis of urea and the calcium ions (Ca^{2+}) supplied in a feeding solution, usually using calcium chloride (2).

$$Ca^{2+} + {CO_3}^{2-} \rightarrow CaCO_3 \quad (2)$$

The main factors affecting the calcite precipitation are calcium ion concentration and the other feeding solution constituents, such as the availability of nucleation sites, pH and temperature. Having this in mind, this work wanted to understand how the treatment duration would influence the mechanical behaviour of the soil by analysing compressibility and the amount of bio cement produced. Durability was evaluated indirectly as well because it depends on the mineralogical form of calcium carbonate and, obviously, on its concentration in the soil pores.

2. State of the Art

The applications of microbial induced calcite precipitation (MICP) in soils are promising and can solve many types of problems such as (i) protection against soil erosion, investigated by Gomez et al. (2015), (ii) mitigation of landslide risk, present in the case of Terzis et al. (2018), (iii) strengthening of foundations, (iv) strengthening of dikes in the occurrence of natural disasters such as floods and storms at sea described by Van Paassen et al. (2011) and (v) in the protection against liquefaction characterized by Montoya et al. (2013).

Soils treated with MICP can have strong mechanical characteristics which make this technique an alternative to other traditional soil improvement methods where a binding agent such as cement is added.

There are several traditional soil improvement techniques, however, jet grouting is one of the most used and which is similar to bio-cement. Both techniques consist of injecting a binder into the soil that promotes the physical bond between the particles.

In order to understand the advantages and disadvantages of the bio-cementation over the jet-grouting, it is important to make a comparative analysis between the two. That analysis is presented in the form of a table that summarises the principal parameters of both techniques. In Table 1, the left part concerns the jet-grouting, while the right part concerns bio-cementation or MICP.

Table 1 - Comparative analysis between jet-grouting and bio-cement techniques.

JET GROUTING	**MICP**
Soil Type	
- All, preferably sandy soils.	- Sandy soils.
Equipment	
- Cement silo - Mixing plant - Injection pump - Compressor - Drilling and injection machine	- Mixing plant - Injection machine - Injection pump - Extraction pump
Quality Control	
- Test columns - Continuous recording of execution parameters	- *In situ* tests - Continuous recording of execution parameters
Mechanical and Hydraulic Properties	
- Increase of UCS - Increase of stiffness - Decrease of permeability	- Increase of UCS - Decrease of permeability

The first differentiating factor is the type of soil to which each technique applies. In this respect, jet-grouting is capable of operating on any type of material, while MICP is appropriated mainly for sandy soils.

In terms of equipment needed to carry out the different treatments, these are partially similar differing only in certain determining factors. For jet-grouting, all machinery associated with the production of cement must be ensured and for bio-cement, it is necessary to ensure that conditions are in place to keep the bacteria on site.

Quality control is similar for both techniques, such as the need for real-time monitoring of the various parameters inherent to the injection and the collection of material.

Based on the material collected on-site, it is possible to assess the soil parameters and verify that both the jet-grouting and bio-cement techniques have an increase in resistance to simple compression and decrease in permeability.

Although both techniques have similarities, there are crucial differences that are mainly reflected in overall costs. Economical analysis (Suer et al., 2009) and the improvement achieved so far when using this technique show the potential of bio-cementation and its advantages over jet-grouting. However, MICP application is still far from being feasible on large scale. There is a need to mechanize the process and to improve certain processes to promote homogeneous treatment and the survival of bacteria, factors that explain the unpredictability of this treatment. This unpredictability has to be minimized for the application of bio-cementation.

3. Materials and Methods

3.1. Soil Properties

For this analysis, soil from a possible case study to be carried out by IST was used. This case consisted of the bio-stabilization of a slope on the A13 motorway, in order to avoid the formation of ravines.

From the grading size distribution analysis (Figure 1), it was found that the percentage of fines was around 2%, with uniformity and curvature coefficients, respectively, 3.40 and 1.42. The soil classifies as well-graded sand (SW) according to the Unified Classification of Soils.

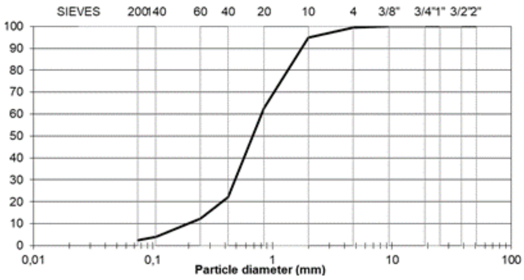

Figure 1 - Grading size distribution of the soil.

