EVALUATION ON THE PERFORMANCE OF MICP TREATED SLOPE SOIL UNDER ACID RAIN ENVIRONMENT

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ABSTRACT

Microbial induced carbonate precipitation (MICP), through the field of bio-mediated geotechnical engineering, has gained significant momentum in research industry during recent past, which has promoted the strategy nearly to field application stage. The aim of this study is to evaluate the durability of MICP specimens under the exposure of acidic environment. The representative soils from an erosion prone slope were treated to different cementation levels on the basis of MICP. The acidic environment was enabled under different pH conditions (ranging 3.0-6.0) through two different mechanisms: (i) infiltration and (ii) immersion, both of which represent acid rainfall and water-logged environments, respectively. The influence of infiltration rate was studied under two potential rainfall intensities, and in each case, specimens were infiltrated by acid rain volume equals fifteen-year rainfall volume. The results indicate that decrease in environment pH increased the corrosion of CaCO₃, resulting considerable loss in both mass and unconfined compressive strength (UCS). However, the increased cementation level showed high durability of specimens. The results also revealed that the contact time of acid rain significantly governed the rate of corrosion, i.e. specimens subjected to lower infiltration rate showed higher loss of mass and UCS compared to that of higher rate. Moreover, the immerged specimens showed relatively a similar response of those subjected to low infiltration. Overall, this study addressed one of the prime environmental hurdles which might possibly be experienced in the field, advancing the understanding on performance of slope near-surfaces conserved by MICP.

Keywords: Microbial induced carbonate precipitation (MICP), slope near-surface stabilization, durability, acid rain, cementation level

INTRODUCTION

Microbial induced carbonate precipitation (MICP), is relatively a novel and environmental-friendly soil stabilization technique and has gained significant momentum in recent years. The MICP treatment relies on urea hydrolysis; the urease produced by supplied or enriched bacteria catalyzes the urea hydrolysis, resulting the production of ammonium and carbonate ions (Eq. 1). The produced carbonate ions then react to precipitate calcium carbonate in the presence of calcium resources (Eq. 2) [1], [2]. This precipitated calcium carbonate functions as bio-cement, bonding the adjacent soil particles preferentially at particle-particle contacts and enhancing the aggregate stability and strength characteristics.

$$CO(NH_2)_2 + 2H_2O \rightarrow 2NH_4^+ + CO_3^{2-} \qquad (1)$$
$$CO_3^{2-} + Ca^{2+} \rightarrow CaCO_3 \text{ (s)} \qquad (2)$$
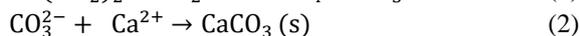
Stabilization of slope near-surface against aggregate instability is one of the serious concerns in the field of Geotechnical Engineering. The widely used traditional methods are primarily cement-based and/or bitumen-based, and they would appear to be ill-advised from a sustainability perspective. In recent years, MICP is often one of the scientists' and/or engineers' proposal as a new sustainable alternative [3], [4]. The researches have already been initiated and demonstrated the viability of the technique in various standpoints, promoting the strategy nearly to field application stage. Accordingly, Salifu et al. [5] showed that the MICP could improve the stability of slope aggregates under repeated tidal currents, concluding that relatively a shallow treatment would be sufficient for coastal protection. The use of native bacteria was another appeal from the sustainable perspective, and that was proposed and demonstrated for stabilizing expressway slope soils [6], [7]. In another work, Gowthaman et al. [8] addressed the effect of particle size distribution on MICP responses, suggesting that presence of silt in small quantity tends to filter more bacteria at near-surface zone and results stable slope surfaces.

Despite of the considerable works, the studies addressing the longevity of MICP treatment under climatic factors are limited. Regarding the rainfall-induced impairments, treating the slope by 1 mol/L cementation resources was found to have better durability responses against erosion [9]. Considering the effects from seasonal frosts (subarctic regions), Gowthaman et al. [10] found that higher the precipitated content of calcium carbonate the stronger the inter-particle connections, leading to high aggregate stability and durability against freeze-thaw

actions. Provided alkaline conditions persist, the longevity of MICP treated soils can be expected to be stable for more than fifty years [11]. On the other hand, $CaCO_3$ material is chemically susceptible to acidic environment; therefore, longevity and durability of MICP treatment under the exposure of acid rainfall is often a question to be explored, and that remains as a hurdle for applications to the fields experiencing acid rainfalls. Therefore, the objective of this paper is to evaluate the durability of MICP treated soils under the exposure of acid rainfall environment.

