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Effects of microbially induced calcite precipitation (MICP) on the soil-concrete interface behavior

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ABSTRACT

Improving the shear strength at the soil-concrete interface can enhance the capacity of structures in various applications, such as horizontal sliding of footings, retaining walls, side friction capacity of pile foundations, and seismic design of tunnel linings. Choosing a sustainable, economical, and environmentally friendly approach is necessary when it comes to engineering practices. To achieve such an approach, the effectiveness of microbially induced calcite precipitation (MICP) in improving the soil-concrete interfacial shear strength was investigated in this study. Soil samples in contact with concrete were treated using the two major MICP pathways, i.e., ureolysis and denitrification. The required microorganisms for both pathways were cultivated from the activated sludge obtained from a wastewater treatment facility. MICP treatment can potentially improve the interface properties through different mechanisms, i.e., bonding the soil particles to the concrete, changing the roughness of the soil particles, and filling the pores between the soil and concrete. The effectiveness of the treatment would also depend on the soil gradation. Therefore, a poorly graded dune sand and a well-graded silty sand were used in this study. A series of laboratory direct shear tests were conducted on both treated and untreated samples. The results of the tests are presented and the effects of MICP pathways, soil gradation, and different contributing mechanisms are discussed.

INTRODUCTION

Concrete structures are an integral part of many construction projects. Foundations, retaining walls, piles, tunnels, culverts, and pavements are some examples where concrete is in direct contact with soil. Various parameters including concrete aggregate size and roughness, whether the concrete is cast-in-place or precast, soil particle size distribution, particle shape, cohesion, and friction angle can affect the behavior of the soil-concrete interface (Haeri et al., 2019; Xiao et al., 2022). In many areas, the in-situ soil does not meet the minimum requirements for geotechnical design and needs to be improved. Enhancing soil parameters can be achieved through various methods, such as dynamic, chemical, and biological approaches (Abdolvand & Sadeghiamirshahidi, 2024). Utilizing any of these methods can change the soil matrix and subsequently affect the behavior of the soil-structure interface.

One of the soil improvement methods that has attracted significant attention in recent years is microbially induced calcite precipitation (MICP)(O'donnell et al., 2016; Thomas O'Donnell & Kavazanjian, 2015; Van Paassen, 2009; van Paassen et al., 2010). This is because soil improvement via MICP can reduce the amount of cement and lime used in construction projects which in turn reduces the carbon footprint of the construction industry. The MICP-precipitated calcite improves the soil's mechanical properties by binding soil particles together, filling pore spaces, and increasing particle size and surface roughness. MICP can be applied through different approaches, with denitrification and ureolysis being the two most popular pathways. In MICP via the denitrification approach, denitrifying bacteria use nitrate as an electron acceptor and a carbon source as an electron donor for their respiration. This denitrifying bacteria facilitates the reduction of nitrate to nitrogen gas, and the byproducts of this reduction react with the calcium in the environment (if a calcium source is present) leading to the biomineralization of calcium carbonate (Eq. 1, 2, and 3) (Lin et al., 2021; van Paassen et al., 2010). In geotechnical engineering, MICP using the denitrification pathway is primarily known as an anaerobic method, which would limit the application of this method in improving the behavior of soil-structure (concrete) interface in shallow projects where oxic conditions prevail. However, many bacteria communities have been identified that are capable of denitrifying under oxic conditions, and their applicability in MICP soil improvement remains to be investigated.

$$CH_3COO^- + 2.6H^+ + 1.6NO_3^- \rightarrow 2CO_2 + 0.8N_2 + 2.8H_2O \qquad 1$$
$$CO_2 + H_2O \leftrightarrow HCO_3^- + H^+ \qquad 2$$
$$Ca^{2+} + HCO_3^- + 2OH^- \rightarrow CaCO_3 + H_2O \qquad 3$$

In the ureolysis pathway, bacteria with the ability to hydrolyze urea produce the urease enzyme, which catalyzes the breakdown of urea into ammonia and carbon dioxide. This reaction increases the pH of the surrounding environment and, in the presence of a calcium source, leads to the precipitation of calcium carbonate (Eq. 4 and 5) (Chen et al., 2021; DeJong et al., 2010).

$$CO(NH_2)_2 + 3H_2O \rightarrow 2NH_4^+ + HCO_3^- + OH^- \qquad 4$$
$$Ca^{2+} + HCO_3^- + OH^- \rightarrow CaCO_3 + H_2O \qquad 5$$

