Evaluation on Solidification Effect of Silty Sand Based on Biomineralization Technology

Yuyuan Chen^{1, *}, Hemanta Hazarika¹, Yuke Wang²

¹Graduate School of Engineering, Kyushu University, Fukuoka, 819-0385, Japan;

Email: hazarika@civil.kyushu-u.ac.jp (H.H.)

² College of Water Conservancy Science and Engineering, Zhengzhou University, Zhengzhou, 450001, China

Email: ykewang@163.com (Y.W.)

*Correspondence: chen.yuyuan.137@s.kyushu-u.ac.jp (Y.Y.C.)

Abstract-Biomineralization has been widely used as an economical, clean and sustainable method for soil consolidation. In this paper, calcium carbonate precipitation process was catalyzed by soybean urease, and the mechanical properties and microstructure of the bio-treated sandy silt were evaluated by unconfined compressive strength (UCS) test, scanning electron microscope (SEM). The procedures of deriving soybean crude urease and bio-treatment for soil column samples were performed. The optimum concentration and grouting time of the biological solidification solution were determined. The findings demonstrate that the unconfined compressive strength of bio-treated sample reaches the maximum value of 9.87 MPa at the cement solution concentration of 1.5mol/L and the grouting times of 10. The CaCO₃ precipitation formed in the samples gathered at the contact point and the surface of the soil particles. meanwhile, it can observe in SEM image that crystal clusters were formed. The above test results provide primary datasets and theoretical instruction for the application in the solidification of sandy silt.

Keywords—Soybean urease, enzyme-induced calcium carbonate precipitation (EICP), unconfined compressive strength, microstructure

I. INTRODUCTION

Due to the shortcomings of insufficient strength and stability, many kinds of soil from the nature cannot be directly used as construction filling material. Conventional soil solidification methods are mainly divided into physical methods and chemical methods. The physical methods basically include drainage consolidation [1, 2], sand compaction pile [3], dynamic compaction [4–6], etc. The chemical methods are mainly include high-pressure jet grouting [7, 8], deep mixing [9], grouting [10, 11] and so on. The conventional solidification methods for soil have the advantages of being simple in process, excellent adaptability and convenient operation. However, the aforementioned methods have the disadvantage of high cost, high energy consumption and poor environmental compatibility, which will produce an adverse effect on the surrounding buildings and environment.

In recent years, enzyme-induced calcium carbonate precipitation (EICP) solidification technology is proposed as a new soil solidification and anti-seepage technology. With the advantages of simple technology, low cost, small construction disturbance and ecological environmental protection, enzyme-induced calcium carbonate precipitation (EICP) technology is widely used in the field of geotechnical engineering [12–14]. EICP is a biogeotechnical technique for improving the engineering properties of granular soil in which calcium carbonate is precipitated from an aqueous solution within the soil pores. Calcium carbonate precipitation improves the strength, stiffness, and dilatancy of the soil by pore filling, particle roughening and interparticle binding. In the EICP process, free urease enzyme catalyzes the hydrolysis of urea in an aqueous solution, which results in carbonate ion production. In the presence of calcium ions, the carbonate ions precipitate as calcium carbonate when the concentration of carbonate ions exceeds the level of supersaturation.

Numerous studies have been carried out on the soil solidification based on EICP method. Yasuhara et al. [15] investigated the unconfined compressive strength and permeability of bio-treated Toyoura sand with equimolar concentrations of urea and calcium chloride. When precipitated carbonate content is 6% and 5%, the corresponding unconfined compressive strength is 0.75 MPa and 1.6 MPa, respectively. Gao et al. [16] proposed a new method for the improvement of silty soil. This method adopts the calcium carbonate precipitation process catalyzed by soybean urease. Crude urease is derived simply by collecting the liquid formed by soaking soybean powder in water. The activity of crude urease is linearly related to the amount of soybean powder added to the water, and is high enough to be used for soil treatment. Putra et al. [17] added magnesium sulfate to the EICP solution, fixing the concentration of urea at 0.50 M and varying the concentration of urease from 1 to 5 g/L. The low concentration of magnesium sulfate promoted the formation of calcite, and the precipitation quality increased with the increase of magnesium sulfate concentration in the tube test was founded. When the magnesium sulfate concentration was 0.10m, a large amount of gypsum was formed. Up to present, the free urease used

Manuscript received July 1, 2023; revised August 23, 2023; accepted September 23, 2023.

in most EICP studies has been isolated from crops such as soybeans, beans, watermelon seeds and peas [18].