MATERIALS AND METHODS

Soil and preparation

An erosion-prone expressway slope located in Onuma (Hokkaido, Japan) was selected for the current study. The slope soil can be classified as poorly graded (SP) fine sand according to the Unified Soil Classification System (USCS). Results from X-Ray fluorescence (XRF) analysis revealed that the silica is the dominant soil mineral, and the organic content was found to be negligible. Table 1 summarizes the fundamental geotechnical properties of slope soil.

Table 1 Fundamental characteristics of slope soil

Property	pH	D_{50}	C_u	C_c	G_s
Values	6.997	0.23	2.5	0.8	2.71

Note: D_{50} is the mean particle diameter (i.e., grain diameter at 50%); C_u is the coefficient of uniformity; C_c is the coefficient of curvature; G_s is the specific gravity.

The soil columns (30 mm in diameter; 50 mm in height) were prepared by tamping in three layers to the average density of around 1.6 ± 0.1 g/cm^3, followed by the MICP treatment.

Bacteria and MICP treatment

Lysinibacillus xylanilyticus were the bacteria used herein to induce urea hydrolysis. The above ureolytic bacteria were isolated from the same slope soil; the detail on isolation process and characterization can be found in previous works [6], [8]. For the cultivation of bacteria, ammonium-yeast extract media (NH$_4$-YE, ATCC 1376) was used, which consisted of 20 g/L of yeast extract, 15.7 g/L of tris buffer and 10 g/L of ammonium sulfate. The media was inoculated with the bacteria pre-culture and subjected to shaking incubation at 25°C and 160 rpm. After 48-72 hours of growth, the bacteria culture was used for the MICP treatment (the optical density OD$_{600}$ was around 4.0).

During the MICP treatment, a two-phase surface percolation method [12] was incorporated: initial supply of bacteria culture, followed by multiple supplies of cementation solution. Between the two phases, a period of 2-3 hours was allowed for bacteria cells to be attached with the soil particles [13]. All the solutions were supplied at the top of the columns at relatively a constant flow rate of 4 mL/min. Concentration of cementation solution used for the treatment was 1 mol/L, and which was similar to many previous works [14], [15]. The cementation media consisted of 110.9 g/L calcium chloride, 60 g/L urea and 6 g/L of nutrient broth.

In this study, three different levels of cementation (i.e., % of $CaCO_3$) were considered (C-1, C-2 and C-3). Different durations of MICP treatments were performed to achieve different levels of cementation. For C-1, around 7 number of cementation injections were performed. Around 10 number of injections were performed for C-2, and 14 injections for C-3. In another terms, the C-1, C-2 and C-3 levels can be demarcated as precipitated $CaCO_3$ content ranging between 12-14%, 16-18% and 21-23%, respectively. Measurement of $CaCO_3$ content was determined using the acid reaction method [8].

Acid rain (AR) simulation test

Accelerated test was applied in acid rain (AR) simulation. In real situations, AR could potentially be in contact with MICP treated slope near-surface at two possible conditions: (i) during infiltration and/ or (ii) submerged, and both conditions were investigated in this study. In order to reproduce the process of AR interaction, an infiltration setup was arranged as shown in Fig. 1. Effects of various parameters such as level of cementation (C-1, C-2 and C-3), pH of AR solution (3.0, 4.5 and 6.0) and intensity of AR (20 and 100 mm/h) were evaluated. The intensity of AR was controlled herein by the rate of delivery of the AR solution at specimen top as shown in Fig. 1.
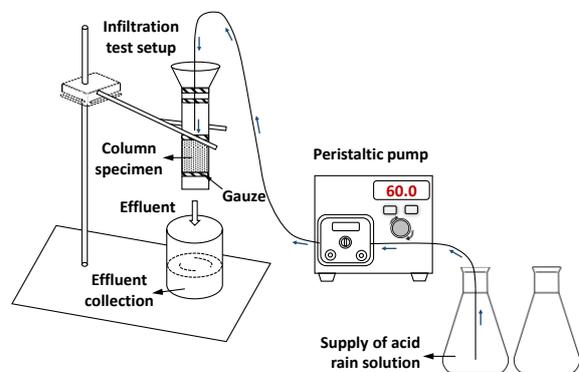
Fig. 1   Experimental set up of the AR infiltration