Although many studies have investigated the effects of these two MICP methods on the behavior of soil, very few studies focused on the effects of such methods on the soil-structure interface behavior (Li et al., 2021; Mortazavi Bak, 2024), with most concentrating primarily on soil-steel interactions.. In this study, the effectiveness of MICP treatment in improving the soil-concrete interface behavior has been studied through a series of laboratory direct shear experiments. Two different soils, a well-graded soil, and a poorly-graded soil have been used to shed some light on the possible effects of soil’s grain size distribution on the soil-concrete interface behavior improvement by MICP. Both bacterial metabolic pathways, i.e., denitrification and ureolysis, were used in this study. The denitrification MICP experiments were conducted under aerobic conditions to examine the possibility of using this pathway in shallow applications where oxic conditions are dominant. Both denitrifying and ureolytic bacteria communities used for the biocementation experiments were obtained under the non-sterile condition from a wastewater treatment facility’s activated sludge. The non-sterile enrichment method used in the study makes it easier and more cost-effective to transient from lab experiments to field applications in the future.

MATERIALS AND METHODS

Sample preparation

To investigate the effect of the MICP method on the interface between soil and concrete, samples were prepared for direct shear tests. Due to the small size direct shear box used in this study (diameter of 6.35 cm), a commercial concrete mix (Quikrete which consist of portland cement, sand, gravel, lime, limestone dust, and calcium sulfate) was sieved through a #8 sieve to prevent the presence of very coarse grains at the interface. Five kilograms of the pre-mixed concrete was mixed with 0.93 liters of distilled water, and then the mixture was poured into the plastic molds with a diameter of 6.35cm and a height of 1.8-1.9 cm (Figure 1 a). Once the concrete had dried, its height was remeasured, and any part exceeding the target height was sanded down to achieve the desired height for the direct shear test mold. After the concrete was cured, the samples were flipped in the molds, to have the smoother side as the soil-concrete interface (simulating a precast concrete-soil interface). In the next step, the soil was poured on top of the concrete disks as shown in Figure 1 b.

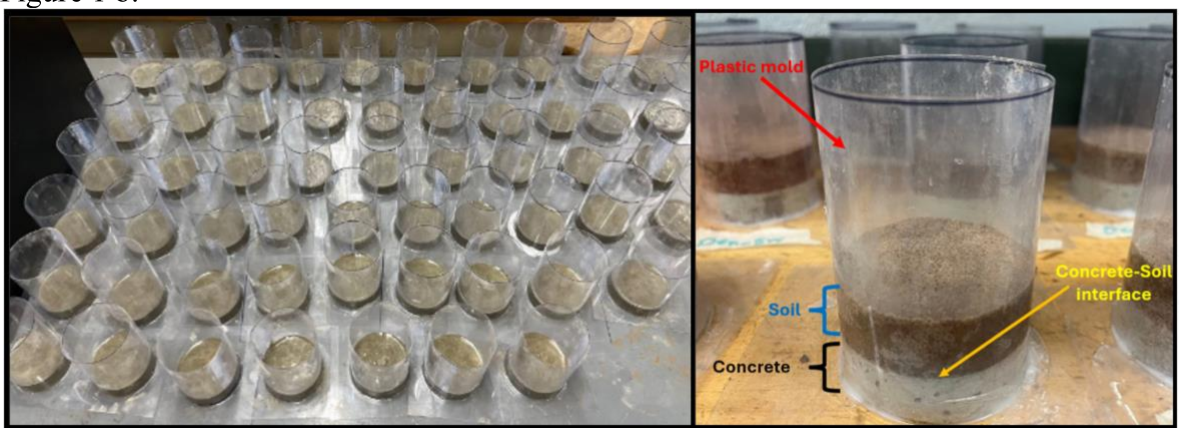

Figure 1. a) Concrete face preparation, b) Prepared soil-concrete sample for interface direct shear test

Two different soils were investigated in this study: a poorly-graded sand and a well-graded sand. The poorly graded sand used in this study was obtained from the sand dunes (dry density: 1.46 gr/cm^3) located near the Eagle River Beach in the Upper Peninsula of Michigan, USA. The well-graded sand (dry density: 1.50 gr/cm^3) was prepared in the lab by mixing glacial sand (from the Upper Peninsula of Michigan, USA) which was acid-washed to remove any calcium carbonates, a commercial poorly graded filter sand, and high plasticity clay to achieve a well-graded size distribution. The grain size distributions of both soils are shown in Figure 2. It is notable that both soils were prepared in the mold in a loose condition (poorly graded soil dry density: 1.46 gr/cm^3, well-graded soil dry density: 1.50 gr/cm^3)

