Proceedings of the 9ICEG
9th International Congress on Environmental Geotechnics
25-28 June, 2023 | Chania, Greece

https://doi.org/10.53243/ICEG2023-79

Effect of cement anisotropy caused by MICP on the mechanical behaviours of bio-improved granular soils

Xing Zhang[1], Bo Zhou[2], Ziyang Wu[3], Huabin Wang[4]

[1]*Mr., Huazhong University of Science and Technology, Wuhan, China; Nanyang Technological University, Singapore, email: xzhang1@hust.edu.cn*

[2]*Dr., Huazhong University of Science and Technology, Wuhan, China, email: zhoubohust@hust.edu.cn*

[3]*Mr., Huazhong University of Science and Technology, Wuhan, China, email: wuziyang@hust.edu.cn*

[4]*Prof., Huazhong University of Science and Technology, Wuhan, China, email: huabin@mail.hust.edu.cn*

ABSTRACT

In recent years, microbial-induced calcium carbonate precipitation (MICP) has emerged as a promising ground improvement method for cementing sand particles together. This study focuses on investigating the anisotropy of bio-cement cohesion behavior at the macroscopic level and evaluates the treating effect on granular soils, specifically calcareous sand and quartz sand. The MICP procedure was performed on specimens of calcareous sand and quartz sand with varying delamination-load angles, treatment rounds, and particle sizes. Subsequently, splitting tensile strength and stiffness of the treated specimens were evaluated through a series of splitting tensile strength tests. Additionally, microstructure and component characterization of the bio-cemented sand were investigated using scanning electron microscopy (SEM) and X-ray diffraction (XRD). The findings indicate that the mechanical behavior of bio-cemented sand is influenced by the gravity-induced bio-cement layer which formed during MICP treatment. Calcareous sand tends to produce more uniform and denser bio-cement compared to quartz sand. Moreover, the irregular particle shape of calcareous sand results in interlocking between particles, leading to higher splitting tensile strength compared to quartz sand specimens that underwent the same MICP treatments.

Keywords: Microbially induced calcium-carbonate precipitation, anisotropy of bio-cement, splitting tensile strength, granular soils

1 INTRODUCTION

A novel ground improvement technique has emerged (Salehzadeh et al., 2012; Goodarzi & Shahnazari) called microbially induced calcium-carbonate precipitation (MICP) (van Paassen et al., 2010; DeJong et al., 2013; Cheng et al., 2014; Montoya & DeJong, 2015; Xiao et al., 2019). In MICP, bacteria were used to initiate chemical reactions to facilitate calcium carbonate precipitation. Sporosarcina pasteurii produces rapidly calcium carbonate crystals when it hydrolyzes urea into carbonate ions (Chu et al., 2014), making it one of the most studied urea hydrolyzing bacteria. Calcium carbonate precipitated during MICP binds the cohesionless granular soils together. The MICP process can be described by the following equations.

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

$$\mathrm{Ca^{2+} + CO_3^{2-} \rightarrow CaCO_3(\,s\,)} \tag{2}$$

Various factors affecting the mechanical behavior of MICP-treated soils have been investigated extensively. Such as properties of granular soils (van Paassen et al., 2009; Nafisi et al., 2018), concentrations of bacteria and reaction solutions (Al Qabany & Soga, 2013), chemical reaction environment (pH and temperature) (Cheng et al., 2014). However, most of these studies focus on the quartz sand, while few studies focus on calcareous sand. It is known that quartz sand particles have a high degree of roundness and a smooth surface (Zhou et al., 2018), which results in the coating of the surface of quartz sand particles and the bridging of the inter-particle gap during MICP (Cui et al., 2017).

Calcareous sand is primarily formed by the weathering of the skeletal remains of marine organisms. It is a type of 'special soil' composed of carbonate calcium as it exhibits poor mechanical properties compared to quartz sand (Coop, 1990; Zhou et al., 2020). As a result of its irregular particle shape and abundance of intra-particle pores, calcareous sand particles' surfaces were coated with bio-cement, and their intra-particle pores were filled with bio-cement during the MICP process (Zhou et al., 2023). Due to the big differences in MICP treatment effect between calcareous sand and quartz sand, their mechanical behaviour will also differ. Tensile splitting tensile, which directly reflects the cohesion strength of bio-cement, is expected to be investigated further.