AR solution was prepared by dropping a certain volume of 1 mol/L nitric acid (HNO_3) and sulfuric acid (H_2SO_4) to deionized distilled water, and the pH levels were adjusted to 3.0, 4.5 and 6.0. The infiltration quantity of AR (Q_{inf}) was estimated based on the average annual precipitation in study location (Onuma, Hokkaido), as per the given equation (Eq. 3). Similar to the previous works, it was assumed that one-third of the total precipitation infiltrates in to the slope and two-third would be surface-runoff [16], [17].

$$Q_{inf} = {}^1\!/_3\ P_{avg}\ A \qquad (3)$$

where, the P_{avg} stands for the annual average precipitation of study location (1210 mm), and A is the cross-sectional area of the specimen (7.06 cm^2). Accordingly, specimens were infiltrated by AR solution equivalent to the precipitation of 15 years. The AR solution was delivered to the specimens at two rates, and the rates were chosen on the basis of typical high and low rain fall intensities (around 20 and 100 mm/h, respectively).

For the submerged condition, specimens were soaked completely into corrosion-resisting cylindrical molds containing AR solution. As the pH values of the AR solutions were changing due to the consumption of protons (H^+) by the reaction with $CaCO_3$, the solution was replaced every 24 hours by newly prepared AR (to the total volume equivalent to the precipitation of 15 years). At the same time, the concentration of leached calcium ions was measured in the reacted solution. In addition, with the different immersion times, the weight loss was measured. Finally, all the treated specimens were oven-dried for 48 hours, followed by the estimation of the UCS. The UCS of the treated specimens were estimated by needle penetration tests in accordance with JGS 3431-2012 [18].
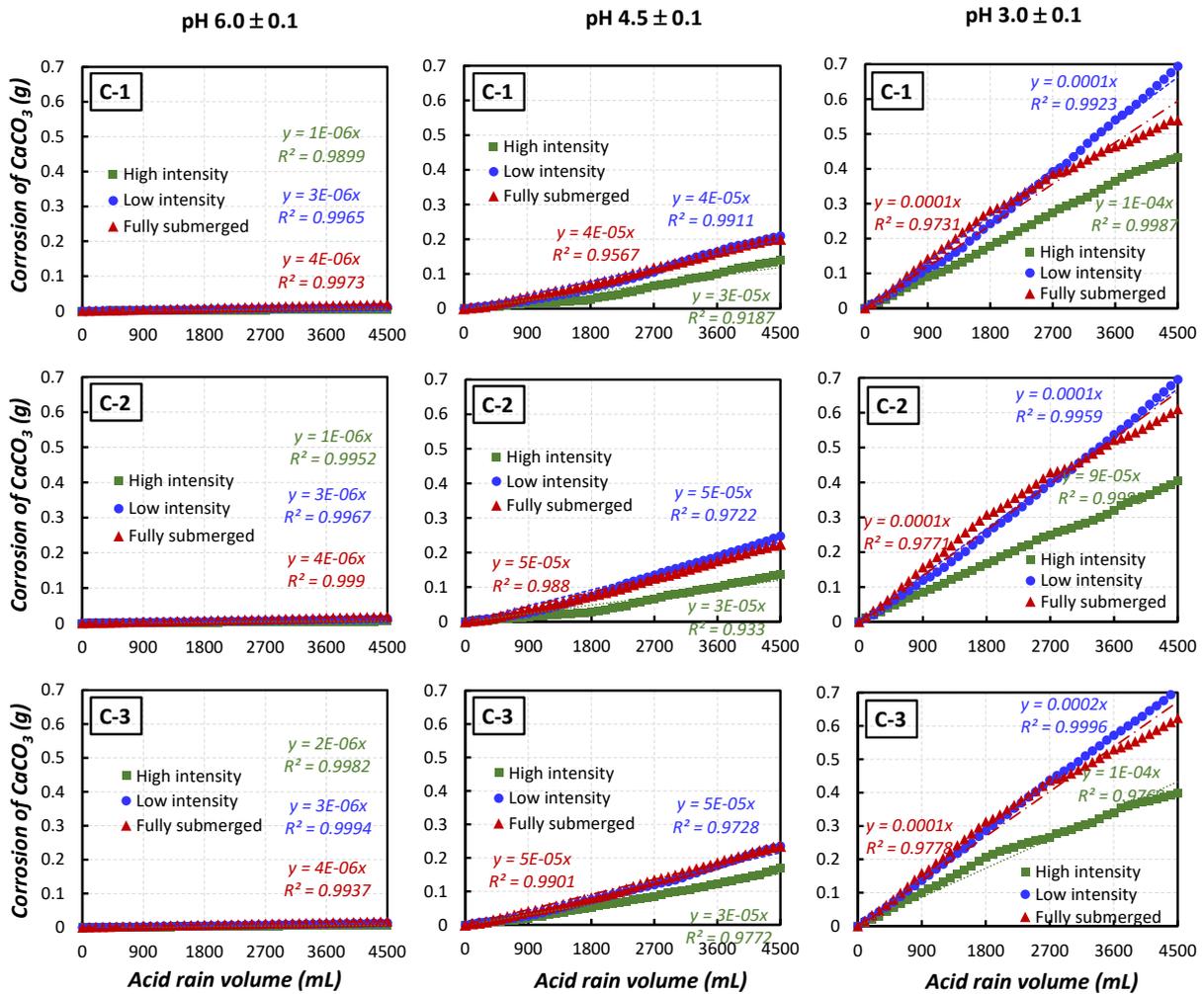
Fig. 2   The corrosion of calcium carbonate during the AR simulation test
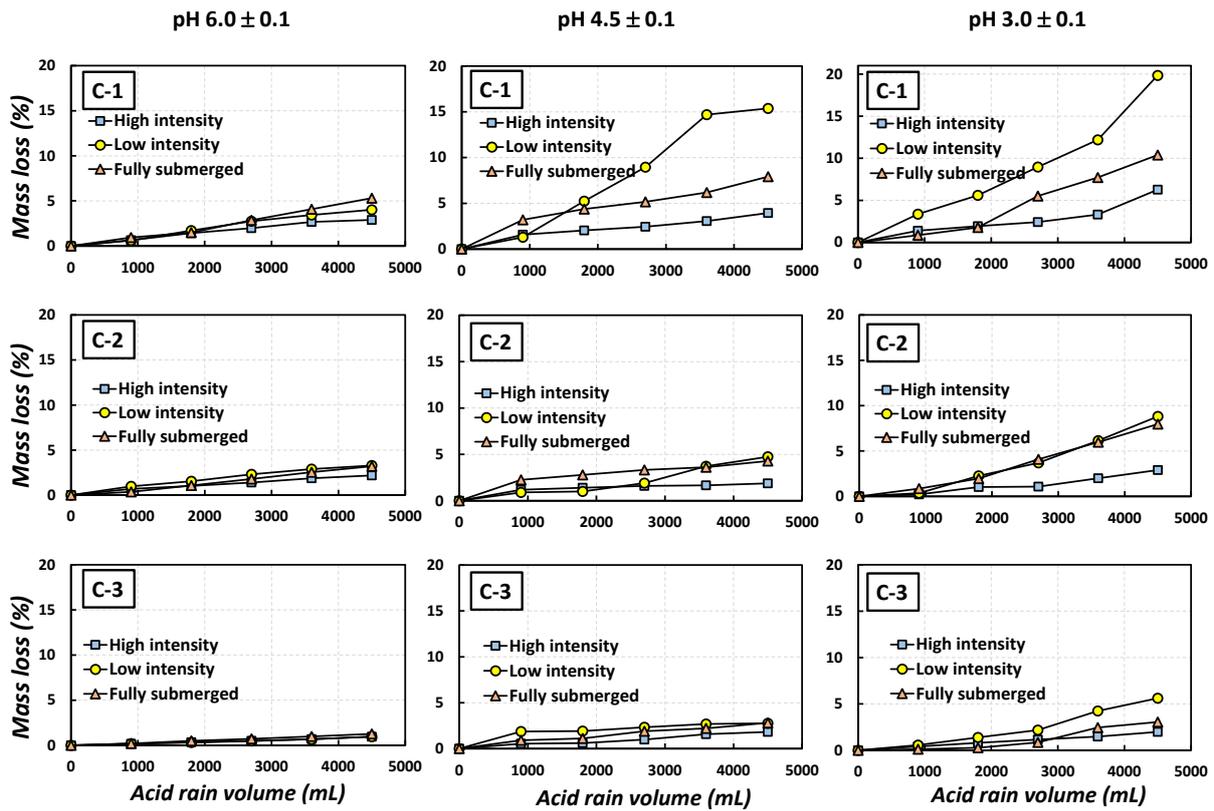
Fig. 3 Mass loss of the specimens subjected to AR