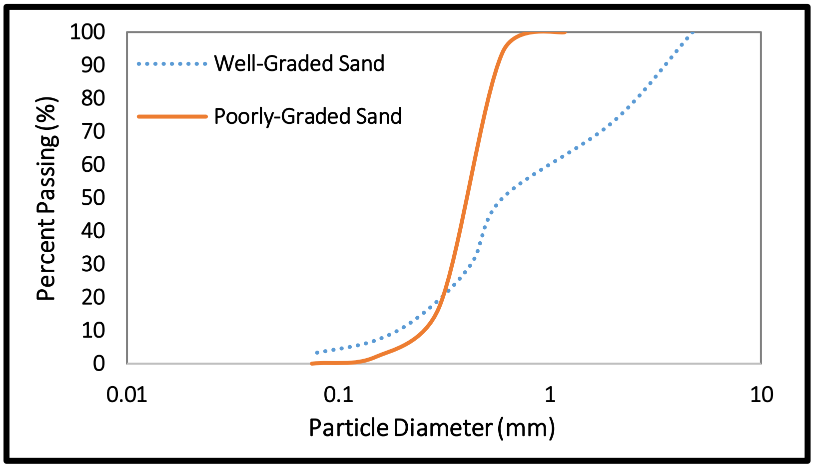

Figure 2. the grain size distributions of the soils used in this study

Inoculation and substrate preparation

Two different MICP pathways, i.e., aerobic denitrification and ureolysis, were used in this study. For both methods, the bacteria were enriched from the activated sludge obtained from a local wastewater treatment facility near Houghton, Michigan.

For the denitrification pathway, a nutrient broth including 0·003 mM ammonium sulfate ($(NH_4)_2SO_4$), 0·0024mM magnesium sulfate ($MgSO_4$), 0·006 mM monopotassium phosphate (KH2PO4), 0·014 mM dipotassium phosphate (K_2HPO_4) and 1ml/l trace element solution SL12B (EDTA-$Na_2.2H_2O$, 3000 mg/L; $FeSO_4.7H_2O$, 1100 mg/L; H_3BO_3, 300 mg/L; $CoCl_2.6H_2O$, 190 mg/L; $MnCl_2.4H_2O$, 50 mg/L; $ZnCl_2$, 42 mg/L; $NiCl_2.6H_2O$, 24 mg/L; $Na_2MoO_4.2H_2O$, 18 mg/L; $CuCl_2.2H_2O$, 2 mg/L; pH adjusted to 7.2-7.3) was used in all cultivation or treatment cycles. To cultivate denitrifying bacteria, substrates containing 60 mM calcium acetate and 50 mM calcium nitrate were added to the AS. The suspension was kept at room temperature (25±1°C) for 6 days to cultivate denitrifying microorganisms. After this 6-day inoculation, the precipitated solids were discarded and the supernatant was used as the bacterial source. The bacterial source was then mixed with a fresh substrate solution containing the nutrient broth described above and 80 mM calcium acetate and 50 mM calcium nitrate in equal proportions (Pham et al., 2018). This solution was also used to measure nitrate and nitrite content over time to validate nitrate reduction under aerobic conditions, which is one of the novelties of this paper.

The solutions for the ureolysis pathway were prepared according to Yang et al. (2020): 990 ml of culture media containing 5 g/L ammonium chloride, 20 g/L yeast extract, 0.01 g/L nickel chloride, and 10 g/L urea were added to 110 ml of activated sludge, which was sieved through a #18 sieve, and then the pH was adjusted to 10 using NaOH. The microorganism suspension was stirred well for 36 hours at room temperature (25±1°C) to cultivate ureolytic bacteria. The cultivated solution was centrifuged for 5 minutes at 5000 rpm, and the biofilms were mixed with 1100 ml of 0.9% saline solution. This bacterial solution was then mixed with a substrate solution containing 60 g/L urea and 111 g/L calcium chloride in equal volumes (Yang et al., 2020).