In this study, enzyme-induced calcium carbonate precipitation (EICP) method was applied to solidify the sandy silt. The unconfined compressive strength (UCS) tests and scanning electron microscope (SEM) analysis were conducted to explore the influences of cement solution concentration and grouting times on the solidification effect of sandy silt. These findings provide some insight into the application of enzyme-induced calcium carbonate precipitation (EICP) technology in the biomineralization.

II. MATERIAL AND MEHTODS

A. Test Material

The test material in this study is dredged river silt. To remove contaminants, the dried river silt was put through a 2mm sieve. Particle analysis test and other fundamental physical property tests were conducted on the river silt. The grading curve for river silt was depicted in Fig. 1. The basic physical characteristic parameters of the test material are as follows. The coefficient of uniformity C_u is 5.08. The coefficient of graduation C_c is 1.662. The maximum dry density ρ_{dmax} is 1.65 g·cm⁻³. The minimum dry density ρ_{dmin} is 1.357 g·cm⁻³. The liquid limit ω_L is 23.8%. The plastic limit ω_L is 12.4%. The plastic index I_p is 11.4. The specific density G_s is 2.7.



B. Test Program

In this paper, the samples with diameter of 50 mm and height of 50 mm were prepared by PVC cylindrical mold. The mold was placed vertically, and a layer of 300 mesh nylon screen was placed at the top and bottom as the filter screen. The levels of cement solution concentration were set to 0.75 mol/L, 1 mol/L, 1.25 mol/L, 1.5 mol/L, and 1.75 mol/L. The grouting times were set to 4, 6, 8 10. The relative density was controlled to 40%. The test program is shown in Table I. There were 20 samples prepared in this study.

C. Methods

In this study, the urease was extracted from soybean. The following are the specific steps. A grinder was used to reduce soybeans to powder. A screen with a mesh size of 100 was used to separate the powdered soy beans. Deionized water was added to the soybean powder, and the mixture was agitated for 15 minutes. The soy bean powder solution was then centrifuged for 30 minutes at a speed of 4000 revolutions per minute. The extracted soybean urease solution was filtered from the supernatant in the centrifuge bottle.

TABLE I. TEST PROGRAM

Sample	cement solution concentration C (mol/L)	Grouting times N	Relative density (%)
S-1	0.75	4	40
S-2	0.75	6	40
S-3	0.75	8	40
S-4	0.75	10	40
S-5	1	4	40
S-6	1	6	40
S-7	1	8	40
S-8	1	10	40
S-9	1.25	4	40
S-10	1.25	6	40
S-11	1.25	8	40
S-12	1.25	10	40
S-13	1.5	4	40
S-14	1.5	6	40
S-15	1.5	8	40
S-16	1.5	10	40
S-17	1.75	4	40
S-18	1.75	6	40
S-19	1.75	8	40
S-20	1.75	10	40

To examine the mechanical characteristics of soil column samples that have undergone biotreatment, unconfined compressive strength tests were performed. The dirt column sample was subjected to axial loading at a loading rate of 1 mm/min until the sample ultimately failed. Unconfined compressive strength σ is used to define the maximum axial stress.

Following the UCS testing, the pieces from the damaged sample were collected for microstructure examination. The evolution of precipitate's structure and shape were examined using scanning electron microscopy (SEM). The broken samples were first processed into 5 mm \times 5 mm \times 2 mm Square sheet specimen and vacuum freeze dried. For precision and sufficient microstructure data, the samples' microstructures were enlarged by 1000x and 3000x, respectively.

III. RESULTS AND DISCUSSION

Fig. 2 displays the stress-strain curves derived from unconfined compressive strength (UCS) tests. Unconfined compressive strength is the greatest value of the axial stress. Fig. 3 summarizes the distribution of unconfined compressive strength of soil column samples with various bio-treatment methods.





Figure 2. Unconfined compressive strength test results, (a). Stress-strain curves of UCS tests (N=4; C=0.75/1/1.25/1.5/1.75 mol/L), (b). Stress-strain curves of UCS tests, (N=6; C=0.75/1/1.25/1.5/1.75 mol/L), (c). Stress-strain curves of UCS tests (N=8; C=0.75/1/1.25/1.5/1.75 mol/L), (d). Stress-strain curves of UCS tests, (N=10; C=0.75/1/1.25/1.5/1.75 mol/L).