Furthermore, during MICP, the bio-cement crystals grew in the solution and then stacked and remained static because of various forces (Xiao et al., 2021). There is a possibility that microbially induced calcium-carbonate precipitation exhibits a particular anisotropic microstructure as a result of gravity. Due to this characteristic, different physical properties will be observed in different directions, such as strength and stiffness in the vertical and horizontal directions, which will have an impact on the design of different applications. Foundation reinforcement would be the need to increase the vertical compressive strength and reduce settlement, while breakwaters or slopes would require reinforcement to strengthen the horizontal load capacity. Considering that anisotropy in mechanical behavior affects engineering properties, it is imperative to examine the location and distribution of precipitated carbonate within bio-cemented granular soils (Terzis & Laloui, 2018). Studying pore-scale characteristics using a microfluidic chip experiment (Wang et al., 2021; Xiao et al., 2021, 2022) has demonstrated inhomogeneity in bio-cement crystal distribution, but this only relates to the observation of precipitated carbonate morphology and distribution, and not mechanical properties.

The main objective of this study is to compare the splitting tensile strength of quartz sand and calcareous sand when treated under the same conditions using microbial-induced calcium carbonate precipitation (MICP). The study also aims to quantify the mechanical behavior resulting from the anisotropy of the bio-cement structure. Experimental investigations were conducted to assess the impact of load direction on splitting tensile strength by comparing the behavior of bio-cemented specimens with varying treating rounds and particle sizes. Additionally, microstructure and component characterization were performed using scanning electron microscopy (SEM) and X-ray diffraction (XRD). The findings of this study provide valuable insights into the mechanical behavior of bio-cemented granular soils, shedding light on their performance and potential applications.

2 MATERIALS AND METHODOLOGY

In this study, the calcareous sand and quartz sand used were collected from the Spratly Islands in the South China Sea and Fujian province in China, respectively. Two particle size ranges were sieved for the sand, namely 0.5 to 1.0 mm and 1.0 to 2.0 mm. Figure 1 provides a comparison of the two sands, where the calcareous sand is characterized by irregular shapes and pores containing biological remains. On the other hand, quartz sand exhibits different physical properties, such as higher roundness and smoothness compared to calcareous sand.

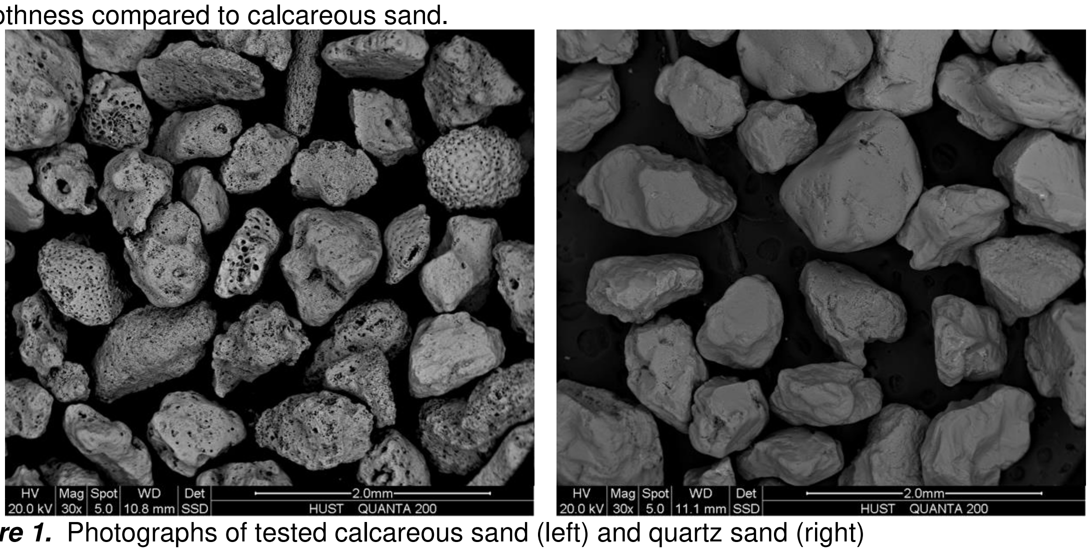

Figure 1. Photographs of tested calcareous sand (left) and quartz sand (right)

Sporosarcina pasteurii (ATCC 11859), a commonly used bacterium, was selected for MICP treatment. We grew bacterial cells obtained from the frozen stock in a liquid medium containing 20 g/L yeast extract

(NH_4-YE), 10 g/L NH_4Cl, 12 mg/L $MnSO_4 \cdot H_2O$, and 24 mg/L $NiCl_2 \cdot 6H_2O$ at a pH of 8.5 and at a temperature of 30°C. A 24-hour culture of the bacterial suspension yielded an average urease activity of 9.9 - 10.2 mM urea hydrolyzed/min. An ultraviolet spectrophotometer was used to determine the optical density (OD_{600}) of the obtained bacterial suspension, which was in the range of OD_{600} = 1.8 – 2.0. Based on earlier studies (Al Qabany & Soga, 2013), the cementation solution was composed of urea (CON_2H_4) and $CaCl_2$, each with a concentration of 0.5 mol/L.