MICP treatment

A sponge was placed on top of the molds (containing the soil and the concrete disk) and bacterial suspensions and substrate solutions associated with each pathway were introduced through the sponge onto the soil using the gravity percolation method. The volume of substrates and bacteria suspension used was equal to one soil's pore void volume (28 ml to the poorly-graded sand and 31 ml to the well-graded soil). The sponge was used to ensure uniform distribution and prevent soil displacement.

For the aerobic denitrification MICP, at first one pore volume of substrates and bacteria suspension was introduced to the sample in the mold and the sample was left exposed to the air for a week. Considering the small size of the soil sample, it can be assumed that by keeping the sample exposed to air throughout the treatment, denitrification is happening in the presence of oxygen. Although this does not guarantee aerobic denitrification (as anoxic microenvironments can develop in the soil which could be enough for anaerobic denitrification to occur and precipitate calcite), if successful it shows that the denitrification pathway can be used for geotechnical projects where oxic condition (at macro scale) are expected. Denitrification pathways were thought to be inapplicable in such conditions. After that, one pore volume of substrates was introduced into the model every week for two weeks (a total of three denitrification treatment cycles).

For the ureolysis pathway, two sets of samples were prepared. The first set was only treated by adding one pore volume of substrates and bacteria suspension (one ureolysis MICP treatment cycle). For the second set, one pore volume of substrates and bacteria suspension was introduced to the sample in the mold and the sample was left exposed to the air for a week. After that, one pore volume of substrates was introduced into the model every week for two weeks (a total of three ureolysis MICP treatment cycles to compare with 3 cycles of denitrification MICP treatment). The ureolysis pathway only requires a couple of days for the complete calcite precipitation. In our experiments, however, because there was no drainage in the mold, an extra 7 days were required between the cycles to make sure all the water introduced in the previous cycle was evaporated before introducing the next solution.

Interface direct shear and other Laboratory experiments

Direct shear tests were conducted on the interface between soil and concrete, for both treated and untreated samples according to ASTM D3080/D3080M-1. The direct shear mold used in this study has a diameter of 6.35 cm and a total height of 4 cm. For measuring the interface behavior of untreated samples, concrete disks were taken out of the mold and placed in the bottom half of the direct shear box. To make sure that the shearing occurred at the interface between the concrete and the soils, the height of the concrete disks was designed to be exactly the same size as the height of the bottom half of the shear box. In cases where the disks were taller than expected, a lab-scale concrete grinder was used on the other side of the disks (the side that was not in contact with soil) until the appropriate height was achieved. Then top half of the shear box was placed on the bottom half and filled with soil in a loose state (with the same density used for treated samples before treatment). To develop a Mohr-Coulomb failure envelope for the interface behavior of untreated samples, nine identical samples were prepared for each soil, and direct shear tests were conducted on these samples (three tests under 69 kPa (10 psi), three tests under 138 kPa (20 psi), and three tests under 207 kPa (30 psi) normal stresses). The average of the shear stresses at failure for each normal stress was calculated and then used to develop the Mohr-Coulomb plot and the failure envelope.

The treated samples, prepared as explained before, were taken out of the mold and placed in the direct shear box. Figure 3 shows the treated samples after removal from the molds and before the direct shear tests. The direct shear tests on treated samples were conducted 14 days after the last treatment cycles. The same normal stresses used for untreated samples, i.e., 69 kPa, 138 kPa, and 207 kPa, were applied to the treated samples. Again, three identically treated samples were prepared for each normal stress and the average of the shear stresses at failure for each normal stress was used to develop the Mohr-Coulomb failure envelope of the treated samples.

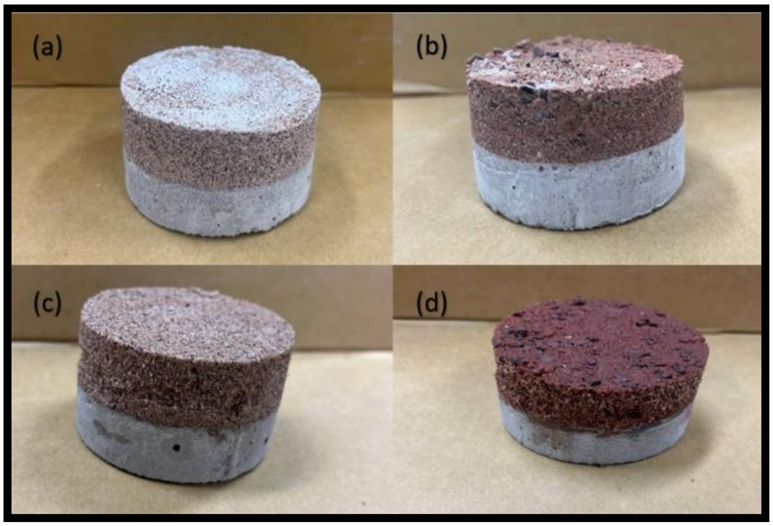

Figure 3. Treated samples a) Poorly graded-Denitrification, b) Well graded-Denitrification, c) Poorly graded-Urea hydrolysis, d) Well graded Urea hydrolysis