RESULTS

Corrosion of CaCO₃

The chemical reaction between AR and calcium carbonate could result in the dissolution of $CaCO_3$ that precipitated in MICP specimens. During the test, leached calcium ions was continuously measured from the effluent of the specimens. From the Ca^{2+} concentrations, the corroded calcium carbonate was theoretically estimated in each case, and the results are plotted and compared in Fig. 2. From the plots, it can be clearly observed that regardless of specimen cementation level, the corrosion of $CaCO_3$ was highly reliant on the pH condition of AR solution. Minor corrosion rates of calcium carbonate were observed to the specimens exposed to AR of pH 6.0, and the corrosion rate showed an increasing tendency with the decrease in pH condition. For instance, when the pH of AR decreased from 6.0 to 4.5, the corrosion rate increased by around 10 times, and that rate further increased at pH of 3.0.

The corrosion rate of calcium carbonate was also considerably influenced by the intensity of AR (Fig. 2). The low intensity of AR (20 mm/h) resulted higher corrosion rate compared to that under high intensity (100 mm/h). For example, by the end of the test (at pH 3.0), the specimens showed the corrosion of around 0.7 g $CaCO_3$ under low intensity of AR, which

was around 1.75 times higher than the corrosion experienced under high intensity of AR. This could be attributed to the contact time of AR within MICP specimens. During the high intensity of AR, certain hydraulic pressure was developed on the surface (due to ponding), which tended to infiltrate the AR at high rates. The less contact time of AR with soil MICP matrix resulted the less reaction with $CaCO_3$. On the other hand, the low intensity facilitated relatively slow infiltration, allowing sufficient time to the reaction with $CaCO_3$ during the percolation.

Mass Loss

Fig. 3 shows the mass loss of MICP samples as a function of delivered volume of acid rain at different pH conditions. As shown, the mass loss rate of MICP treated specimens increased with increasing delivered volume, and that increases with the increase in pH of the AR. The tendency of mass loss rate (Fig. 2) is in a good agreement with the corrosion rate of calcium carbonate (Fig. 3). However, the cementation level highly governed the rate of mass loss. For example, by the end of the test (at pH 3.0), all the specimens (C-1, C-2 and C-3, i.e. irrespective of the level of cementation) showed the same corrosion of calcium carbonate. The soil losses, on the other hand, were up to around 20%, 9% and 5% for C-1, C-2 and C-3 specimens, respectively, suggesting that high

cementation level attributes to high durability of MICP treatment against AR induced erosion.