To make the specimens, a specially designed mold was used to produce specimens of calcareous sand and quartz sand. These specimens had a diameter of 38 mm and a length of 30 mm. The mold was made from geotextile with a thickness of 2 mm and a polyethylene bracket. As Figure 2 illustrates, the geotextile maintains the cylindrical shape of the loose sand and permits the infiltration of bacterial suspension and cementation solution into the specimens. The sand was poured into the mold and compacted to a controlled relative density of 0.62 ± 0.02.

The procedures of every MICP treatment round are followed: (1) putting the specimens into polystyrene containers, adding bacterial suspension (approximately 300 mL per specimen) and immersing each specimen in the bacterial suspension for 24 hours in an incubator at 30°C. (2) transferring these specimens into a new empty container inside the incubator, and slowly adding cementation solution (approximately 500 mL per specimen) and immersing them in the cementation solution for 24 hours at 30°C to trigger carbonate precipitation within each specimen. (3) repeat step (2). During every round of MICP treatment, specimens were immersed in bacterial suspension for 24 hours and in cementation solution for 48 hours.

Once the MICP treatment was completed, the molds were removed and the soluble salts (e.g., calcium chloride) inside the specimens were washed away with deionized water. After that, the specimens were completely dried at 60°C for a period. As a final step, all bio-cemented specimens were ready for the subsequent splitting tensile strength test.

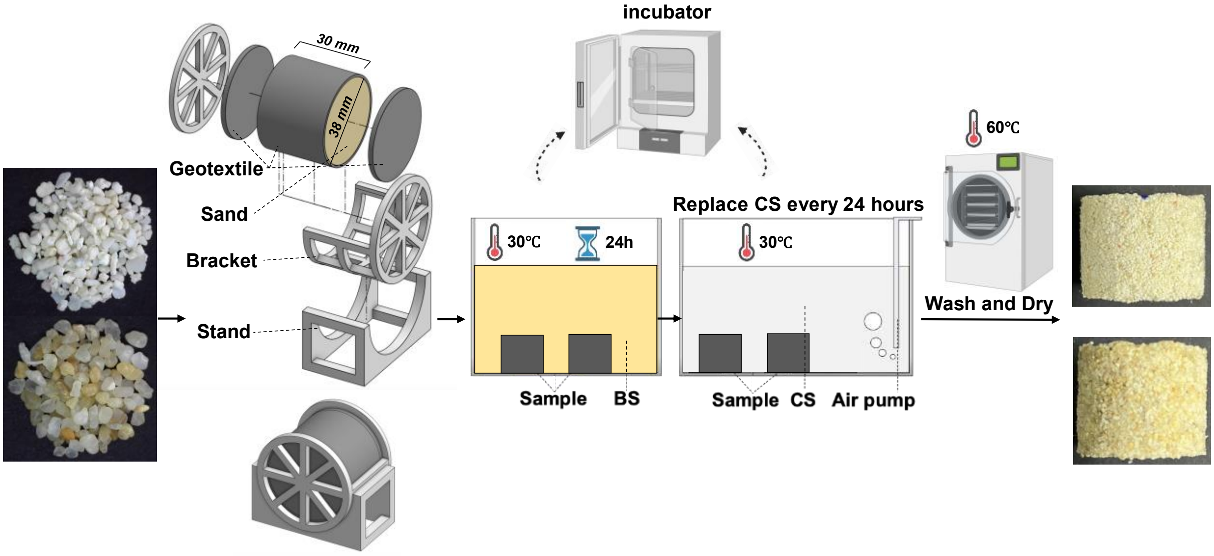

Figure 2. *Procedures of MICP treatment*

The bio-cement content is a major index to describe the degree of calcium carbonate precipitation, it will increase with the increasing of treating rounds. In this study, according to the MICP treating rounds, the specimens were divided into three groups, including 1-round, 3-rounds, and 5-rounds. Bio-cement content *C* of both calcareous sand and quartz sand was calculated by the following Equation (3):

$$C = \frac{M_1 - M_0}{M_0} \times 100\% \quad (3)$$

Where M_0 is the weight of untreated dry sand, and M_1 is the weight of the bio-cemented sand in a dry state.

Additionally, the presence of calcium carbonate crystals resulting from the MICP process was observed to deposit on the surface of sand particles due to gravity, forming a layered structure as depicted in

Figure 3. To investigate the anisotropic behavior of tensile failure strength resulting from this layered structure, three deposition-load angles, namely 0°, 45°, and 90°, were chosen as the angles between the gravity deposition direction during MICP treatment and the loading direction during the splitting tensile strength test, as shown in Figure 3. The SRDL-50 testing machine (Changchun Research Institute for Mechanical Science, Changchun, China) was used for conducting the splitting tensile strength tests.