After the direct shear tests were conducted, the calcite content of the treated samples was measured according to ASTM D4373-22 in the top section of the soil. Soil samples from the soil-concrete interface zone were also collected and tested for calcite content. Additionally, using Ion Chromatography (IC) equipment, nitrate and nitrite concentrations were measured over four consecutive days during the inoculation stage to confirm denitrification is occurring under aerobic conditions. Scanning Electron Microscopy (SEM) was also conducted on the soil-concrete interface of treated samples after the direct shear tests to investigate the precipitated material under different conditions.

RESULTS AND DISCUSSION

Measurement of nitrate and nitrite during the aerobic inoculation for the denitrification pathway revealed that the nitrate concentration decreased from 50 mM to 28 mM, and the nitrite concentration increased from 0 mM to 5 mM in the first four days (the experiments were not continued after four days due to required instrument maintenance). This reduction in nitrate concentration, however, confirms that denitrification occurred under aerobic conditions.

Figure 4 shows the Mohr-Coulomb failure envelopes derived from direct shear tests on the treated and untreated samples. As shown in this figure, MICP treatment generally improved the soil-concrete interface strength of samples. The results of the direct shear test show that all the treated samples exhibited an increase in the friction angle, which can be attributed to the increase in particle size and the filling of pores due to calcium carbonate precipitation. In addition, the increase in the cohesion parameter confirms the formation of calcium carbonate bonds between particles, leading to an improvement in soil cohesion. After the direct shear tests were completed, the calcite contents of the treated soils were measured in the soils and at the interface and the results are summarized in table 1. Interestingly, calcite content measurements shown in Table 1 revealed that calcite precipitation in the soil-concrete interface zone was more than the average calcite content in the rest of the soil matrix in all the treated samples. This observation can be explained by two mechanisms:1) The reaction between the lime composition of the concrete and CO_2 produced due to bacteria respiration leads to $CaCO_3$ formation which increases the amount of $CaCO_3$ precipitated at the soil-concrete interface, 2) The soil-concrete interface provides a stable environment for biological and chemical reactions and the small concrete pores with rough surfaces act as excellent nucleation sites for calcite formation (Sohail et al., 2022; K. Zhang et al., 2023; Y. S. Zhang et al., 2024). In other words, the lower permeability characteristic of the concrete might act as a barrier, effectively trapping bacteria and chemicals, thereby resulting in greater precipitation at the interface region, both of which lead to higher calcite contents at the interface.

Table 1. Calcite content of treated and untreated samples

Sample	Calcite content (% by weight)	
	At the interface zone	The average amount in the soil matrix
Well graded-Untreated	0	0
Poor graded-Untreated	0	0
Well graded-Denitrification treated	2.7%	0.23%
Poor graded-Denitrification treated	1.4%	0.14%
Well graded-Urea hydrolysis treated-1 cycle	1.6%	Very small
Poor graded-Urea hydrolysis treated-1 cycle	0.26%	0.25%
Well graded-Urea hydrolysis treated-3 cycle	1.7%	1.4%
Poor graded-Urea hydrolysis treated-3 cycle	1.7%	1.4%

