

# Effects of Hydroxypropyl Methylcellulose (HPMC) on the Reinforcement of Sand by Microbial-Induced Calcium Carbonate Precipitation (MICP)

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**Abstract:** Microbial-induced calcium carbonate precipitation (MICP) is a new technology used for reinforcing soils through microbial mineralization. However, the existing MICP treatment technology is more suitable for deeper soils due to its high permeability. In this study, HPMC, a cohesive material combined with *Sporosarcina pasteurii*-induced calcium carbonate precipitation was used to improve the surface layer of the soil. It was also tested in different contents of bacterial solutions and cementation solutions and with a different number of MICP treatments, and was analyzed and discussed by measuring the surface layer strength, calcium carbonate content generated in the crust, rainfall erosion resistance, wind erosion resistance, and ammonia retention rate of the specimens. The microstructure of the samples was investigated by XRD and SEM. It was shown that the addition of HPMC and increasing the number of MICP treatments were effective in the increase in the calcium carbonate content in the surface crust, but had no effect on the total amount of induced calcium carbonate. The combination of HPMC addition and MICP technology can effectively reduce rainwater scour loss and wind erosion loss, and increase its rainfall erosion resistance and wind erosion resistance. Rainfall losses were reduced by up to 30% in specimens treated with HPMC for MICP technology. When the wind speed was 12 m/s, the maximum mass loss rate of the specimens treated with HPMC for MICP was only 0.828%. The addition of HPMC can effectively improve the ammonia absorption rate and reduce the release of ammonia in the process of MICP technology, which is of great significance for environmental protection. The microstructure showed that the addition of HPMC and the increase in the number of treatments using MICP technology can make the surface structure of the specimens more compact, and the calcium carbonate can more effectively fill the pores and cement the soil particles, while the addition of HPMC may not change the calcium carbonate crystal type.

**Keywords:** MICP; calcium carbonate; HPMC; surface strength; ammonia retention rate; wind erosion resistance

## 1. Introduction

Soil surface treatment can improve soil erosion resistance, strength, and stability, which is of great significance in soil–water conservation and dust prevention. It is practical to improve the soil surface using physical methods (such as covering dust-proof net) and using chemical substances (such as cement, lime, fly ash, gypsum, surfactant, polymer, asphalt products, waterborne polyurethane, etc.) [1]. In recent years, the application

of MICP in soil improvement has attracted extensive attention [2]. MICP technology mainly uses bacteria to produce urease to hydrolyze urea and generate  $\text{CaCO}_3$  to achieve cementation and reinforcement. Although MICP technology presents greater implementation complexity due to the use of living organisms, and has a higher cost compared with traditional technologies [1], it has broad application prospects in soil improvement, concrete crack repair, erosion prevention, cultural relic restoration, and heavy metal pollution control [3–7] due to its environmental friendliness and sustainability [8,9].

MICP can effectively cement soil particles together and improve the performance of soil. Cheng et al. [10] used surface filtration technology to place bacteria and cementation solution. Under high permeability, bacteria and cementation solutions could be used alternately many times to fix bacteria in a column of more than 1 m, and the homogenization of the improved sand column reached a reasonable degree. Van Paassen et al. [11] used MICP technology to carry out a reinforcement test on a 100 m<sup>3</sup> large sand foundation, which effectively improved the bearing capacity of the sand foundation. Moreover, soil surface could also be reinforced by MICP. Stabnikov et al. [12] used a mixture of calcium salts, urea, and bacterial suspension to make a crust on the surface of the sandy soil, which reduced the permeability of the sand layer from  $10^{-4}$  m/s to  $16 \times 10^{-7}$  m/s (14 mm/d). Xiao et al. [13] used a surface spraying method and an MICP technique to surface treat a series of model slopes prepared by different grades of sand, which effectively reduced the rainfall erosion of sandy slopes. Liu et al. [14] applied MICP treatment to five groups of soil slope model samples, and showed that MICP treatment is an effective method to control the surface erosion of clayey soil by analyzing the soil erosion pattern and soil loss rate. Zomorodian et al. [15] studied the application of the MICP technique in different grades of silica sand and carbonate rocks, determined the optimum reagent concentration, and investigated the variation pattern of shear strength and resistance to wind erosion after a number of different treatments. The wind tunnel tests showed that the treated crustal sands could remain stable at a wind speed of 20 m/s.

The desired result of surface reinforcement should be a significant increase in soil strength within the shallow layer [16]. However, the existing MICP treatment solutions, such as grouting or surface penetration, may be difficult to achieve this desired treatment due to their higher permeability, which is more suitable for deeper treatment. Due to the large permeability of the soil to be treated, existing studies generally use micro-dose injection of MICP solution in order to make the MICP treatment solution and to more precisely reinforce the shallow soil. In addition, in order to achieve the desired treatment effect, the MICP solution is generally infiltrated several times. Salifu et al. [17] injected the bacterial solution and the cementing solution into the soil 18 treatments. In the experiments of Shanahan and Montoya et al. [18], they also made multiple infiltrations to reach the target due to the large permeability.

Materials such as hydrogels (xcan, ganlan) or polyvinyl alcohol (PVA) can reach such shallow depths of cementation, whereby the crust is formed by cementing the shallow layers to improve the soil surface [19]. Hamdan et al. [20] used hydrogel-assisted enzyme-induced carbonate precipitation (EICP), which showed that hydrogels retained water for a longer time, reduced the infiltration of solution in the soil, prolonged the reaction time, improved the efficiency of precipitation, and enhanced the formation of the crust. Sun et al. [21] utilized a biopolymer-assisted EICP approach to improve soil water retention and reduce permeability by retaining carbonate around soil particles. Based on MICP technology, Wang et al. [22] used polyvinyl alcohol (PVA) solution instead of water to prepare cementitious solution. The viscous polymer solution immobilized bacteria and cementitious medium on the surface, promoted calcium carbonate precipitation, formed uniform soil crust on the surface area, and reduced the corrosion ability of the soil.

Hydroxypropyl methylcellulose (HPMC) is abundant, cheap, and biocompatible [23]. A gel can be formed by heating the solution. HPMC, because of its good water retention performance, is widely used in cement mortar as water retention agent and retarder,

to improve the hardening strength, save the amount of cement, and then achieve the purpose of cost-saving. Because of its viscosity, HPMC is combined with MICP technology to react in the soil surface better, forming a hard crust on the surface and enhancing the surface strength.

As mentioned above, MICP reinforcement of surficial soil is usually not ideal due to the high permeability of the soil; therefore, it has great potential for research on effectively stabilizing the surface layer of soil via MICP technology. In this study, a series of laboratory experiments were conducted to investigate the feasibility of using MICP and HPMC in the surficial treatment of soil for the purpose of improving the properties of the soil surface. The surficial treatment and ammonia adsorption were achieved by utilizing MICP and HPMC on the soil surface. The macro-scale characteristics of the treated specimens were studied, including the surface strength of sand, the content of  $\text{CaCO}_3$ , and the resistance to wind and rain erosion. Scanning electron microscopy (SEM) and X-ray diffraction (XRD) were used to further analyze the microstructure and formation mechanism under different treatments.