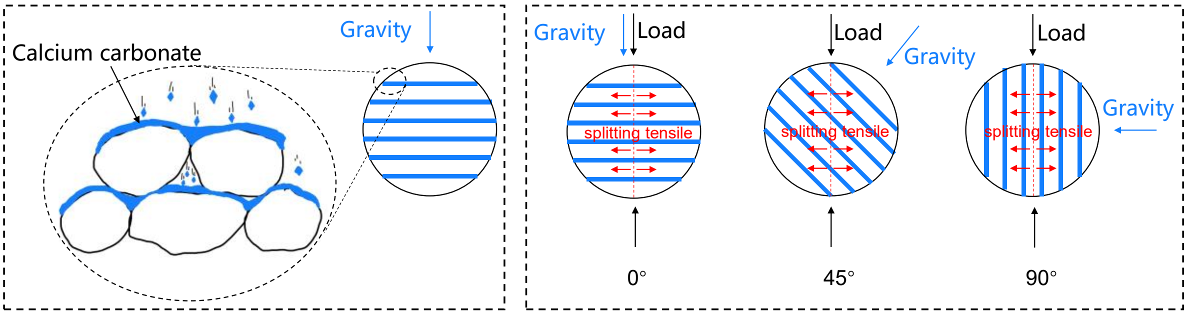

Figure 3. Layered structure of bio-cement and the deposition-load angle

3 RESULTS AND DISCUSSIONS

3.1 Strength and stiffness

The mechanical behavior of bio-cemented sand was evaluated using strength and stiffness as indicators. Stiffness was determined as the tangent modulus at 50% of the peak stress on the stress-strain curve, while strength was defined as the peak stress on the stress-strain curve (Van Paassen et al., 2010). The relationship between strength and bio-cement content, as well as stiffness and bio-cement content, was plotted in Figure 4 and Figure 5, respectively, under different deposition-load angles. Both calcareous sand and quartz sand, with different particle sizes, exhibited anisotropy in their mechanical behavior.

The bio-cement content in calcareous sand was higher compared to quartz sand under the same conditions. The bio-cement content in calcareous sand ranged from approximately 0.19-0.22, 0.29-0.33, and 0.35-0.40 for 1-round, 3-rounds, and 5-rounds of MICP treatment, respectively. In contrast, the bio-cement content in quartz sand ranged from approximately 0.04-0.06, 0.13-0.2, and 0.21-0.25 for 1-round, 3-rounds, and 5-rounds of MICP treatment, respectively.

The higher bio-cement content in calcareous sand can be attributed to the presence of abundant intra-particle pores and surface holes, which enhance the contact area between calcareous sand particles and the MICP reaction solution. The bio-cement in calcareous sand not only coats the surface of particles but also fills the intra-particle pores. On the other hand, in quartz sand, the bio-cement only coats the surface of particles without filling the intra-particle pores. However, it should be noted that although the bio-cement filling the intra-particle pores in calcareous sand results in a higher percentage of bio-cement content compared to quartz sand, it does not contribute to the bonding between particles (Zhou et al., 2023). As a result, the splitting tensile strength of MICP-treated calcareous sand is activated when the bio-cement content reaches 0.19, whereas for quartz sand, the splitting tensile strength is activated when the bio-cement content reaches 0.04.

The splitting tensile strength gaps between bio-cemented calcareous sand and quartz sand increased with increasing bio-cement content for different deposition-load angles. Specimens with a deposition-load angle of 0° exhibited the highest splitting tensile strength, while specimens with a deposition-load angle of 90° exhibited the lowest splitting tensile strength. For specimens with particle sizes ranging from 0.5 to 1.0 mm, the splitting tensile strength of bio-cemented calcareous sand ranged from approximately 0.03 to 2.0 MPa, while for bio-cemented quartz sand, it ranged from approximately 0.02 to 1.5 MPa. Similarly, for specimens with particle sizes ranging from 1.0 to 2.0 mm, the splitting tensile strength of bio-cemented calcareous sand ranged from approximately 0.02 to 1.2 MPa, while for bio-cemented quartz sand, it ranged from approximately 0.02 to 0.8 MPa. Overall, bio-cemented calcareous sand exhibited higher splitting tensile strength than bio-cemented quartz sand under the same MICP treatment conditions. Additionally, specimens with smaller particle sizes for both types of bio-cemented sand displayed higher cumulative bio-cement content and higher splitting tensile strength.