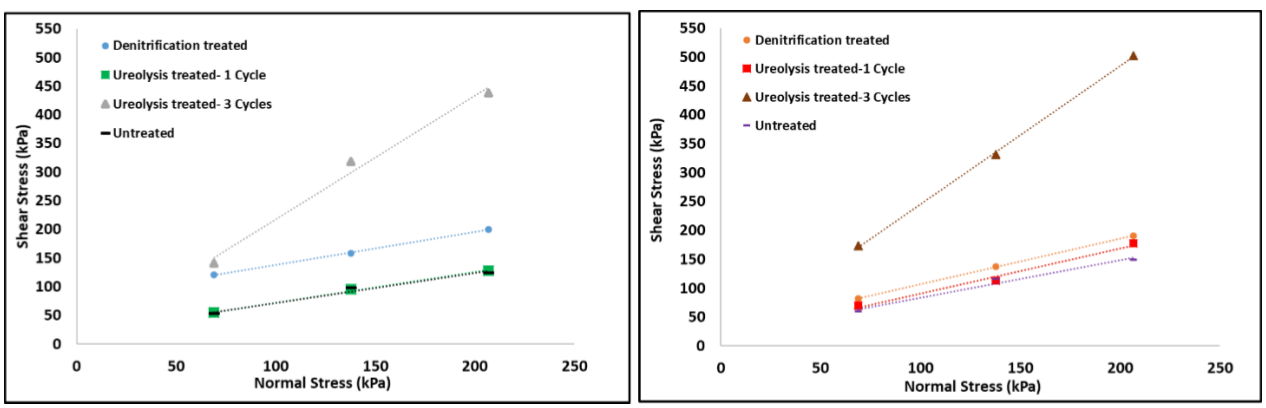

Figure 4. Mohr-Coulomb failure envelope (Left: Well-graded soil, Right: Poorly-graded soil)

A closer look at the data presented in Figure 4 and Table 1, reveals that the treatment by aerobic denitrification MICP precipitated a significant amount of calcite at the interface and some calcite was also precipitated in the rest of the soil. The calcite precipitated at the interface bonds the soil particles to the concrete surface and during the shearing phase (at least at small strains), these particles move with the concrete surface (similar to soils at the interface of cast-in-place concrete structures), and the shearing actually occurs within the soil, this observation is illustrated in Figure 5. As the calcite is also precipitated within the soil matrix, the improvement of the interface behavior seen in Figure 4 is the result of calcite precipitation and its cementation effect within the soil. The same condition is seen in the one-cycle ureolysis MICP treatment of the poorly graded soil. Again calcite is precipitated at the interface and within the soil matrix, so the improvement seen in the behavior is due to the precipitation within the soil. In the case of one-cycle ureolysis MICP treatment of the well-graded soil, almost all the calcite was precipitated at the interface, and zero (or very little) calcite was precipitated within the soil. During the shearing phase, the soil particles at the interface are cemented to the concrete and move with the concrete and the shearing failure happens within the soil (between soil particles) which in this case has not been stabilized due to zero calcite precipitation away from the interface. Therefore, the failure envelope is almost identical to the untreated soil. In the case of three-cycle ureolysis MICP treatments of both soils, a significant amount of calcite precipitated both at the interface and within the soil.Again, because of the cementation at the interface, soil particles move with the concrete during the shearing and the shearing failure happens within the soil which in this case is highly cemented (the highest amounts of calcite precipitated within the soil). This high cementation amount within the soil matrix causes the significant improvement in the behavior observed in Figure 4. It is notable that, extended treatment periods in the MICP mrthods can offer several potential benefits for soil improvement. One significant benefit of extended treatment periods is the potential for increased strength and durability of the treated material. Longer treatment times allow for more extensive precipitation of calcium carbonate, leading to improved mechanical performance and groundwater control (Botusharova et al., 2020). This can result in stronger, more stable soil structures that are better able to resist deformation and liquefaction (Montoya et al., 2012). Additionally, extended treatment periods may allow for more uniform distribution of precipitates throughout the treated volume, addressing issues of spatial variation in improvement (Dejong et al., 2014).

Figure 5 illustrates some examples of the soil-concrete interface after the direct shear tests. As can be seen in the figure, the soil particles at the interface of the treated samples are still attached to the concrete surface. This confirms that these particles moved with the concrete during the shearing phase and the shear failure actually happened within the soil (not exactly at the interface).

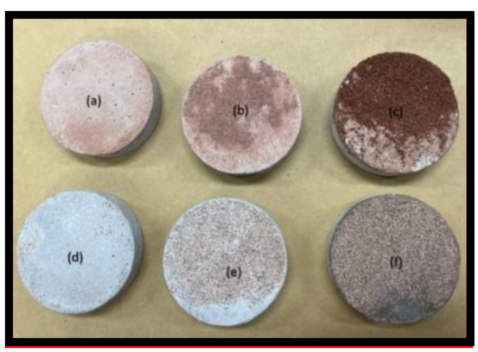

Figure 5. The interface of Soil-Concrete samples after the direct shear test: a, b, and c: well-graded (untreated (a), treated with the denitrification pathway (b), and treated with the urea hydrolysis pathway (c)); d, e, and f: poorly graded (untreated (d), treated with the denitrification pathway (e), and treated with the urea hydrolysis pathway (f)) soils after shearing

Figure 6 shows the stress-strain plot of treated and untreated soil samples. Untreated samples show ductile behavior, and the treatment changes the behavior to brittle failure. This result is in agreement with the findings of previous studies on the effects of calcite cementation on soil stress-strain behavior (Shakeri et al., 2018; Shan et al., 2022). The produced calcium carbonate acts as a cementing agent which increases the soil's shear strength and and brittleness.