## 2. Materials and Methods

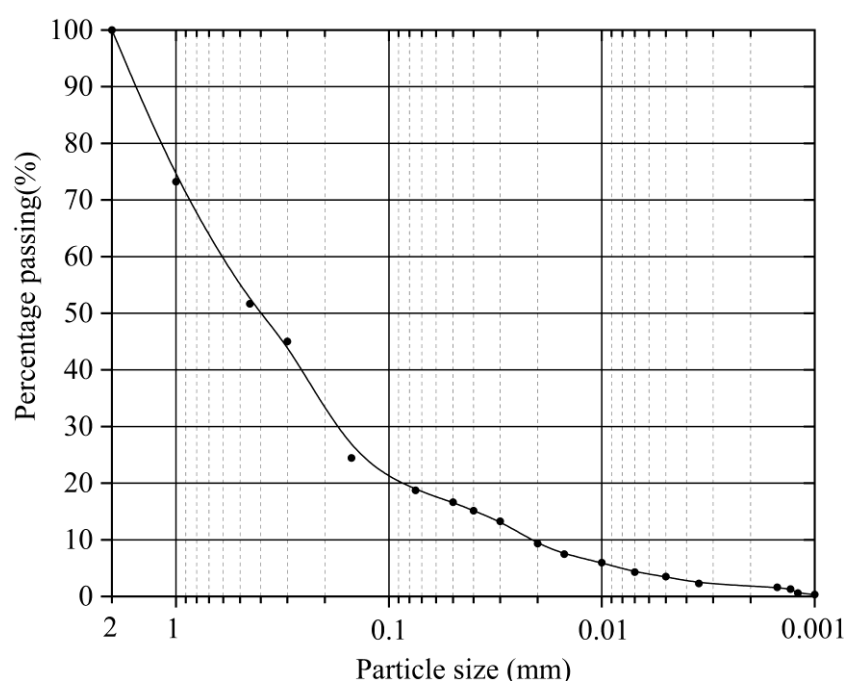
### 2.1. Biological Treatment Process

The microorganism used in this study was *Sporosarcina pasteurii*, a kind of bacteria that can produce urease [24], purchased from China General Microbiological Culture Collection Centre (CMCC1.3687). In this study, optical density was measured by a spectrophotometer at a wavelength of 600 nm (OD600), and the urease activity of bacteria was determined at 25 °C by the conductivity method [25].

Under aerobic conditions at 32 °C, first, the bacteria were pre-cultured for about 8 h in a medium containing peptone (5 g/L), beef extract (3 g/L), and urea (20 g/L). Then, the bacteria were cultured in a medium containing yeast powder (20 g/L) and ammonium sulfate (10 g/L) at 5% inoculum for about 24 h. Both media used in the study had a pH of 8 [26]. The urease activity of the bacterial solution used in the study was controlled at  $22 \pm 2$  mM urea/min, and the OD600 was  $3 \pm 0.5$ .

### 2.2. Properties of the Soil and HPMC

The gradation curve of the tested soil is shown in Figure 1, wherein  $D_{10}$ ,  $D_{30}$ , and  $D_{60}$  were 0.02 mm, 0.18 mm, and 0.62 mm, respectively. In addition, the coefficient of uniformity ( $C_u$ ) and the coefficient of gradation ( $C_g$ ) of the soil sample were 3.10 and 2.61, respectively. According to ASTM D2487, the tested soil used in this study was silty sand, which was sterilized at 160 °C for 24 h.



**Figure 1.** Particle gradation curve.

In this study, HPMC was used as the gelling material, which was purchased from Tianjin Kemeiou Chemical Reagent Co., Ltd. (Tianjin, China). HPMC is a fibrous white powder at room temperature. HPMC has relatively stable properties, superb thickening, cohesiveness, and good compatibility with water. Moreover, HPMC has good film-forming properties and is safe, non-toxic, and non-polluting. The basic physical parameters of HPMC are shown in Table 1. The viscosity of HPMC used in this study was 25 mpa.s.

**Table 1.** Basic physical parameters of HPMC.

Name	Density	Specific Gravity	Viscosity (mpa.s)	Surface Tension (dyn/cm)
HPMC	0.5	1.3	5~200,000	42~56

### 2.3. Sample Preparation and MICP Procedure

In this study, cuboid boxes made of transparent plastic were used as molds, whose length, width, and height were 100 mm, 70 mm, and 40 mm, respectively. To evenly pour the sand into the mold, a funnel was used [27], and the height was flush with the mold. A number of uniform 1 mm holes were drilled at the bottom of each mold to allow the gas to be discharged in the pores during grouting and the gas generated during MICP.

MICP treatment was performed in the laboratory at  $25 \pm 2$  °C. The cementation solution is a mixed solution of urea (1.5 M) and  $\text{CaCl}_2$  (1.0 M). The ratio of bacterial solution to cementation solution was 1:4. There were four gradients in total, and each gradient was sprayed once, twice, and three times for surface MICP treatment, with an interval of 12 h. The bacterial solution and the cementation solution were mixed in a ratio of 1:4, and the mixed solution was sprayed evenly on the surface layer of the specimen using a spray bottle. Four solution volume gradients were set in the test, and the ratio of spraying was controlled when spraying the mixed solution so that the same volume of solution was sprayed under the same gradient. The mixed solutions were sprayed at 0.7, 1.1, 1.4, and 1.7 mL/cm<sup>2</sup> under the four gradients. Among them, for the HPMC combined with MICP-treated specimens, HPMC was dissolved in a mixture of bacterial solution and cementation solution at room temperature of 25 °C before spraying at the same ratio as the MICP-treated specimens.

#### 2.4. Tests and Methods

The content of  $\text{CaCO}_3$  was determined using acid digestion [28]. It is worth noting that the determination of the  $\text{CaCO}_3$  content was not carried out on the whole sample, but separated the crust formed after MICP treatment from the lower soil to measure the content of calcium carbonate in each part.

Using an ultraviolet–visible spectrophotometer, the concentration of ammonium ion was tested using the Nessler method [26]. Under a constant temperature of 25 °C, the MICP-treated sample was placed in a conical flask with distilled water and oscillated at a rate of 120 r/min for 12 h. Then, 50 mL of supernatant was directly taken, and the absorbance was measured. The ammonia concentration in the supernatant can be calculated as follows:

$$\rho_N = \frac{A_s - A_b - a}{b \times V} \quad (1)$$

where  $\rho_N$  is the mass concentration of ammonia in the supernatant (mg/L);  $A_s$  is the absorbance of water specimen;  $A_b$  is the absorbance of blank control;  $a$  is the intercept of calibration curve;  $b$  is the calibrate the slope of the curve;  $V$  is the volume of the specimen solution.

The concept of the ammonia retention rate was introduced in the study. Ammonia retention rate  $N$  was defined as the ratio of ammonia amount after MICP complete reaction to ammonia amount before MICP. The ammonia retention rate  $N$  was calculated using the following formula:

$$N = \frac{M_2}{M_1} \quad (2)$$

where  $M_2$  is the mass of ammonia in the specimen after reaction;  $M_1$  is the mass of ammonia in the sample before the reaction.

The soil particles washed out by rainfall were collected by simulating the scouring of the surface layer of the MICP cured specimens. Three rainfall intensities of 0.8, 1.5, and 2.5 mm/min were set for this test, corresponding to light, medium, and heavy rainfall, respectively. The slope of the test specimen was adjusted to 30°, and the continuous scouring was carried out for 10 min under each rainfall intensity. The mass loss before and after the simulated rainfall test was calculated. The apparatus used in the trial is shown in Figure 2.

