Experimental Investigation on the Effect of Using Different Calcium Sources with Biogrouting on the Improvement of **Poorly Graded Fine Sand**

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Abstract: In light of the cities' growth, it has now become necessary to establish infrastructure in areas that were previously deemed inappropriate. To improve the engineering characteristics of these problematic lands, many methods have been applied. Recently, an innovative and sustainable technique called Microbial Induced Calcite Precipitation has emerged for soil improvement. Many factors that affect the efficiency of this technique include the concentrations of bacteria solution, the concentrations, and the type of the chemical solutions, in addition to methods to introduce the bacteria and these chemical solutions to the soil, pH, etc. The objective of this study is to evaluate the performance of the Microbial Induced Calcite Precipitation as a technique to improve and enhance the very fine poorly graded sandy soils and expand knowledge about improving a loos sand by using the MICP technique. Furthermore, this study was carried out using six types of calcium sources in the cementation solution, two concentrations of cementation solution as well as two types of soils. This study shows that the use of low concentrations provides a greater improvement in the engineering properties of the treated soil. The pH values were not greatly affected by soil type or the type of cementation chemical solutions and their concentration. The samples with axial splitting had a lower unconfined compressive strength than that shown by the samples with shear fracture.

Keywords: microbial-induced calcite precipitation; fine sand; soil improvement; pH; strength

1. Introduction

Cementation is an important method for soil improvement. Cementation, whether naturally occurring or added by man-made activities, has been shown to increase the ability of sand to resist failure when subjected to static or dynamic loading [1]. Artificial cementation mostly includes the injection of colloidal silica grout, Portland cement, or gypsum. In general, a diversity of chemical, jetting, and permeation grouting techniques are used to distribute the artificial grouts in the subsurface. Recently, bio-mediated methods have been developed as promising alternatives for ground improvement. As a new approach, microbial induced calcite precipitation (MICP) soil improvement method has attracted great interest. MICP soil improvement is defined as "a chemical reaction network inside the soil that is maintained and controlled by bacteria activity and whose byproducts modify the engineering characteristics of the soil."[2]. Recent researches have shown that many types of bacteria have the ability to produce biocementation. These include urease producing bacteria, iron reducing bacteria, nitrifying bacteria, oligotrophic microaerophilic bacteria, sulfate reducing bacteria, and dimorphic phytase-active yeast for the production of calcium-phosphate precipitation[3]–[6].

This study was conducted in laboratories of Huazhong University of Science & Technology and aims to explore the effect of calcium source type, the concentration of the cementation solution, the soil particle size, the use of a combination of calcium sources as a cementation solution, on the content of calcium carbonate, the porosity, and the strength. In this study, section 2 deals with the materials and methods of preparation the soil specimens, bacterial culture and cementation solution. Section 3 deals with, results and discussion. Finally, Section 4 discusses and conclusions.

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2. Methods and Materials

2.1 Preparation of Soil Specimens

The current investigation used two types of poorly graded sand. The first type was fine silica sand, with particale size of 0.1 mm. The second type was river sand collected from the Yangtze River's bank. The poorly graded sand column was made by packing dry sand (with unit weights of 16 kN/m³ and 14 kN/m³, porosities of 37.67% and 48.67%, and pore void volumes of approximately 33 ml and 43 ml for silica sand and river sand, respectively) into an 80 mm high and 37 mm inner diameter PVC sample.

2.2 Bacterial Culture and Cementation Solution

Sporosarcina pasteurii (ATCC 11859) was the ureolytic bacterium used in this study. ATCC 11859 was grown in a yeast extract medium (20 g/l yeast extract, 10 g/l ammonium sulfate, 0.13 M Tris buffer, pH = 9) in sterile aerobic batch conditions. The bacterial culture's optical density OD600 ranged between 1.4 and 1.5. The bio-cementation was carried out using four different cementation solutions. The first is a cementation solution with two concentrations of equal moles of pure anhydrous calcium chloride (1 M; 0.5 M) and urea (1 M; 0.5 M). The second type is a cementation solution with two concentrations made up of equal moles of pure calcium acetate monohydrate (1 M; 0.5 M) and urea (1 M; 0.5 M). The third type is made from urea (1 M, 60 g/l) and eggshell calcium chloride (1 M). The fourth type is made from urea (1 M) and eggshell calcium acetate (1 M).