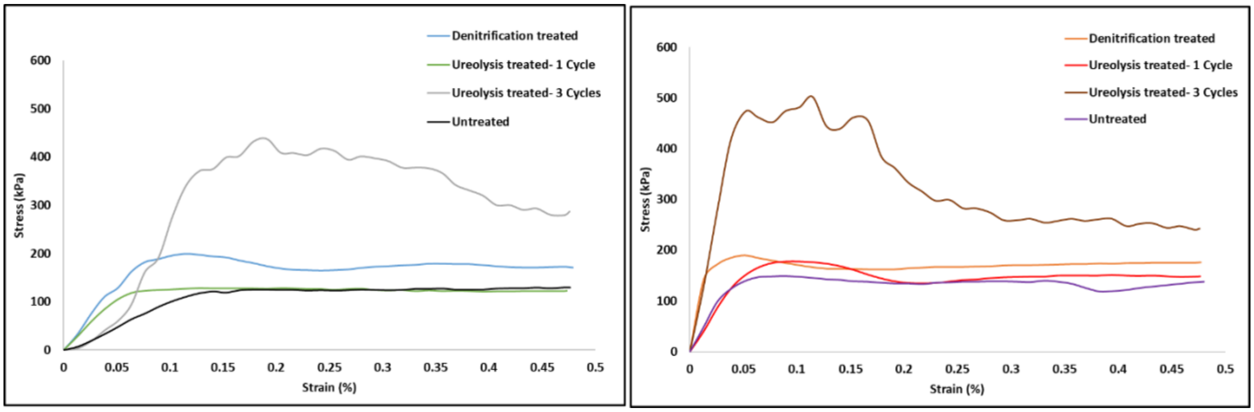

Figure 6. Example of Stress-Strain curves during the direct shear test of treated soil-concrete interface (Normal stress: 69 kPa (30 psi), Left: Well-graded soil, Right: Poorly-graded soil)

The results of SEM tests on the soil-concrete interfaces of MICP-treated samples are shown in Figure 7. Figures 7 a, b, and c show the SEM of the treated poorly graded sand with different resolutions treated via MICP denitrification. Figure 7 c shows the precipitated calcite which connected soil particles to each other and also to the concrete material. Figures 7 d, e, and f show the MICP-treated via urolysis pathway of well-graded soil-concrete interface. The clay-coated zone as well as the calcite bridge are specified and also the rhombohedral morphology of the calcite crystals can be seen in Figure 7 f. Figures 7 g, h, and i indicate poorly graded sand treated with urea hydrolysis pathway. In Figure 7 g, an EPS (Extracellular polymeric substance) band associated with the microorganism activity can be seen which can connect and keep particles together. Figure 7 j, k, and l show the MICP-treated via urolysis pathway of poorly-graded soil-concrete interface with different resulutions. Figure 7 j, sho clay-coated surface of the concrete as well as thread shape EPS and sand particles. In Figure 7 k and l, show the calcite connecting bonds.