3.2. Bacteria and Feeding Solution

The bacteria species used in this study were *Bacillus Pasteurii*. They were growth at the Bioengineering laboratory of IST and then lyophilized in order to allow their use in a more expeditious way. This procedure consists in drying the bacteria so that they were always ready to be used. The dosage used was around 10^8 cells/mL.

For the MICP process to start, not only the bacteria were required, but also their feeding solution. It contains the nutrients necessary to remain physiologically active (urea and calcium chloride as a source of calcium). Please see Damas Pinto (2020) for further details.

3.3. Sample Preparation

Three types of treatment were defined to be applied to the samples: with water, with feeding solution and with bacteria. The water treatment served as a reference for the others. The treatment with feeding solution served as a control and was done to prove that only reagents, such as calcium chloride, one of the components of the feeding solution, do not by itself allow the precipitation of calcium carbonate in sufficient quantities to confer some resistance to the soil. The treatment with bacteria had different durations to allow them to understand if the formation of bio-cement is enhanced with time.

All specimens were prepared in the same way. They were assembled inside oedometer steel rings 70 mm in diameter and 20 mm in height (to be subjected to treatment with water, feeding solution and bio-cementation treatment (bacteria and food solution) with 4 different durations. For all types of treatment, the soil was compacted already thinking that it would be subjected to an oedometer test at the end of the treatment (Figure 2).

In total, six specimens were prepared, two of them simply submerged in water or feeding solution, and four specimens treated with bacteria and feeding solution, but with different treatment durations.

Figure 2 - Treated sample inside the oedometer cell.

The treatment with bacteria had two phases. The first consisted of a 3-day operation where the bacteria solution was injected followed by a superficial watering with a feeding solution every 24 hours. For the second phase, the specimens were injected with the feeding solution every 24 hours for 3, 8 and 14 days, respectively for specimens 2, 3 and 4. After the treatment, all specimens were subjected to an oedometer test. The cases studied are presented in Table 2.

Table 2 - Description of all cases tested.

Specimens	Observations
Water	Reference
Feeding Solution	Control
Specimen 1	Tested after injecting bacteria for 3 days
Specimen 2	Tested after injecting bacteria for 3 days, followed by 3 days of feeding
Specimen 3	Tested after injecting bacteria for 3 days, followed by 8 days of feeding
Specimen 4	Tested after injecting bacteria for 3 days, followed by 14 days of feeding

3.4. Tests performed

Oedometer tests were performed in all samples to measure compressibility and how it would be affected by the presence of precipitated calcium carbonate. The treated samples were submerged in feeding solution during the oedometer test to avoid dissolution.

Pieces of the samples were extracted after the oedometer tests to perform several other complementary tests:

- Leaching tests with 0.5M NaCl acid to measure the amount of calcium carbonate present at the end of the treatment;

- Mercury intrusion porosimetry tests, MIP, fundamental to understand if the amount of bio-cement produced would reduce pore size;

- Mineralogical analysis by X-ray Diffraction Analysis, XRD, to characterize if the mineral form of calcium carbonate is calcite or vaterite. This would provide information about the durability as well in case of detecting calcite because is insoluble;

- Scanning electron microscope images, SEM, to see if the minerals appear to be calcite (rhombohedra shape) or vaterite (spherical shape).

4. Results

4.1. X-ray Diffraction Analysis

The XRD results show in all samples that there was a predominance of silicate minerals from sand, whether in the form of quartz, microcline or muscovite.

The samples prepared with water and feeding solution served as a reference to evaluate the efficiency of treatment with bacteria. This assessment was determined by the presence of calcium carbonate minerals, with calcite and vaterite being the most and least stable forms, respectively. In the table below it is shown the results of that evaluation in Table 3.

Table 3 - Minerals identified in each specimen.

Mineral/ Score	Calcite	Vaterite
Water	-	-
Feeding Solution	5	-
Specimen 1	32	28
Specimen 2	41	28
Specimen 3	51	22
Specimen 4	57	12

From the values of Table 3, it can be seen that the values of vaterite are higher in specimen 1 and 2 than in specimen 3 and 4 and that there is an increase on calcite score from sample 1 to sample 4. The explanation for this phenomenon is based on the number of feeding days and their respective quantities. The greater the amount of feeding solution provided, the longer the calcium carbonate has to precipitate, which leads to the formation of more stable minerals such as calcite.