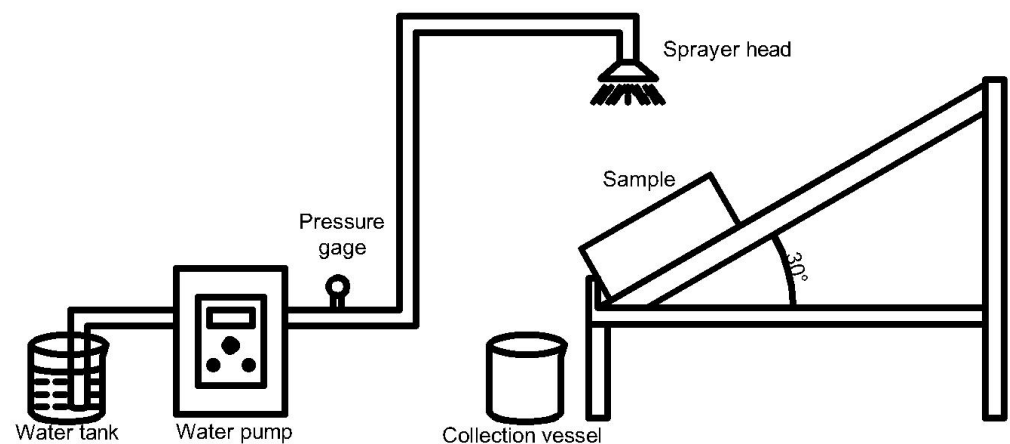
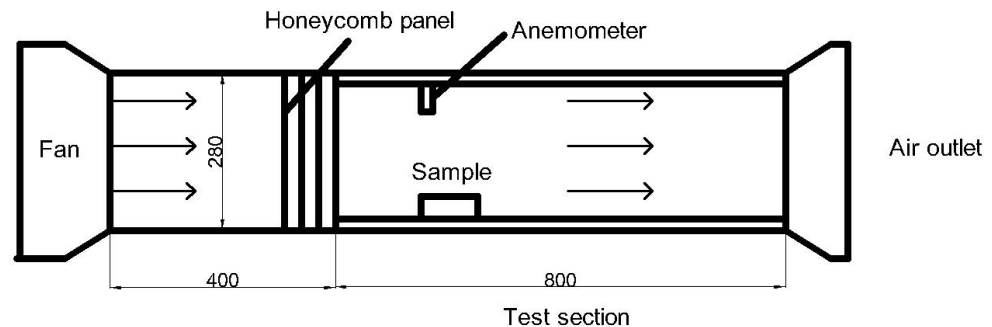


Figure 2. Rainfall device diagram.

Using the apparatus in Figure 3, wind erosion resistance tests were performed on the MICP-treated and MICP combined with HPMC-treated specimens at wind speeds of 4 and 12 m/s, respectively. The mass loss of the specimens before and after the wind erosion

tests was measured, and the mass loss rate of the specimens was calculated for different amounts of MICP solution and at different times of treatment.



**Figure 3.** Diagram of wind erosion resistance device.

There are four gradients and three processes for MICP grouting. The four gradients are I: 10 mL bacterial solution + 40 mL cementation solution; II: 15 mL bacterial solution + 60 mL cementation solution; III: 20 mL bacterial solution + 80 mL cementation solution; IV: 24 mL bacterial solution + 96 mL cementation solution. The three processes were divided into one time, two times, and three times to spray uniformly on the surface of the specimen, with an interval of 12 h. The solution with HPMC composition was sprayed one time, two times, and three times as G1, G2, and G3, respectively. The viscosity of the solution was 25 mpa.s by adding HPMC. Similarly, the experiment was carried out according to the above scheme without the addition of HPMC and was compared with the specimen with the addition of HPMC for MICP treatment. Among them, the solution without HPMC component was sprayed once, twice, and three times, which were respectively marked as G4, G5, and G6. The samples after MICP treatment were dried for 48 h in an oven at 60 °C. Under the same process and the same amount of solution, eight samples were prepared, respectively, with and without HPMC. The surface strength of 2 samples was measured by a pocket penetrometer, and the samples collected after the determination were tested for ammonia mass concentration. Two samples were tested for calcium carbonate content in the upper crust and lower crust, two specimens were tested for rain erosion resistance under simulated rainfall conditions, and two specimens were tested for wind erosion resistance in a wind tunnel instrument.

### 3. Results and Discussion

#### 3.1. Calcium Carbonate Content

The acid digestion method was used to measure the content of calcium carbonate in specimens treated by MICP. The content of calcium carbonate generated by each experimental group is shown in Figure 4, and the percentage of calcium carbonate production in the 3–7 mm surficial layer in the total amount of calcium carbonate generated is shown in Figure 5. The results showed that the precipitation of calcium carbonate at the thinner skin layer was effectively improved by the addition of HPMC, and the highest percentage of calcium carbonate to total calcium carbonate in the hard shell reached 78.05%. However, as for the total  $\text{CaCO}_3$  content in the samples, the total  $\text{CaCO}_3$  mass of the samples with and without HPMC is roughly the same after MICP treatment. This phenomenon illustrated that HPMC did not improve the total content of calcium carbonate, but could enhance the viscosity of reactants and increase the time of liquid infiltration so that more calcium carbonate precipitation was generated at the thinner surficial layer. At the same reaction amount, the highest calcium carbonate produced in the surficial layer with the addition of HPMC for MICP treatment could reach 1.82 times higher than that with only MICP treatment.

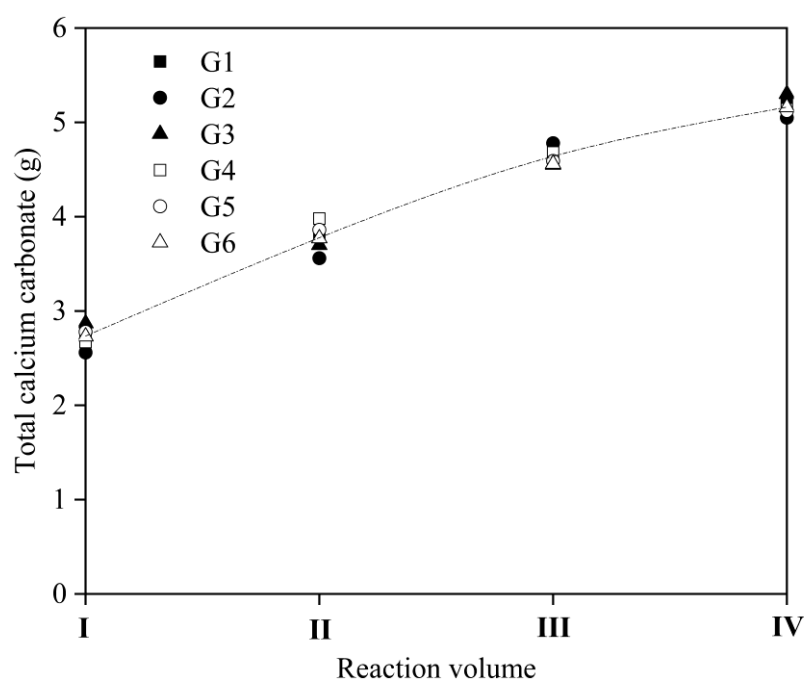


Figure 4. Total calcium carbonate content.