From the physical observation, it could be seen that the specimens treated to high cementation level (C-3) showed lower physical impairments than those of low cementation levels (C-1). The specimens subjected to low intensity of AR delivery showed higher surficial damage compared to that of high intensity of AR. As the AR was delivered to the specimens from the top, the proton concentration of AR was high and effective at the supply point, resulted high dissolution of $CaCO_3$ at surface. When the solution reached specimen depths, the certain quantity of protons has already been reacted and neutralized, which leads to the less reaction at bottom specimens. At the same time, the specimens tested under submerged conditions showed clear damage structure on cylindrical surfaces as well, as the entire surface of the specimens was directly exposed to AR solution when submerged. The specimens treated by AR of pH 3.0 showed higher observable damages compared to those subjected to AR of pH 4.0. It also should be noted that specimens subjected to AR of pH 6.0 did not show any observable changes.
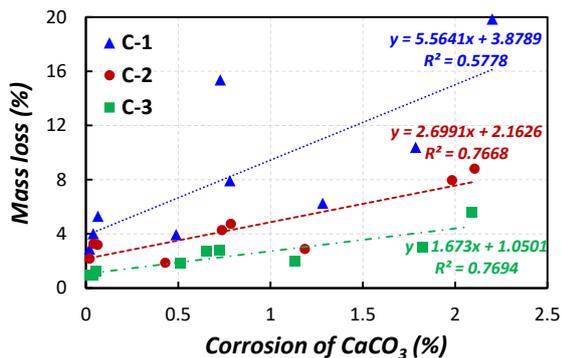
Fig. 4 Relationships between corroded $CaCO_3$ and mass loss at different cementation levels
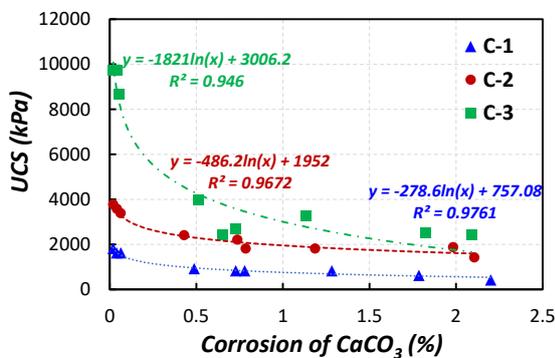
Fig. 5 Variation of UCS with corrosion of $CaCO_3$ content at different cementation levels

DISCUSSION

The corrosion of calcium carbonate bio-cement is the degradation mode of MICP under the exposure of AR environment. Under the high intensity AR delivery, the corrosion of calcium carbonate was low. In contrast, when the AR is delivered by low rate of infiltration or fully submerged conditions, the rate of corrosion was high, which is due to the sufficient reaction time given. Regardless of the conditions tested, the ultimate factor that determines the responses of the specimens exposed to AR, is the corroded quantity of calcium carbonate. Fig. 4 presents the compilation of mass loss data versus corresponding corrosion of calcium carbonate. The results suggest that the mass loss, with the corrosion of $CaCO_3$, can be fairly evaluated by linear relationships.

Fig. 5 presents the compilation of test data, i.e., UCS plotted against the corrosion of calcium carbonate. The results indicate that the UCS of the specimens exponentially decrease with the increase in corrosion of $CaCO_3$ for all the levels of cementation. The exponential tendency in decrease can be explained by the decay of matrix support. During the MICP treatment, the cementation begins at particle contact, increasing the strength gradually (progression of contact cementation); with the increasing treatment, the crystals starts to grow within the pore spaces, sometimes bridging the particles [2], [6], leading to rapid increase in strength at latter stage. The exponential progression of UCS in MICP soils was evidenced in many previous studies [6], [19]. The results presented herein (Fig. 5) demonstrate that the UCS follows relatively the similar path for the decrease during corrosion.

The results (Figs. 4 and 5) prove that the treatment durability essentially depends on high cementation levels. At high cementation level (e.g. C-3), particle contact points are cemented stiffly by higher quantity of calcium carbonate compared that in lower levels (e.g. C-1). For the same supply of AR, the corrosion of calcium carbonate might be same, irrespective of the level of cementation; however, the connections points could more quickly be debonded/ weakened in low cementation levels, showing high susceptibility to AR. On the other hand, for debonding/ weakening the particle contacts in high cementation levels, more calcium carbonates are required to be corroded, suggesting high longevity in performance.

CONCLUSIONS

Hypothetically, it was believed that the MICP treated soils are susceptible to acid rain conditions. However, this study has demonstrated that the durability and longevity can be considerably

enhanced by the levels of cementation. The tests suggest that the corrosion rate of calcium carbonate depends on the pH of acid rain and intensity. However, the loss of soil particles and strength loss due to corrosion of $CaCO_3$ is highly governed by the level of cementation. In another words, for the same quantity of corrosion, the higher the cementation level the lower the loss of mass and UCS, enhancing the longevity of the treatment.

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