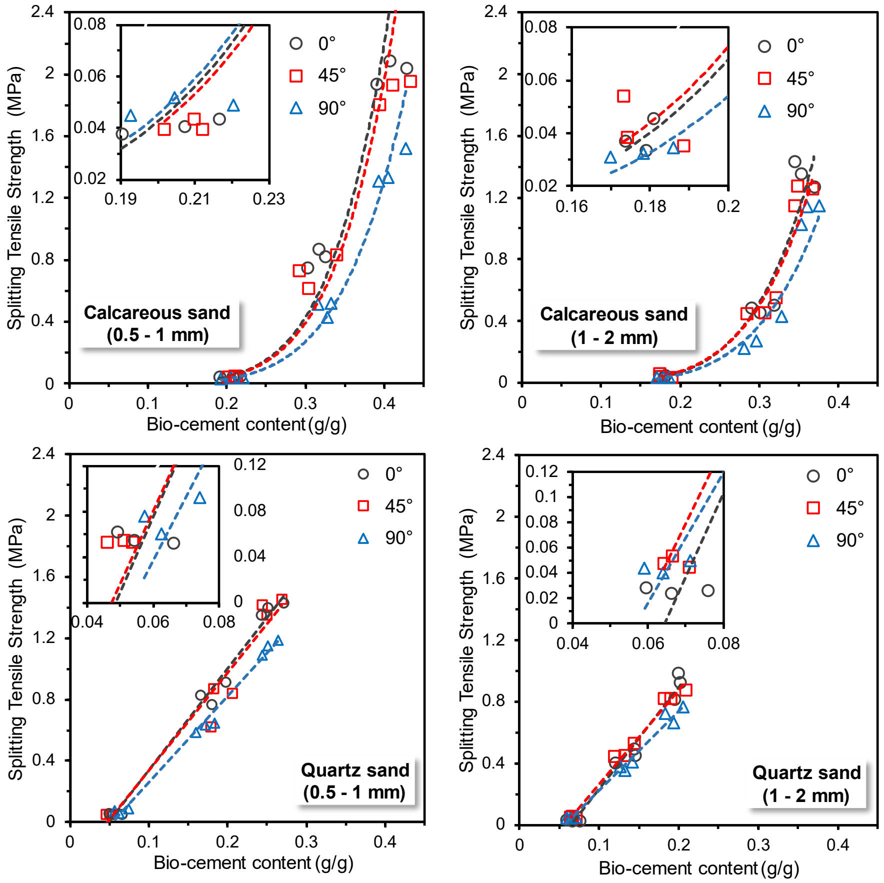

Figure 4. *Relationship between splitting tensile strength and bio-cement content*