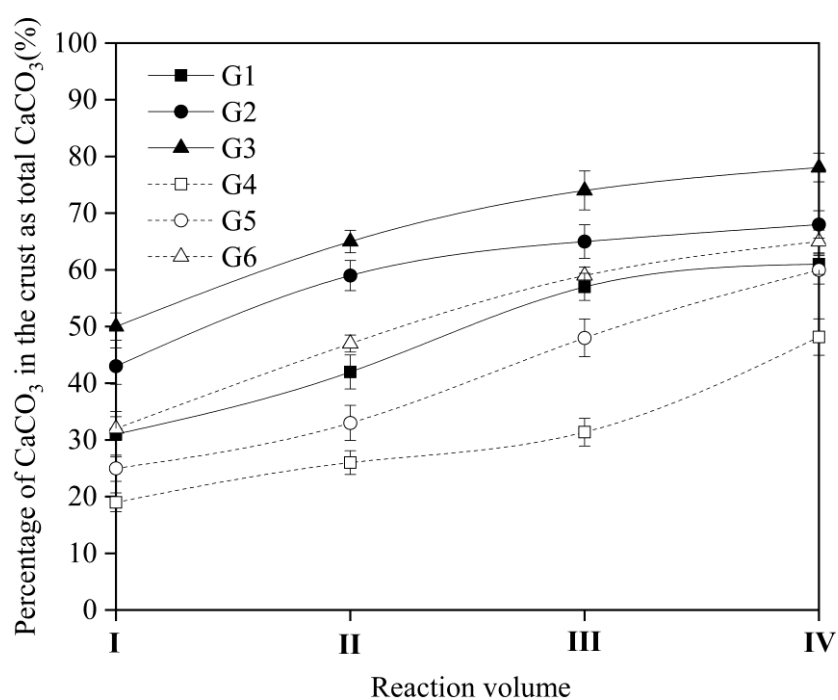


Figure 5. Percentage of CaCO<sub>3</sub> in the crust as total CaCO<sub>3</sub>.

In addition, it can be seen from Figure 5 that under the same reaction amount, with the increase in MICP treatment times, the percentage of CaCO<sub>3</sub> content in the surface hard shell increases. This trend is consistent with experimental results of Cheng et al. [29]. They found the average calcium carbonate content could almost double from 1.9% to 3.7%, as the MICP treatment cycles increase from 3 to 7. The possible reason is that with a rise in the number of MICP treatments, the first cycle MICP treatment could generate CaCO<sub>3</sub> on the surface layer and effectively block the pores of the sample, and more bacteria and

cementation solution could be retained in the surface layer for the following treatment cycles.

### 3.2. Surface Strength Test

The penetration test is one of the strength test methods (e.g., triaxial test and shear test) used to evaluate soil strength. The pocket penetrometer was used by many scholars to assess the strength of soil surface layers [27,30]. Followed by them, the surface strength was evaluated with a pocket penetrometer. The average thickness of the treated hard crust layer of the specimens in this study was 3–7 mm, and the strength results are shown in Figure 6. The surface layer strength of the specimens could be improved using HPMC, a viscous material, and the highest surface layer strength of the specimens with HPMC added was 1.61 times the surface layer strength of the specimens without HPMC added. It can be seen from Figure 6 that the surface layer strength gradually increased with an increase in the reaction amount, but for the slight reaction amount, the surface layer strength of the specimens could be improved quickly by increasing the amount of reactants, but for the reactants larger than III, the surface layer strength of the specimens increased more slowly by increasing the bacterial solution and the cementation solution. The calcium carbonate generated by MICP treatment glued the soil particles together and improved the consolidation of the soil sample. When there were less reactants, less calcium carbonate was generated, and as the reactants increased, the calcium carbonate content also gradually increased, filling the surface pores of the specimen more to bond the soil particles. In the case of sufficient reactants, the generated calcium carbonate adhered more to the surface and did not play a role in increasing the strength of the bonded soil particles (as shown in Figure 7a,b). In Figure 6, it can also be seen that the strength of the surface layer of the specimen increased with the number of MICP treatments at the same reaction volume, a trend similar to that of the  $\text{CaCO}_3$  content (Figure 5). Furthermore, it is interesting to compare the strength data obtained from different strength test (e.g., penetration test, triaxial test, and shear test) in future MICP research.

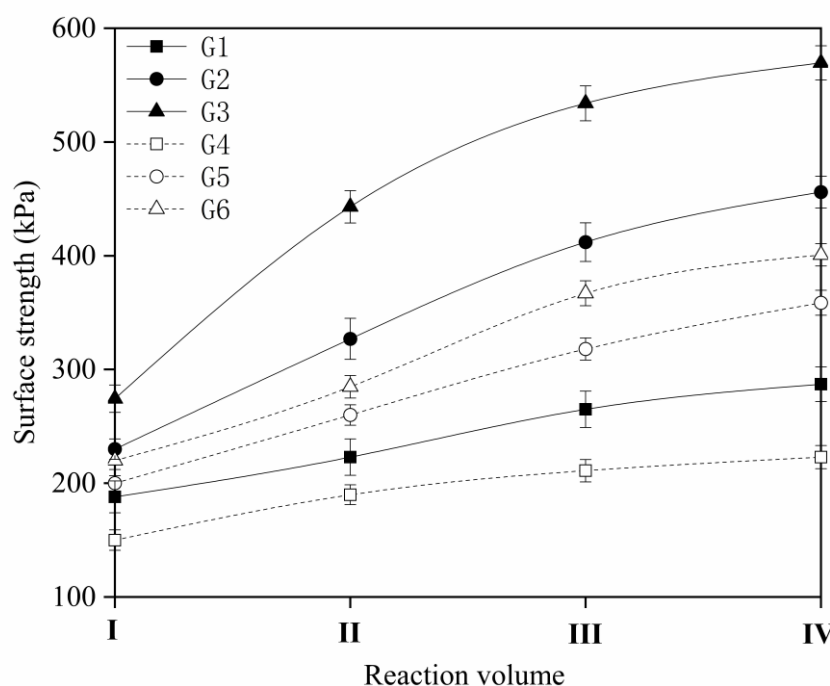


Figure 6. Surface strength of the specimens.