2.3 MICP Procedure

Gravity-induced downward precipitation was used to conduct microbially induced carbonate precipitation for soil treatment. The sand columns were initially divided into Silica sand and Yangtze river sand samples. The columns were flushed with bacterial culture, followed by 3 hours of retention time to allow bacteria cells to settle in the soil granules. Each group was divided into six groups depending on the type of cementation solution. The first group was flushed with a cementation solution made of CaCl₂ (eggshells), the second and third groups were flushed with a cementation solution made of pure CaCl₂ and (1 M, 0.5 M), and the fourth group was flushed with a cementation solution made of C₄H₆O₄Ca (eggshells). While the fifth and sixth groups were flushed with a cementation solution made of pure C₄H₆O₄Ca (1 M, 0.5 M). Additionally, to the previous groups, another two groups of Yangtze river sand were prepared, the first group was flushed with a cementation solution made of a mixture of calcium chloride and calcium acetate (1 M), while the second group was flushed with a cementation solution made of calcium chloride (1 M) and calcium acetate (1 M) in alternating order. The MICP reaction time for cementation solution with a concentration of (1 M) was 24 hours and for cementation solution with a concentration of (0.5 M) was (12 hours). The cementation solution flushing was repeated every 24 hours for cementation solution with a concentration of (1 M) and every 12 hours for cementation solution with a concentration of (0.5 M). The test was carried out at a temperature of 30°C + 2°C. The treatment was stopped after 4 days of flushing with cementation solution, and the soil specimens were placed in a 60°C oven for 7 days. Table 1 lists the variables and details of the experimental combinations.

Type of soil	Cementation solution				groups symbols
Yangtze river sand	CaCl ₂	and	urea	(1M)	A
	(eggshells)				
	C ₄ H ₆ O ₄ Ca	and	urea	(1M)	В
	(eggshells)				
	CaCl ₂ and urea (1M)			(AR)	С
	C ₄ H ₆ O ₄ Ca and urea (1M)			(AR)	D
	CaCl2 and urea (0.5M) (AR)			(AR)	Е
	C ₄ H ₆ O ₄ Ca and	urea (0.5M)		(AR)	F
	(CaCl ₂ + C ₄ H ₆ O ₄ Ca) and urea (1M)				G
	(CaCl ₂ or C ₄ H ₆ O ₄ Ca) and urea (1M) (alternate)				Н
Silica sand	CaCl ₂	and	urea	(1M)	I
	(eggshells)				
	C ₄ H ₆ O ₄ Ca	and	urea	(1M)	J
	(eggshells)				
	CaCl ₂ and urea (1	M)		(AR)	K
	C ₄ H ₆ O ₄ Ca and urea (1M) (AR)			L	
	$CaCl_2$ and urea $(0.5M)$ (AR)			M	
	C ₄ H ₆ O ₄ Ca and urea (0.5M)			(AR)	N

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Table 1 Cementation solutions of MICP treatments

2.4 CaCO₃ Content

To determine the amount of CaCO₃ in the soil samples, the samples were crushed, then oven-dried, and the dry weights were recorded. The dry soil was soaked in Hydrochloric acid solution to dissolve precipitated CaCO₃, then washed with water, and finally oven-dried and recorded the weights. The weight of the CaCO₃ precipitated within the specimen is determined by comparing the weights before and after soaking in hydrochloric acid.

2.5 Unconfined Compressive Strength (UCS)

Unconfined Compressive Strength (UCS) tests were performed on bio-cemented soil specimens with selected diameter-to-height ratios of 1:2 using the procedure described in ASTM D2166 (ASTM, 2013). Before UCS measurements, the samples were flushed with deionized water (about four pore volumes) and dried at 60°C for 24 hours.

2.6 porosity

The porosity was determined by calculating the difference in sample mass. The water's volume in a saturated sample of known volume can be used to determine porosity. The water volume is calculated by dividing the mass of the saturated sample minus the oven-dry mass of the sample by the density of water. Porosity is calculated by dividing the volume of water by the original volume of the sample.

$$n = (M_{sat} - M_{dry}) / (\rho_w V_t)$$

Where: n : porosity;

 M_{sat} : mass of saturated sample; M_{dry} : mass of the dry sample;

 ρ_w : density of water; V_t : volume of sample.

3 Results and Discussions 3.1 pH

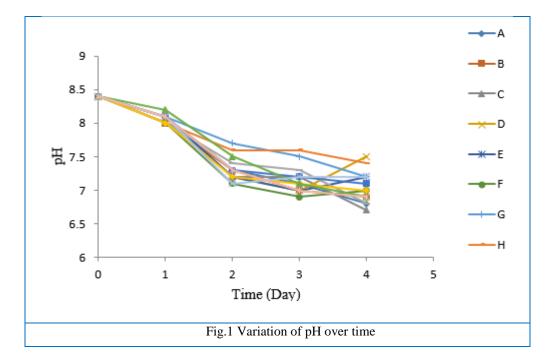
MICP method has many effects in the treated soil, one of these effects is the increase of pH of the environment near the bacteria cells. The local pH rise can be achieved by the production of ammonia resulting from the enzymatic hydrolysis of urea, known as urease activity. During MICP treatment, pH was measured in all groups. pH was measured for the specimens treated with cementation chemical solutions 0.5M twice per day, while, the specimens treated with cementation chemical solutions of 1M were measured once per day. Fig.1 presents the variation in pH of effluent over time.

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The initial value of pH for bacteria suspension was 8.4. The first effluent pH value oscillated between 8 and 8.2, while the effluent pH value after that fluctuated around 7, which achieves the requirements of downstream biological treatment processes. This rate of decrease can be caused by a reduction in the rate of enzyme activities or by the accumulation of organic complexes [7]. Nevertheless, a lower ureolysis rate can be beneficial for the distribution of chemical solutions and result in more uniform calcium carbonate precipitation, given a uniform distribution of microbes [8].