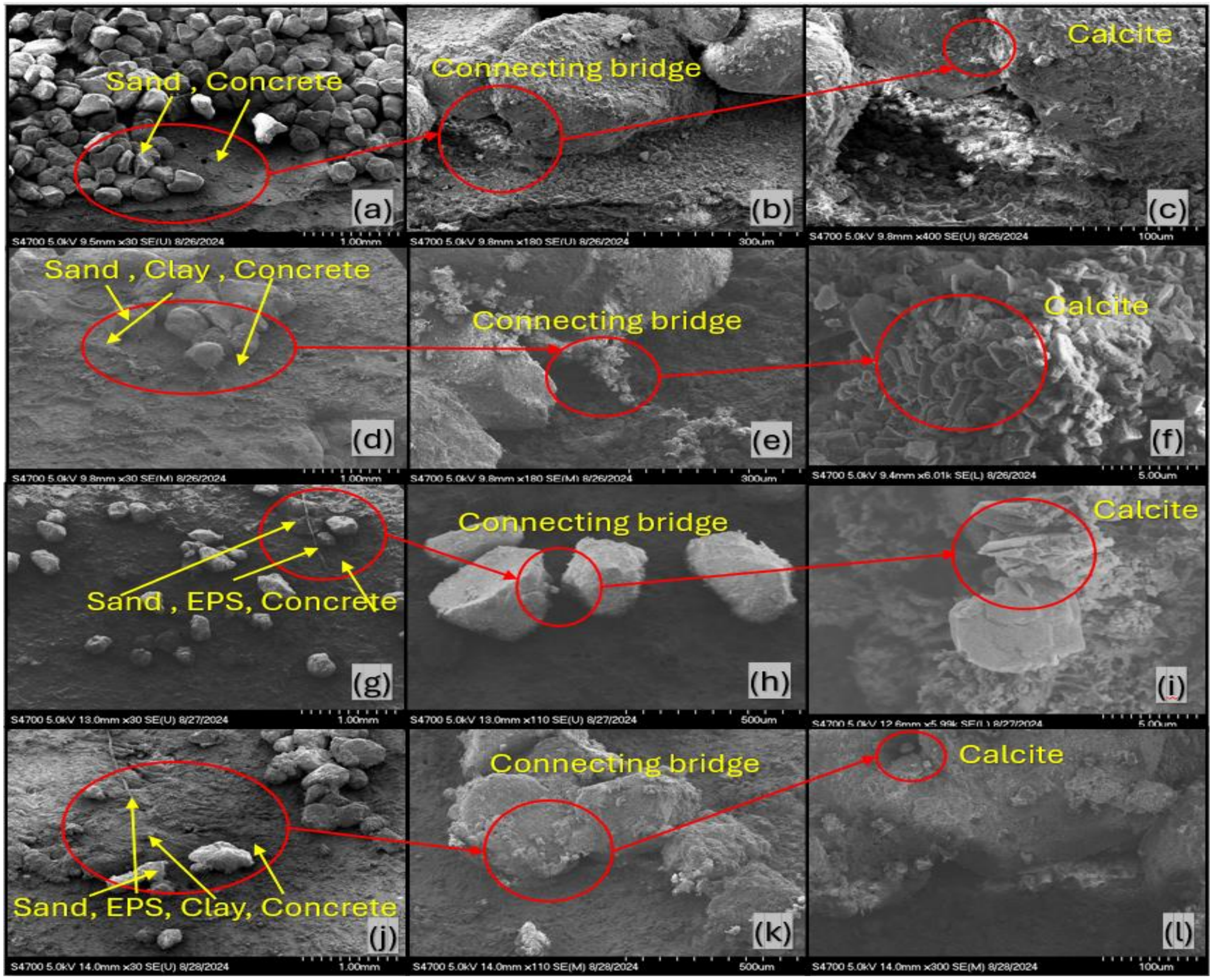

Figure 7. SEM of MICP-treated samples with different resolutions: a,b,c) Poorly-graded soil treated via denitrification pathway. d,e,f) Well-graded soil treated via denitrification pathway. g,h,i) Poorly-graded soil treated via ureolysis (1 Cycle) pathway. J,k,l) Well-graded soil treated via ureolysis pathway

CONCLUSION

This study confirmed that denitrification occurred under aerobic conditions, leading to significant calcite precipitation, particularly at the soil-concrete interface. Direct shear tests revealed that MICP treatment (with both aerobic denitrification and ureolysis pathways) can improve the soil-concrete interface behavior of both well-graded and poorly-graded soils. The amount of improvement depends on the amount of calcite precipitated in the soil matrix, not at the interface. Calcite precipitated at the interface bonds the soil particles to the concrete surface and during the shearing phase, these particles move with the concrete surface and the actual shear failure plane develops within the soil. If the calcite was precipitated at both the interface and within the soil, the cementation caused by calcite within the soil itself improves the interface behavior by increasing the shear strength on the failure plane that develops within the soil. SEM analysis confirmed the precipitation of calcite and bonding of the soil particles with concrete at the soil-concrete interface. The results also revealed that MICP treatment can change the soil-concrete interface behavior from ductile to brittle. This could have significant implications when designing geotechnical projects.

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