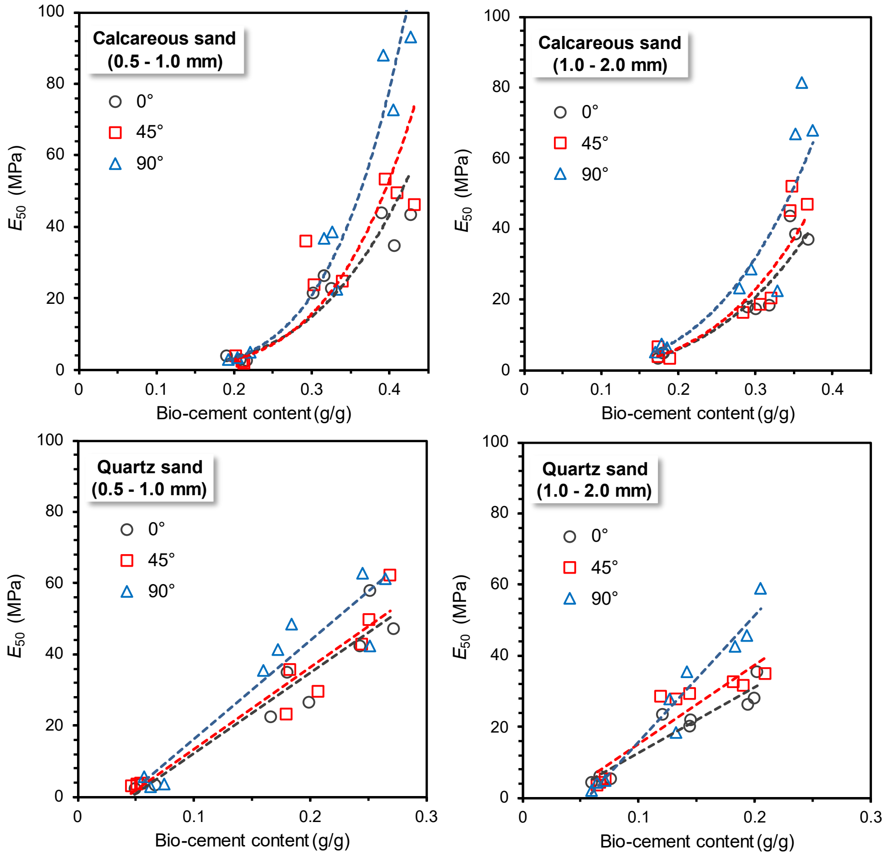

Figure 5. *Relationship between E_{50} and bio-cement content*

The stiffness of the bio-cemented specimens showed a distinct trend with varying deposition-load angles. When the deposition-load angle was set at 90°, the stiffness of the specimens reached its maximum. As the deposition-load angle was reduced to 45°, the stiffness weakened, and it reached its lowest point when the deposition-load angle was further reduced to 0°. For specimens with particle sizes ranging from 0.5 to 1.0 mm, the stiffness of bio-cemented calcareous sand ranged from approximately 4 to 90 MPa, while for bio-cemented quartz sand, it ranged from approximately 4 to 60 MPa. Similarly, for specimens with particle sizes ranging from 1.0 to 2.0 mm, bio-cemented calcareous sand exhibited a stiffness range of approximately 4 to 70 MPa, while bio-cemented quartz sand exhibited a stiffness range of approximately 4 to 50 MPa. Overall, under the same MICP treatment conditions, bio-cemented calcareous sand demonstrated superior stiffness compared to bio-cemented quartz sand. Additionally, specimens with smaller particle sizes exhibited higher stiffness for both types of bio-cemented sands.

The results obtained from the above findings reveal an interesting trend: the tensile strength and stiffness of the sample exhibit opposite trends with changes in the loading-deposition angle. This can be observed from Fig. 3. When the deposition-load angle is 0°, the bio-cement microstructure along the loading direction appears sparse, resulting in lower stiffness. However, the bio-cement along the direction of tensile stress is denser, resulting in higher tensile strength. Conversely, when the deposition-load angle is 90°, the bio-cement microstructure along the loading direction is denser, leading to higher stiffness. However, the connection between the bio-cementation layers is weaker, making them prone to failure under horizontal tension generated by loading, resulting in lower tensile strength.

3.2 Microstructure characteristics

In order to further understand the mechanism of MICP treatment and the differences observed in bio-cement content and splitting tensile strength between calcareous and quartz sands, microstructure and component characterization of bio-cement can provide valuable insights. For this purpose, three specimens were separately ground into powder for phase identification: calcareous sand before MICP treatment, calcareous sand after MICP treatment, and quartz sand after MICP treatment. X-ray diffraction (XRD) analysis, as shown in Figure 6, revealed that the mineral component of calcareous sand was aragonite, whereas the mineral component of quartz sand was quartz. On the other hand, the bio-cement produced during MICP treatments was identified as calcite based on the XRD analysis. This characterization of the microstructure and components of bio-cement provides important information for understanding the MICP treatment mechanism and the differences observed in calcareous and quartz sands.

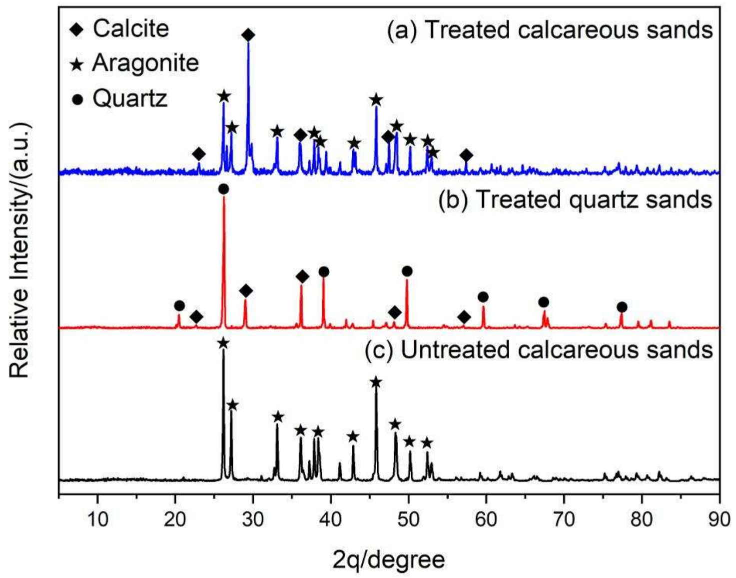

Figure 6. *XRD phase identification of the test materials*

The results of the scanning electron microscopy (SEM) test were depicted in Figure 7. After MICP treatment, it was observed that nearly all surfaces of the calcareous sand were coated with bio-cement crystals, whereas the quartz sand surfaces were not fully coated and exhibited an agglomerated morphology. This could be attributed to the differences in the natural morphology of calcareous and quartz sands. Calcareous sand typically has irregular particle shapes and contains a higher number of intra-particle pores, which provide more opportunities for bacteria and bio-cement crystals to be absorbed. In contrast, quartz sand has rounder particles with smoother surfaces and fewer intra-particle pores, resulting in less intense adsorption of bacteria and bio-cement crystals.