As depicted in Fig. 3, it is clear that the increase of grouting periods increased the compressive strength of the soil column samples. The compressive strengths of the soil column samples with grouting times N=4, 6, 8, and 10 are 1.45MPa, 3.82MPa, 6.89MPa, and 9.81MPa, respectively, when the cement solution concentration is C=1.5mol/L. When the cement solution concentration C=1.5mol/L, the unconfined compressive strength of the solidified soil column samples peaked at 9.81MPa. However, as the cement solution concentration approaches 1.75 mol/L, the unconfined compressive strength rapidly decreases. The cause of this phenomenon may be due to the fact that when the concentration of cement solution above a particular threshold, the activity of soybean urease was inhibited and the yield of calcium carbonate was impacted.



The distribution and development of calcium carbonate precipitation in the sample are clearly analyzed by the microstructure image. Fig. 4 displays the SEM microstructure picture of bio-treated soil column samples. The developed calcium carbonate crystals were continuously piled as the EICP process went on, as seen in Fig. 4. The calcium carbonate crystals that were created by the reaction filled the spaces between the soil particles and adhered to their surface. Calcium carbonate that had already formed acted as the nuclear site, resulting in the formation of crystallized calcium carbonate.

As demonstrated in Fig. 4(a) and Fig. 4(c), crystal clusters accumulated and formed at the point of contact with the soil particles. A cladding layer was built on top of the crystal-line layer, and as the calcium carbonate crystals atop the cladding layer interacted with one another, a bedding structure gradually emerged, as depicted in Fig. 4 (c). The magnified calcite crystal's shape was shown in Figures 4(b) and 4(d)). The gradual filling of the gaps by calcium carbonate in this deposit pattern is an important feature. This calcium carbonate accumulates in the adjacent pores and creates a strong adhesive force that can increase high shear strengths.







Figure 4. Microstructure picture of bio-treated samples, (a). Calcium carbonate crystal attached on the surface (the microstructure images magnified by 1000x), (b). Calcium carbonate crystal attached on the surface (the microstructure images magnified by 3000x), (c). Calcium carbonate crystal filling the pores (the microstructure images magnified by 1000x), (d). Calcium carbonate crystal filling the pores (the microstructure images magnified by 3000x), (d). Calcium carbonate crystal filling the pores (the microstructure images magnified by 3000x).

IV. CONCLUSIONS

In order to evaluate the influence of cement solution concentration and grouting times on the solidification effect of sandy silt with bio-mineralization technology, a series of unconfined compressive strength (UCS) and scanning electron microscope (SEM) analysis were carried out in this study. The main conclusions were drawn: (1) The unconfined compressive strength of bio-treated soil column sample with a initial relative density of 40% is 9.87MPa when the optimal cement solution concentration C=1.5mol/L and the grouting times N=10. (2) The calcium carbonate crystals produced by the reac-tion were filled in the pores between the soil particles and stuck to the surface of the soil particles. (3) After the biotreatment, the calcium carbonate gathered in the pores formed a strong bonding force, which enhanced the shear strength of the soil column samples.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

AUTHOR CONTRIBUTIONS

Dr Wang contributed to the conception of the study. Mr Chen performed the data analyses and wrote the manuscript; Prof. Hazarika helped perform the analysis with constructive discussions; all authors had approved the final version.

ACKNOWLEDGMENT

The work present in this paper was supported by the JST (Japan Science and Technology Agency) under the "SPRING - Support for Pioneering Research Initiated by the Next Generation" program, (Grant No. JPMJSP2136); National Natural Science Foundation of China (Grant No. 52178369; 52109140); Key Specialized Research and Development Breakthrough in Henan Province (Grant No. 212102310977); Key Projects of High Schools of Henan province (Grant No. 20A560021); Natural Science Foundation of Henan Province (Grant No. 202300410424); Youth Talent Promotion Project of Henan Province (Grant No. 2021HYTP016); China Postdoctoral Science Foundation (Grant No. 2019M662533). These financial supports are gratefully acknowledged.