4.2. Scanning Electron Microscopy

From this technique, associated with the EDS system, it was possible to compare the images obtained in the samples of untreated soil with the images of the soil samples with the presence of bacteria. It was also possible to evaluate its presence of calcium.

In all treated specimens, it was possible to verify that they all presented calcium carbonate under some form of arrangement. In specimens with less feeding solution provided, it was visible the formation of a calcium carbonate precipitate in a spherical form (Figure 3), while in the specimens with the longest time of treatment it was observed the cubic structures of calcium carbonate, one of the most favourable and stable arrangements typical of calcite (Figure 4).

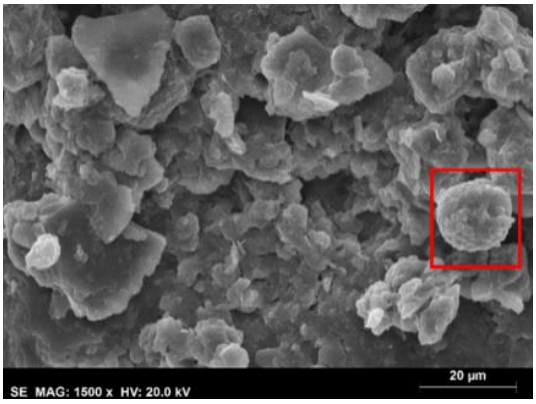

Figure 3 - SEM image of specimen 2.

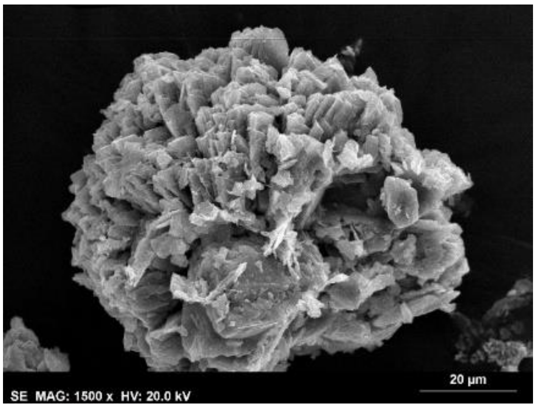

Figure 4 - SEM image of specimen 4.

4.3. Mercury intrusion porosimetry (MIP)

In order to understand the evolution of soil pore size over time, mercury intrusion porosimetry (MIP) tests were performed on all samples.

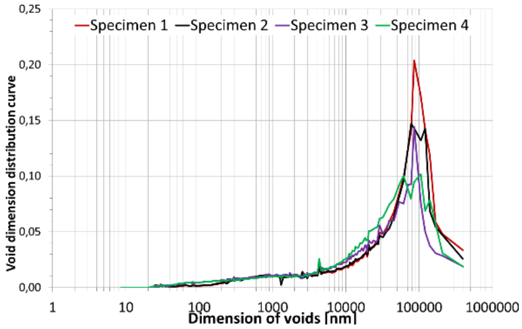

Figure 5 - Pore size distribution curve for test specimens 1, 2, 3 and 4.

By evaluating the curves for the 4 specimens treated with bio-cement shown in Figure 5, it is clear that, with the increase in the number of treatment days, there is a decrease in the dimension of the largest voids. This conclusion is drawn by the fact that the curve found for specimen 4 has the peak slightly more to the left than all other samples. Specimen 4 has a proportion of voids with smaller sizes, which makes it the richest in bio-cement. Strictly speaking, it can be said that for all the treated specimens, there was a reduction in the size of the pores because they were clogged with calcium carbonate, which seems to have been greater the longer the duration of the treatment.

4.4. Quantification of Calcium Carbonate

For the quantification of calcium carbonate present in the samples, it was performed the leaching tests. The values found are in Table 4.

Table 4 - Quantification of calcium carbonate

	$\% \, CaCO_3$
Water	2.165
Feeding Solution	1.841
Specimen 1	2.604
Specimen 2	1.853
Specimen 3	4.776
Specimen 4	7.422

For the untreated specimens, it can be seen a larger quantity of calcium carbonate for the specimen tested with water than for the specimen tested with feeding solution. This was unexpected and is probably explained by sample heterogeneity and the fact that the sample collected may not be sufficiently representative. Except for specimen 2, there is a visible increment in the percentages found for the calcium carbonate for increasing treatment duration, as would be expected.

4.5. Oedometer tests

To evaluate the compressibility of the soil, an edometric test was performed. The compressibility should decrease for increasing amounts of bio-cement since the presence of this mineral corresponds to the presence of bonds between the particles.