Furthermore, due to the irregular shapes of calcareous sand particles, they tend to interlock and form denser bio-cement structures. On the other hand, the high roundness of quartz sand particles makes them less prone to interlocking. This difference in particle morphology may explain why the splitting tensile strength and stiffness of calcareous sand were greater than those of quartz sand, as evidenced in Figures 4 and 5. The interlocking of calcareous sand particles facilitated by bio-cementation could result in a stronger and stiffer matrix compared to quartz sand, which had less interlocking due to its smoother and rounder particles.

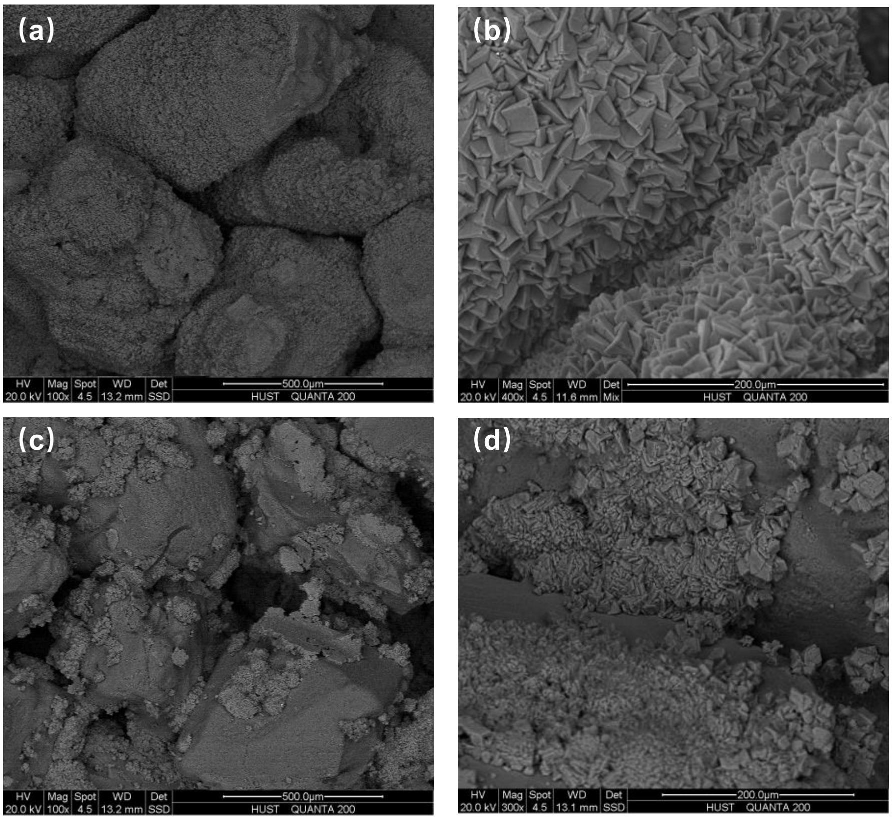

Figure 7. *SEM images of MICP treated (a)(b) calcareous sand and (c)(d) quartz sand*

4 CONCLUSIONS

This study examined the treating effect and anisotropic mechanical behavior of MICP-treated calcareous sand and quartz sand and provided a deep understanding of their MICP-treated mechanisms. In summary, the following three conclusions can be drawn:
(1) Calcareous sand surfaces with dense pores, as opposed to quartz sand with a smooth surface, promoted greater production of bio-cement. The surfaces of calcareous sand particles were uniformly coated, and the intra-particle pores were filled. In contrast, bio-cements coated quartz sand surfaces in agglomeration-like pattern.
(2) Compared to quartz sand, MICP-treated specimens of calcareous sand exhibited higher splitting tensile strength and stiffness due to interlocking of particles and dense bio-cement structures. Smaller particle sizes resulted in denser bio-cement and better mechanical properties of the treated specimens.
(3) During the MICP process, bio-cement crystals were observed to form horizontally layered structures, leading to anisotropy in splitting tensile strength and stiffness of the treated specimens. In particular, the direction parallel to gravity (deposition-load angle = 0°) exhibited the highest strength and lowest stiffness. Conversely, the direction vertical to gravity (deposition-load angle = 90°) showed the lowest strength and highest stiffness.