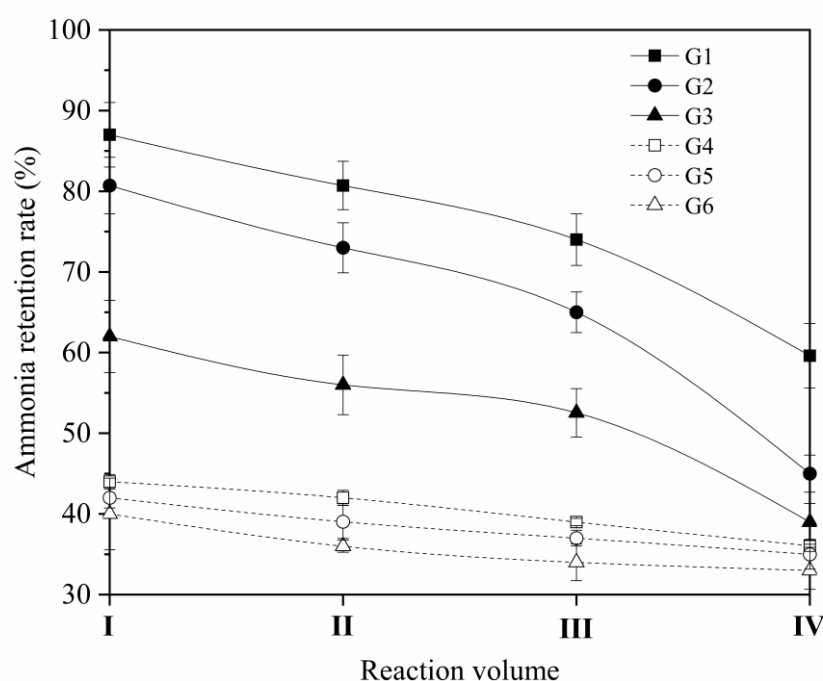




**Figure 7.** Specimen surface features: (a) with HPMC; (b) without HPMC.

### 3.3. Ammonia Retention Rate of Specimens

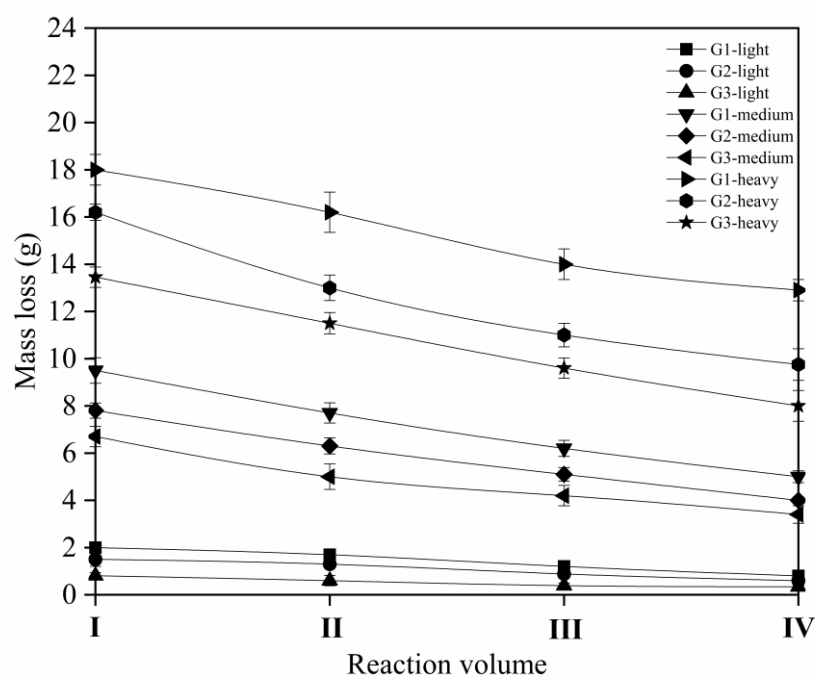
In addition to the precipitation of  $\text{CaCO}_3$  during the MICP reaction, ammonia was also released, which has a negative impact on air pollution, so it is important to determine the quality of ammonia. The concentration of ammonium ( $\text{NH}_4^+$ ) was measured and the ammonium retention rate was calculated based on the concentration of ammonium ( $\text{NH}_4^+$ ) (Figure 8). It can be seen that by simply conducting MICP without the addition of HPMC component, the specimen could retain less ammonia, due to the fact that the soil also has a certain adsorption capacity. The maximum amount of ammonia adsorbed was only 44%, while when this component of HPMC was added to it, the amount of ammonia adsorbed reached up to 87%. Furthermore, as the number of treatments increased, the ammonia retention rate gradually decreased, as shown in Figure 8. When the reactants were sprayed several times for MICP treatment, among them, when the first spray was made, a thinner crust was formed in the surface reaction, so that more reactions of the reactants could be exposed to air in the subsequent treatment, and the ammonia retention rate of the specimens could certainly be reduced. The concentration of ammonium ( $\text{NH}_4^+$ ) in the specimens after MICP treatment was measured by Cheng et al. [29], and the concentration of ammonium ( $\text{NH}_4^+$ ) in the specimens after 7 treatments was 14 g/L in their study. They also found that the concentration of ammonium ( $\text{NH}_4^+$ ) in the specimens increased with the number of MICP treatments.



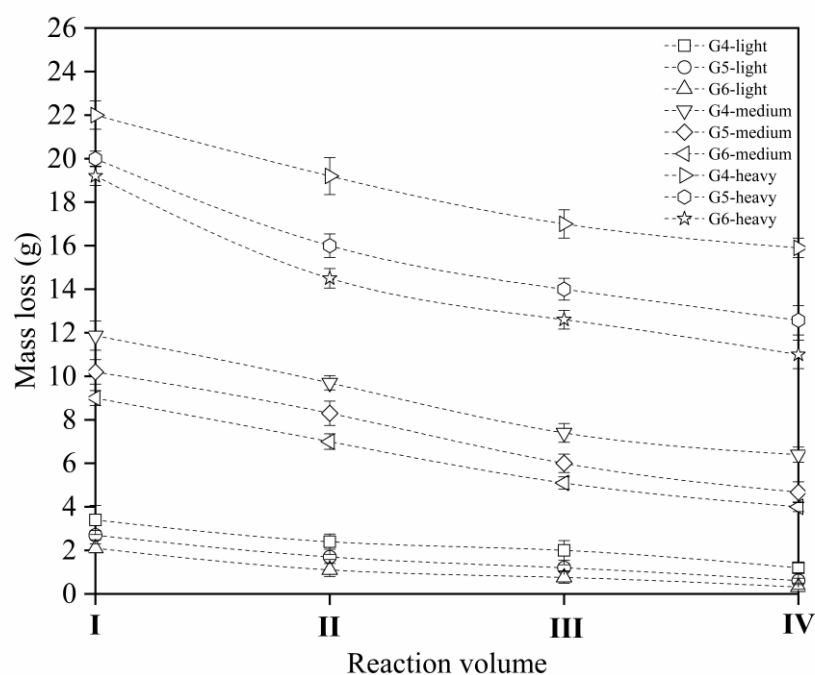
**Figure 8.** Ammonia retention rate.

### 3.4. Simulated Rainfall Test

By simulating three rainfall levels, i.e., large, medium, and light, the masses of the specimens lost by rainfall under these conditions were collected separately. The rainfall losses for each test group are shown in Figures 9 and 10. The loss of specimens treated with HPMC for MICP (Figure 9) was less than that of specimens treated without HPMC for MICP (Figure 10) under the three rainfall levels. In particular, under heavy rainfall levels, the rainfall loss with HPMC and three MICP treatments was about 30% less than that without HPMC under the same conditions. By adding HPMC as a component, the viscosity of the reactants was increased so that more  $\text{CaCO}_3$  was produced in the surface layer of the specimens, and because of its excellent solubility in cold water, the combined effect was to improve the water resistance of the surface layer. Moreover, it can be seen from the Figure that the rain erosion resistance of the surface layer of the specimen could be effectively improved after several treatments, and more calcium carbonate was generated in the surface layer after several treatments (Figure 5), making the surface layer of the specimen more compact and steady in the structure and enhancing its rain erosion resistance.