The compressibility and swelling indexes, respectively Cc and Cs, are presented in Table 5. This table also includes the void ratio at preparation. The values for specimen 3 are not presented because it suffered some disturbances.

Starting with the value of the swelling index (C_S), it appears that it does not remain constant throughout the various tests, which means that the soil presents an inelastic behaviour. This may be explained by experimental error, or eventually by some bond breakage being the bonds formed due to precipitated calcium carbonate. Regarding the compressibility index (C_C), this does not show an increasing value among the specimens. Some reduction would be expected due to the presence of the bonds and the increment of their amount with treatment duration. The value of a Cc is almost constant, which may indicate that the bonds between the particles were broken. They could be broken due to loading but maximum stress reached is not high enough to justify it, they could be interrupted due to the interference of fine particles also covering the grains of sand, or these bonds could be broken already before loading, which may have occurred during the injection of the feeding solution.

Table 5 - Oedometer test parameters.

	e_i	C_C	C_S
Water	0.68	0.139	0.018
Feeding solution	0.62	0.129	0.012
Specimen 1	0.66	0.119	0.008
Specimen 2	0.60	0.115	0.011
Specimen 4	0.63	0.142	0.002

The curves found for the samples tested with water, specimen 1 and specimen 2 are presented in Figure 6 using normalized void ratio. It can be seen the similar paths for all, although it appears that yielding stress had increased for specimen 2. This may be explained by the lowest void ratio at its preparation.

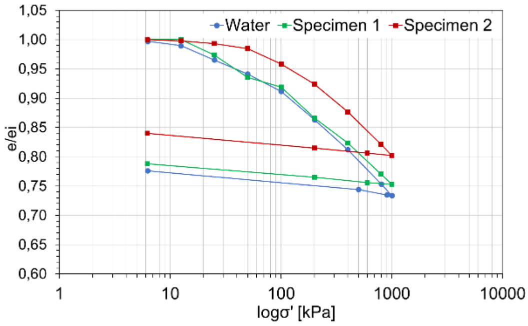

Figure 6 - Compressibility curves of water, specimen 1 and 2.

4.6. Final comments

Considering the compressibility values measured in the oedometric tests, the conclusions were somewhat inconclusive about the influence of the duration of the treatment. In fact, stiffness did not increase with the increase in the number of days of feeding, which would indicate that calcite precipitation had not occurred. However, the leaching tests have demonstrated the presence of calcium carbonate in all treated specimens, increasing with duration. The largest quantity was found for the longest treatment period, sample 4. To corroborate the knowledge acquired so far, by mercury intrusion porosimetry (MIP) it was possible to understand the evolution of the size of the pores over time, which for specimen 4, resulted in a clear decrease in the size of the voids larger dimensions. As a final proof, using the scanning electron microscopy it was possible to ascertain the shape of the bio-cement crystals and confirm the most stable shape, present in sample 4.

Concluding, the precipitation of bio-cement increased with the treatment time, being the sample 4 the one with the best results. Increasing the time of treatment and consequently, the quantity of feeding solution leads to a large amount of precipitate. For some reason, however, the large amount of calcium carbonate does not correspond to a large number of bonds probably because they were broken during the treatment or they were not continuous due to the interference of fine particles also covering the grains of sand.

5. Conclusions

The main goal of this study was to find out how the variation in the treatment time would affect the mechanical behaviour of the soil and, indirectly, its durability.

Leaching tests, MIP and SEM showed, as expected, that the precipitation of bio-cement increased with the treatment time, being the sample 4 the one with the best results. Oedometer tests results were inconclusive concerning stiffness increment with increasing duration. Increasing the time of treatment leads to a large amount of precipitate and should increase stiffness if the calcium carbonate bonds remain intact but it is believed they were broken due to the treatment process adopted or to the influence of the fines interfering with their continuity connecting the large particles of sand.

To conclude, bio-cementation is an innovative technique that changes the hydro-mechanical characteristics of the soil. This has enormous potential however, it is necessary to optimize the treatment process to promote bond formation and obtain homogeneous distributions throughout the treatment so that favourable resistance, stiffness and permeability values can be reached. For the time being, this balance has not yet been reached, but there is a strong scientific investigation on the subject and all this inefficiency will soon be overcome. In the future, bio-cementation will be a sustainable and viable alternative to other soil improvement techniques that constitute a global environmental problem.

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