Fig.1 shows that the pH values were not greatly affected by soil type or the type of cementation chemical solutions and their concentration.



3.2 Failure Mode Analysis of Sandstone

Typical failure modes of treated specimens under uniaxial compression are shown in Fig. 2. It can be seen that major inclined shear cracks formed along the specimens with the cracking plane inclined 90° concerning the horizontal plane and the specimen cannot burst into pieces after failure (longitudinal splitting), while the second type of failure is simple shear. The weak regions for all specimens with the second failure mode mainly concentrated on one-third of the bottom of the sample.

According to Bobet [9], under uniaxial stress, pre-existing microcracks with appropriate dimensions and directions concerning the maximum principal stress get closed when the applied compressive stress reaches a specific level called the crack-closure stress.

When the local tensile strength becomes less than the tensile stresses produced by compression at the edges of the pre-existing flaws, then reproduce new cracks from those edges and propagating cracks, known as wing cracks.

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These wing cracks align themselves parallel to the maximum principal stress [9]. This indicates that when the microstructure of a specimen does not hinder the propagation of wing cracks, the specimen fails in axial splitting mode (Fig. 2 a).

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However, when wing crack propagation along the maximum principal stress is constrained because of the existing microstructure, coalescence of nearby wing cracks or wing cracks in close proximity generated from the edges of the appropriately oriented microcracks takes place to release the strain energy in the form of shear fracture (Fig. 2 b) resulting in higher unconfined compressive strength than that showed by axial splitting.

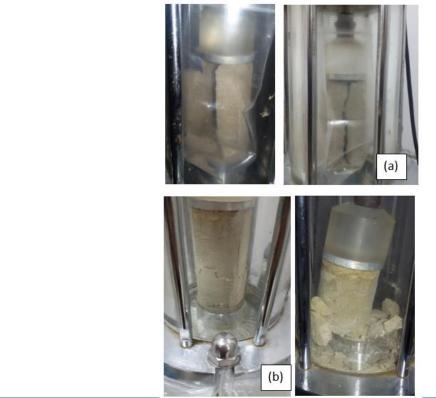


Fig. 2 Typical failure modes of treated specimens under uniaxial compression. (a) axial splitting (b) sh ear fracture

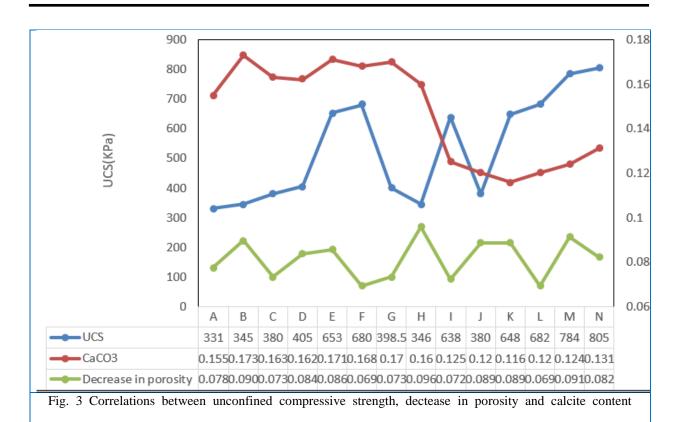
3.3 The relationship between Cementation solution, Calcite Content, and Shear Strength

Fig. 3 shows the relationship between calcium carbonate content on one side and shear strength and porosity on the other.

The figure showed that calcium acetate tended to decrease in porosity similar to that produced by calcium chloride but with a higher resistance, and this means that calcium carbonate precipitation worked better to bind sand grains.

Fig. 3 also shows that the final precipitation amounts of calcium carbonate were similar in the same type of soil regardless 3 of the type and concentration of the nutrient solution, but lower concentrations of the solution gave higher resistance, and this means that the greater part of calcium carbonate worked to increase the resistance rather than reduce the permeability.

The figure also shows that sand with lower initial porosity (silica sand) gave less calcium carbonate precipitation compared to river sand; nevertheless, the decrease in porosity was almost the same in the two types, and the compressive strength was higher in the finer sand.



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4 Conclusions

The process of microbial induced calcite precipitation, especially when it involves sand particles, is extremely complex. This process may be impacted by a number of factors, including the soil type, calcium source, pH and concentration of the cementation solution.

The findings of this study demonstrated that

- Using the MICP technique can improve the engineering properties of poorly graded fine sand.
- The cementation solution's type and concentration significantly influence the effectiveness of MICP.
- Unconfined compressive strength is mainly affected by the fineness of the soil.
- The use of industrial grade materials (i.e. urea, hydrochloric acid, acetic acid) to make the calcium salts would be useful for the biocementation process to be implemented in the site. Cost-effective chemical solutions would be beneficial.
- The pH values were not greatly affected by soil type or the type of cementation chemical solutions and their concentration.
- The unconfined compressive strength of the samples with shear fracture was higher than what is typically demonstrated by axial splitting.
- Compared to calcium chloride, calcium acetate produces calcium carbonate with higher resistance.

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