**Figure 9.** Mass loss under rainfall conditions when HPMC was added.

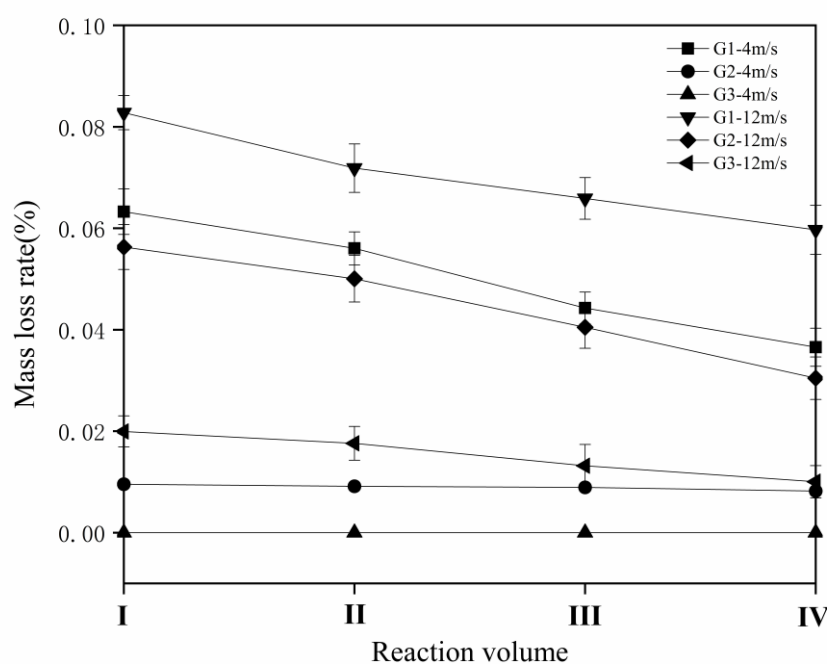


**Figure 10.** Mass loss under rainfall conditions when HPMC was not added.

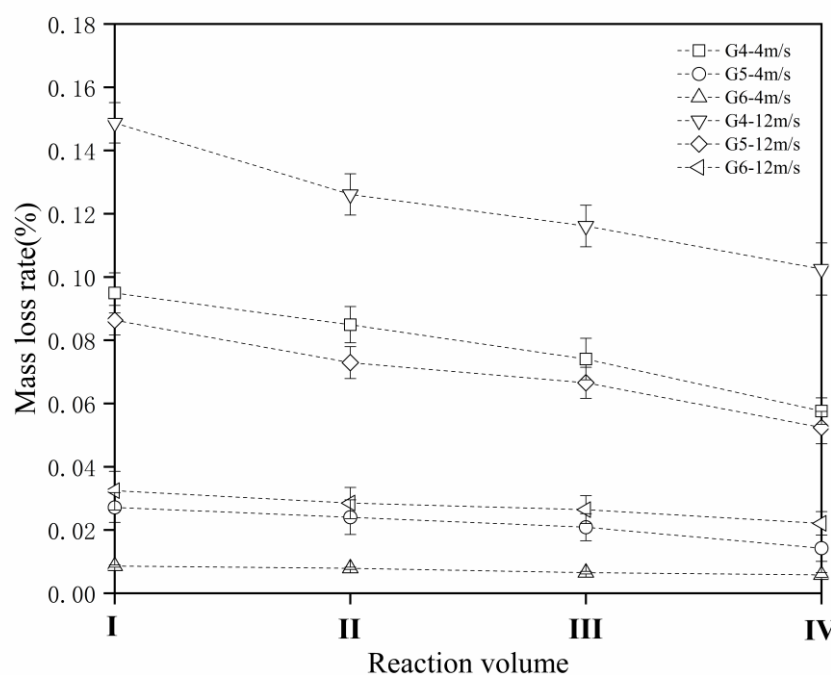
### 3.5. Wind Erosion Test

Wind erosion resistance tests were performed on MICP-treated and MICP combined with HPMC-treated specimens at wind speeds of 4 m/s and 12 m/s, respectively. The mass loss of the specimens before and after the wind erosion resistance test was measured, and the mass loss rate of the specimens was calculated for different amounts of MICP solution and different times of treatments, and the results are shown in Figures 11 and 12. The mass-loss rate of the specimens treated with HPMC for MICP (Figure 11) was lower than

that of the specimens treated with MICP without HPMC (Figure 12) at both wind speeds. When the wind speed was 12 m/s, the maximum mass loss rate of the specimens treated with HPMC for MICP was 0.828%, while the maximum mass loss rate of the specimens treated with MICP was 0.1487%. Devrani et al. [31] found that the mass loss of the MICP-treated specimens was 1.02% at a wind speed of approximately 12 m/s when the urea concentration was 1 M. The addition of HPMC, a viscous substance, increased the viscosity of the MICP solution, which allowed more calcium carbonate to be produced in the surface layer of the specimens and improved the resistance of the surface layer to wind erosion. It can also be seen from Figures 10 and 11 that the mass loss rate of the specimen surface layer gradually decreased with an increase in the number of MICP treatment, a trend consistent with the results of Wang et al. [32].



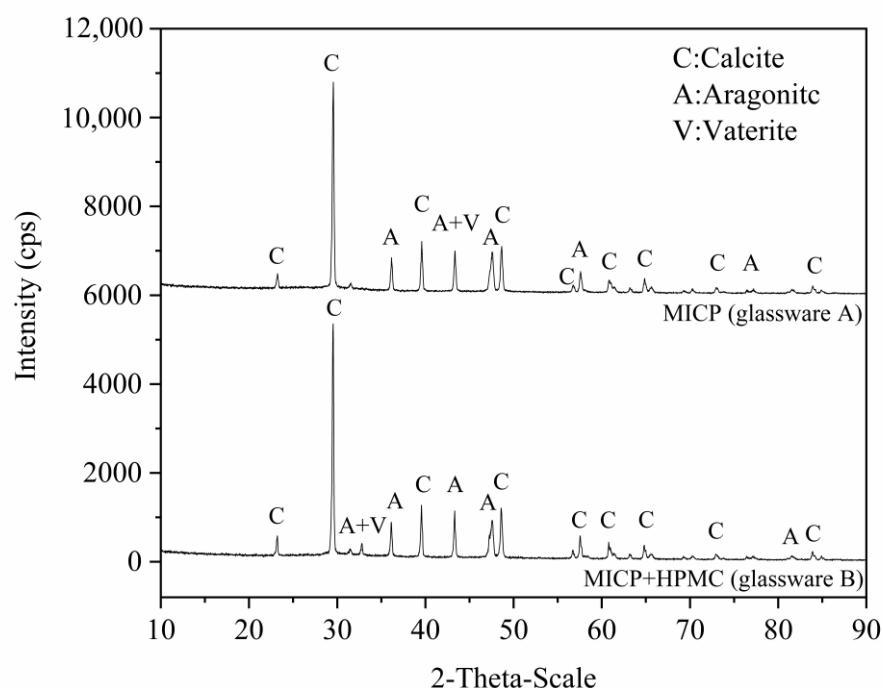
**Figure 11.** Mass loss rate when adding HPMC.