5 ACKNOWLEDGEMENTS

This study was supported by Research Grants 41877233, 42072298 and 41931286 from the National Natural Science Foundation of China.

REFERENCES

Al Qabany, A, Soga K (2013) Effect of chemical treatment used in MICP on engineering properties of cemented soils. Ge´otechnique 63:331–339

Cheng L, Shahin M. A., Cord-Ruwisch R (2014) Bio-cementation of sandy soil using microbially induced carbonate precipitation for marine environments. Ge´otechnique 64:1010–1013

Cheng, L., Shahin, M. A., & Chu, J. (2019). Soil bio-cementation using a new one-phase low-pH injection method. Acta Geotechnica, 14(3), 615-626.

Chu J et al (2014) Optimization of calcium-based bioclogging and biocementation of sand. Acta Geotech 9:277–285

Coop, M. R. (1990). The mechanics of uncemented carbonate sands. Géotechnique, 40(4), 607-626.

Cui M. J., Zheng J. J., Zhang RJ, Lai H. J., Zhang J. (2017) Influence of cementation level on the strength behaviour of bio-cemented sand. Acta Geotech 12:971–986

DeJong J. T., Soga K., Kavazanjian E. et al (2013) Biogeochemical processes and geotechnical applications: progress, opportunities and challenges. Ge´otechnique 63:287–301

Goodarzi, S., & Shahnazari, H. (2019). Strength enhancement of geotextile-reinforced carbonate sand. Geotextiles and Geomembranes, 47(2), 128-139.

Montoya B. M., DeJong J. T. (2015) Stress-strain behavior of sands cemented by microbially induced calcite precipitation. J Geotech Geoenviron Eng 141:04015019

Nafisi, A., Montoya, B. M., & Evans, T. M. (2020). Shear strength envelopes of biocemented sands with varying particle size and cementation level. Journal of Geotechnical and Geoenvironmental Engineering, 146(3), 04020002.

Salehzadeh, H., Hassanlourad, M., & Shahnazari, H. (2012). Shear behavior of chemically grouted carbonate sands. International Journal of Geotechnical Engineering, 6(4), 445-454.

Terzis, D., & Laloui, L. (2018). 3-D micro-architecture and mechanical response of soil cemented via microbial-induced calcite precipitation. Scientific reports, 8(1), 1-11.

van Paassen, L. A., Van Loosdrecht, M. C. M., Pieron, M., Mulder, A., Ngan-Tillard, D. J. M., & Van der Linden, T. J. M. (2009). Strength and deformation of biologically cemented sandstone. In ISRM Regional Symposium-EUROCK 2009. OnePetro.

van Paassen L. A., Ghose R., van der Linden T. J., van der Star W. R., van Loosdrecht M. C. (2010) Quantifying biomediated ground improvement by ureolysis: large-scale biogrout experiment. J Geotech Geoenviron Eng 136:1721–1728

Wang, Y., ASCE, M., Soga, K., ASCE, F., DeJong, J. T., ASCE, F., & Kabla, A. J. (2021). Effects of Bacterial Density on Growth Rate and Characteristics of Microbial-Induced CaCO3 Precipitates: Particle-Scale Experimental Study. J. Geotech. Geoenviron. Eng, 147(6), 04021036.

Xiao, Y., He, X., Evans, T. M., Stuedlein, A. W., & Liu, H. (2019). Unconfined compressive and splitting tensile strength of basalt fiber–reinforced biocemented sand. Journal of Geotechnical and Geoenvironmental Engineering, 145(9), 04019048.

Xiao, Y., He, X., Wu, W., Stuedlein, A. W., Evans, T. M., Chu, J., ... & Wu, H. (2021). Kinetic biomineralization through microfluidic chip tests. Acta Geotechnica, 16(10), 3229-3237.

Xiao, Y., He, X., Stuedlein, A. W., Chu, J., Evans, T. M., & Van Paassen, L. A. (2022). Crystal Growth of MICP through Microfluidic Chip Tests. Journal of Geotechnical and Geoenvironmental Engineering, 148(5), 06022002

Zhou, B., Wang, J., & Wang, H. (2018). Three-dimensional sphericity, roundness and fractal dimension of sand particles. Géotechnique, 68(1), 18-30.

Zhou B, Ku Q, Wang H, Wang J (2020) Particle classification and intra-particle pore structure of carbonate sands. Eng Geol 279:105889

Zhou, B., Zhang, X., Wang, J., Wang, H., & Shen, J. (2023). Insight into the mechanism of microbially induced carbonate precipitation treatment of bio-improved calcareous sand particles. Acta Geotechnica, 18: 985–999.