REFERENCES

- C. Rujikiatkamjorn and B. Indraratna, "Analytical solutions and design curves for vacuum-assisted consolidation with both vertical and horizontal drainage," *Canadian Geotechnical Journal*, vol. 44, no. 2, pp. 188–200, 2007.
- [2] C. Rujikiatkamjorn and B. Indraratna, "Analytical solution for radial consolidation considering soil structure characteristics," *Canadian Geotechnical Journal*, vol. 52, no. 7, pp. 947–960, 2015.
- [3] K. Harada and J. Ohbayashi, "Development and improvement effectiveness of sand compaction pile method as a countermeasure against liquefaction," *Soils and Foundations*, vol. 57, no. 6, pp. 980–987, 2017.
- [4] W. F. V. Impe and A. Bouazza, "Densification of domestic waste fills by dynamic compaction," *Canadian Geotechnical Journal*, vol. 33, no. 6, pp. 879–887, 2017.
- [5] J. Kodikara, T. Islam, and P. Rajeev, "Interpretation of the loading-wetting behaviour of compacted soils within the "MPK" framework," *Part II: Dynamic compaction. Canadian Geotechnical Journal*, vol. 53, no. 5, pp. 806–827, 2016.
- [6] M. Shen, C. H. Juang, and Q. Chen, "Mitigation of liquefaction hazard by dynamic compaction — A random field perspective,"

Canadian Geotechnical Journal, vol. 56, no. 12, pp. 1803–1815, 2019.

- [7] Z. Li, H. Liu, Z. Dun, L. Ren, and J. Fang, "Grouting effect on rock fracture using shear and seepage assessment," *Construction* and Building Materials, vol. 242, p. 118131, 2020.
- [8] J. Ca *et al.*, "Comprehensive service properties evaluation of composite grouting materials with high-performance cement paste for semi-flexible pavement," *Construction and Building Materials*, vol. 153, pp. 544–556, 2020.
- [9] S. Y. Liu *et al.*, "Field investigations on performance of T-shaped deep mixed soil cement column-supported embankments over soft ground," Journal of Geotechnical and Geoenvironmental Engineering, vol. 138, no. 6, p. 718, 2012.
- [10] C. Zhang et al., "Formulation and performance of grouting materials for underwater shield tunnel construction in karst ground," *Construction and Building Materials*, vol. 187, pp. 327–338, 2018.
- [11] J. P. Zhang *et al.*, "Development of cement-based self-stress composite grouting material for reinforcing rock mass and engineering application," *Construction and Building Materials*, vol. 201, pp. 314–327, 2019.
- [12] D. Neupane *et al.*, "Distribution of mineralized carbonate and its quantification method in enzyme mediated calcite precipitation technique," *Soils and Foundations*, vol. 55, no. 2, pp. 447–457, 2015.
- [13] V. Krishnan et al., "Variability in the unconfined compressive strength of EICP-treated "standard" sand," Journal of Geotechnical and Geoenvironmental Engineering, vol. 147, no. 4, 2021.
- [14] H. Meng *et al.*, "Multiple-phase enzyme-induced carbonate precipitation (EICP) method for soil improvement," *Engineering Geology*, vol. 294, 2021.
- [15] H. Yasuhara *et al.*, "Experiments and predictions of physical properties of sand cemented by enzymatically-induced carbonate precipitation," *Soils and Foundations*, vol. 52, no. 3, pp. 539–549, 2021.
- [16] Y. Gao *et al.*, "Calcium carbonate precipitation catalyzed by soybean urease as an improvement method for fine-grained soil," *Soils and Foundations*, vol. 59, no. 5, pp. 163–1637, 2019.
- [17] H. Putra, H. Yasuhara, and N. Kinoshita, "Applicability of natural zeolite for NH-forms removal in enzyme-mediated calcite precipitation technique," Geosciences, vol. 7, no. 3, p. 61, 2017.
- [18] A. M. Kayasth and N. Das, "A simple laboratory experiment for teaching enzyme immobilization with urease and its application in blood urea estimation," *Biochemical Education*, vol. 27, no. 2, 114–117, 1999.

Copyright © 2023 by the authors. This is an open access article distributed under the Creative Commons Attribution License (<u>CC BY-NC-ND 4.0</u>), which permits use, distribution and reproduction in any medium, provided that the article is properly cited, the use is non-commercial and no modifications or adaptations are made.