**Figure 12.** Mass loss rate when HPMC was not added.

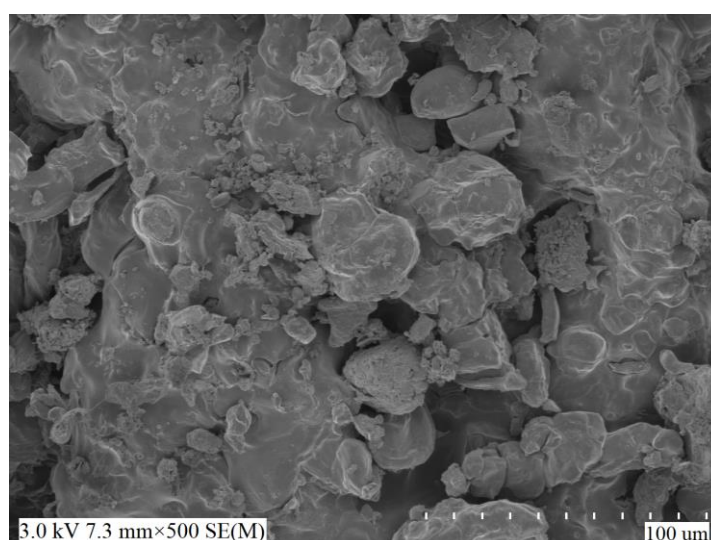
### 3.6. SEM/XRD

To investigate whether the addition of HPMC changed the type of  $\text{CaCO}_3$  crystals produced by the MICP treatment, the MICP reaction was first carried out in sterile glassware A with the addition of bacterial solution and cementation solution. Then, the same amounts of bacterial solution, cementation solution, and HPMC (viscosity of 25 mpa.s) were added in sterile glassware B for the reaction. The products in glassware A and glassware B were washed, filtered, and dried to obtain calcium carbonate. The calcium carbonate in Glass Dish A and Glass Dish B were also analyzed by XRD, as shown in Figure 13. Three types of calcium carbonate crystals were identified in these two samples, including calcite, sphalerite, and aragonite. In sample A, the  $\text{CaCO}_3$  precipitate consisted of 91.6% calcite, 6.3% aragonite, and 2.1% vaterite (Figure 13). Sample B was identified as consisting of 89% calcite, 9.6% aragonite, and 1.4% vaterite (Figure 13). This indicates that the primary  $\text{CaCO}_3$  crystal type of both samples was calcite. HPMC did not change the predominant crystal type of  $\text{CaCO}_3$  generated by the MICP treatment. In this study,  $\text{CaCl}_2$  was used as the calcium source, and the primary crystal type of  $\text{CaCO}_3$  obtained from MICP treatment was calcite, which is also consistent with the conclusion obtained from the study by Zhang et al. [33]. Moreover, the addition of HPMC slightly increased the aragonite content, and it is interesting to investigate if, in time, the addition of HPMC can increase the formation of aragonite and evaluate aragonite's behavior in long-term conditions in future MICP studies.



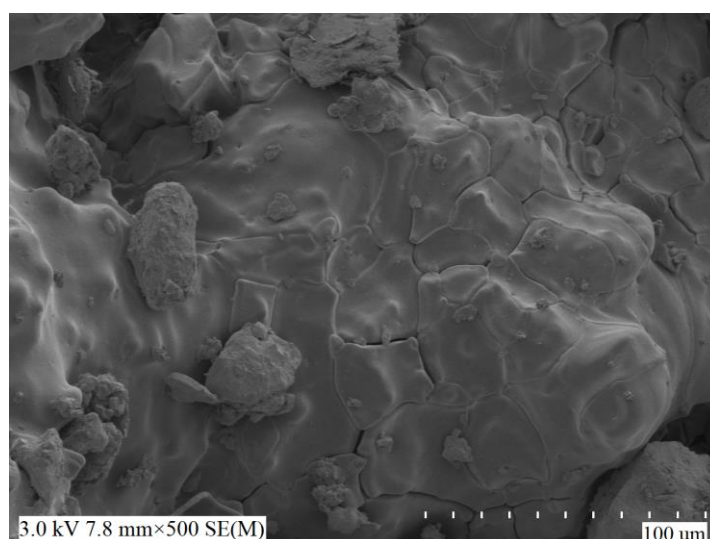
**Figure 13.** X-ray diffraction results.

SEM tests were performed on the MICP-treated specimens in G3 (reactant III) and G6 (reactant III), and the SEM images clearly showed the structural differences between the two specimens with and without the addition of HPMC for the MICP technique. Compared with the specimen without HPMC (Figure 14), the surface layer of the specimen treated with HPMC was more compact, and the soil particles and the pores between the soil particles were filled with more induced calcium carbonate crystals (Figure 15). HPMC increased the viscosity of the reactants and helped to induce more calcium carbonate precipitation in the surface layer of the specimens, thus improving the surface strength of the specimens.



**Figure 14.** SEM image without HPMC addition.





**Figure 15.** SEM image when adding HPMC.

#### 4. Conclusions

This study investigated the application of MICP technology in soil surface cementation curing. The viscosity of the reaction solution was increased by adding HPMC, a cohesive material, and a series of tests, including mechanical properties, rainfall erosion resistance, wind erosion resistance, ammonia retention, XRD, and SEM, were performed on each test group using four reactant dosage gradients and three solution injection rounds. The results of the study showed that the MICP technique incorporating HPMC is effective for soil surface improvement.

1. The content of  $\text{CaCO}_3$  in the crust of the sample induced by MICP treatment increased with an increase in the number of treatments, and the addition of HPMC can effectively improve the content of  $\text{CaCO}_3$  in the crust of the sample. MICP treatment with HPMC can produce up to 1.82 times as much calcium carbonate in the crust as MICP treatment alone, but has no effect on the total calcium carbonate. In addition, the incorporation of HPMC did not significantly change the formation of primary crystal type calcite.
2. The rainfall erosion resistance of the soil surface layer was enhanced by the addition of HPMC combined with MICP technology. Rainfall losses were reduced by up to 30% in specimens treated with this method.
3. HPMC combined with MICP technology enhanced the wind erosion resistance of the surface layer. When the wind speed was 12 m/s, the maximum mass loss rate of the specimens treated with HPMC for MICP was only 0.828%.
4. The addition of HPMC and the increase in treatment cycles can effectively improve the surface strength of the sample. By adding HPMC and multiple treatments, more calcium carbonate precipitated in the surface layer of the sample. SEM images showed that more  $\text{CaCO}_3$  filled the pores between soil particles and cohesive soil particles, forming a dense structure. The surface strength of the sample treated with HPMC was 1.61 times that of those without HPMC.
5. The ammonia retention rate of MICP treated with HPMC was significantly higher than that without HPMC. The ammonia retention rate of adding HPMC was 1.98 times that of not adding HPMC, which could effectively reduce the release of ammonia gas in the process of MICP, which is of great significance to environmental protection.

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M.Y., Y.Z., Z.X., Q.W., and F.M.; supervision, W.Z., F.H., and Y.Z.; validation, W.Z., F.H., and Y.Z.; writing—original draft, W.Z. and M.Y.; writing—review and editing, M.Y. All authors have read and agreed to the published version of the manuscript.

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## References

- Naveed, M.; Duan, J.; Uddin, S. Application of microbially induced calcium carbonate precipitation with urea hydrolysis to improve the mechanical properties of soil. *Ecol. Eng.* **2020**, *153*, 105885.
- Martinez, B.C.; DeJong, J.T.; Ginn, T.R.; Montoya, B.M.; Barkouki, T.H.; Hunt, C.; Tanyu, B.; Major, D. Experimental optimization of microbial-induced carbonate precipitation for soil improvement. *J. Geotech. Geoenviron. Eng.* **2013**, *139*, 587–598.
- Cheng, L.; Shahin, M. Urease active bio-slurry: A novel soil improvement approach based on microbially induced calcite precipitation. *Can. Geotech.* **2016**, *53*, 1376–1385.
- Choi, S.G.; Wang, K.; Wen, Z.; Chu, J. Mortar crack repair using microbial induced calcite precipitation method. *Cement Concrete Comp.* **2017**, *83*, 209–221.
- Comoss, E.J.; Kelly, D.A.; Leslie, H.Z. Innovative erosion control involving the beneficial use of dredge material, indigenous vegetation and landscaping along the Lake Erie shoreline. *Ecol. Eng.* **2002**, *19*, 203–210.
- Tiano, P.; Biagiotti, L.; Mastromei, G. Bacterial bio-mediated calcite precipitation for monumental stones conservation: Methods of evaluation. *J. Microbiol. Methods* **1999**, *36*, 139–145.
- Li, M.; Cheng, X.H.; Guo, H.X. Heavy metal removal by biomineralization of urease producing bacteri-a isolated from soil. *Int. Biodeterior. Biodegrad.* **2013**, *76*, 81–85.
- Sel, I.; Ozhan, H.B.; Cibik, R. Bacteria-induced cementation process in loose sand medium. *Mar. Georesources Geotechnol.* **2015**, *33*, 403–407.
- Brossi, D.L.; Julia, M. Soil-derived microbial consortia enriched with different plant biomass reveal distinct players acting in lignocellulose degradation. *Microb. Ecol.* **2016**, *71*, 616–627.
- Cheng, L.; Cord-Ruwisch, R. In situ soil cementation with ureolytic bacteria by surface percolation. *Ecol. Eng.* **2012**, *42*, 64–72.
- Van Paassen, L.A.; Ghose, R.; van der Linden, T.J.M. Quantifying biomediated ground improvement by ureolysis: Large-scale biogrout experiment. *J. Geotech. Geoenviron. Eng.* **2010**, *136*, 1721–1728.
- Stabnikov, V.; Naeimi, M.; Ivanov, V. Formation of water-impermeable crust on sand surface using biocement. *Cem. Concr. Res.* **2011**, *41*, 1143–1149.
- Xiao, Y.; Ma, G.; Wu, H.; Lu, H.; Zaman, M. Rainfall-induced erosion of biocemented graded slopes. *Int. J. Geomech.* **2022**, *22*, 04021256.
- Liu, B.; Xie, Y.H.; Tang, C.S. Bio-mediated method for improving surface erosion resistance of clayey soils. *Eng. Geol.* **2021**, *293*, 106295.
- Zomorodiana, S.M.A.; Hamideh, G.; Brendan, C.O. Stabilisation of crustal sand layer using biocementation technique for wind erosion control. *Aeolian Res.* **2019**, *40*, 34–41.
- Wang, X.; Tao, J. Polymer-modified microbially induced carbonate precipitation for one-shot targeted and localized soil improvement. *Acta Geotech.* **2018**, *14*, 657–671.
- Salifu, E.; MacLachlan, E.; Iyer, K.R.; Knapp, C.W.; Tarantino, A. Application of microbially induced calcite precipitation in erosion mitigation and stabilisation of sandy soil foreshore slopes: A preliminary investigation. *Eng. Geol.* **2016**, *201*, 96–105.
- Shanahan, C.; Montoya, B.M. Erosion reduction of coastal sands using microbial induced calcite precipitation. In Proceedings of the Geo-Chicago 2016, Chicago, IL, USA, 14–18 August 2016.
- Wang, J.Y.; Snoeck, D.; Vlierbergh, S.V. Application of hydrogel encapsulated carbonate precipitating bacteria for approaching a realistic self-healing in concrete *Constr. Build. Mater.* **2014**, *68*, 110–119.
- Hamdan, N.Z.; Zhao, M.; Mujica, E.; Kavazanjian, E., Jr.; He, X. Hydrogel-assisted enzyme-induced carbonate mineral precipitation. *J. Mater. Civ. Eng.* **2016**, *28*, 04016089.
- Sun, X.; Miao, L.; Wang, H. Enhanced rainfall erosion durability of enzymatically induced carbonate precipitation for dust control. *Sci. Total Environ.* **2021**, *791*, 148369.



22. Wang, X.; Tao, J.; Bao, R. Surficial Soil Stabilization against Water-Induced Erosion Using Polymer-Modified Microbially Induced Carbonate Precipitation. *J. Mater. Civ. Eng.* **2018**, *30*, 4018267.
23. Ty, A.; Ren, H.A.; Uo, A. Biomimetic mineralization of the carbonates regulated by using hydroxypropyl methylcellulose macromolecules as organic templates. *J. Cryst. Growth* **2019**, *508*, 72–81.
24. Li, M.D.; Wen, K.J.; Li, Y.; Zhu, L.P. Impact of oxygen availability on microbially induced calcite precipitation (MICP) treatment. *Geomicrobiol. J.* **2017**, *35*, 15–22.
25. Whiffin, V.S.; van Paassen, L.; Harkes, M.P. Microbial Carbonate Precipitation as a Soil Improvement Technique. *Geomicrobiol. J.* **2007**, *24*, 417–423.
26. Cheng, L.; Cord-Ruwisch, R.; Shahin, M.A. Cementation of Sand Soil by Microbially Induced Calcite Precipitation at Various Degrees of Saturation. *Can. Geotech. J.* **2013**, *50*, 81–90.
27. Almajed, A.; Lemboye, K.; Arab, M.G. Mitigating wind erosion of sand using biopolymer-assisted EICP technique. *Soils Found.* **2020**, *60*, 356–371.
28. Choi, S.G.; Park, S.S.; Wu, S.; Chu, J. Methods for calcium carbonate content measurement of biocemented soils. *J. Mater. Civ. Eng.* **2017**, *29*, 06017015.
29. Cheng, Y.; Tang, C.; Pan, X.H.; Liu, B.; Xie, Y.H.; Cheng, Q.; Shi, B. Application of microbial induced carbonate precipitation for loess surface erosion control. *Eng. Geol.* **2021**, *294*, 106387.
30. Chen, R. Bio Stabilization for Geopolymer Enhancement and Mine Tailings Dust Control. Ph.D. Thesis, The University of Arizona, Tucson, AZ, USA, 2014.
31. Devrani, R.; Dubey, A.A.; Ravi, K.; Sahoo, L. Applications of bio-cementation and bio-polymerization for aeolian erosion control. *J. Arid. Environ.* **2021**, *187*, 104433.
32. Wang, Z.Y.; Zhang, N.; Lin, F.; Ding, J.H.; Yang, H.M. Experimental Study on Wind Erosion Resistance and Strength of Sands Treated with Microbial-Induced Calcium Carbonate Precipitation. *Adv. Mater. Sci. Eng.* **2018**, *2018*, 3463298.
33. Zhang, Y.; Guo, H.X.; Cheng, X.H. Role of calcium sources in the strength and microstructure of microbial mortar. *Constr. Build. Mater.* **2015**, *77*, 160–167.