Application of magnesium to improve uniform distribution of precipitated minerals in 1-m column specimens

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Abstract. This study discussed the possible optimization of enzyme-mediated calcite precipitation (EMCP) as a soil-improvement technique. Magnesium chloride was added to the injection solution to delay the reaction rate and to improve the homogenous distribution of precipitated minerals within soil sample. Soil specimens were prepared in 1-m PVC cylinders and treated with the obtained solutions composed of urease, urea, calcium, and magnesium chloride, and the mineral distribution within the sand specimens was examined. The effects of the precipitated minerals on the mechanical and hydraulic properties were evaluated by unconfined compressive strength (UCS) and permeability tests, respectively. The addition of magnesium was found to be effective in delaying the reaction rate by more than one hour. The uniform distribution of the precipitated minerals within a 1-m sand column was obtained when 0.1 mol/L and 0.4 mol/L of magnesium and calcium, respectively, were injected. The strength increased gradually as the mineral content was further increased. The permeability test results showed that the hydraulic conductivity was approximately constant in the presence of a 6% mineral mass. Thus, it was revealed that it is possible to control the strength of treated sand by adjusting the amount of precipitated minerals.

Keywords: precipitation; magnesium; uniformity; reaction rate; soil improvement

1. Introduction

Calcite precipitation techniques have been developed as bio-chemical grouting for their various possible applications as soil-improvement techniques. Calcite precipitation techniques are proposed as promising methods for improving the mechanical properties (Akiyama and Kawasaki 2012, Ivanov and Chu 2008, Putra *et al.* 2015), for reducing the permeability of porous materials (Harkes *et al.* 2010, van Paassen *et al.* 2009, Sidik *et al.* 2014, Whiffin *et al.* 2007, Yasuhara *et al.* 2012), for decreasing the swelling potential of clayey soil, and for mitigating the liquefaction potential (Ivanov and Chu 2008, Putra *et al.* 2015). Specifically, microbial-induced calcite precipitation (MICP) was confirmed as a potential soil-improvement technique (DeJong *et al.* 2010, Harkes *et al.* 2010, Ivanov and Chu 2008, van Paassen *et al.* 2010a, b). It has been shown

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that the engineering properties of soil, such as shear strength, stiffness, and permeability, can be significantly improved by this technique (DeJong *et al.* 2010, Harkes *et al.* 2010, Martinez *et al.* 2013). In this technique, microbes are used to catalyze the hydrolysis of urea and to dissociate the urea into ammonium and carbonate ions. The produced carbonate ions are precipitated as calcite crystals in the presence of calcium ions. However, the bacterial incubation may be difficult to control because special treatments are required (Chang and Cho 2014, Mitchell *et al.* 2005). The high concentration of reagents may have an inhibitory effect on the transport of bacteria (and hence, the bacteria activity) (Nemati *et al.* 2005, van Paassen *et al.* 2010b, Yasuhara *et al.* 2011).

Understanding and controlling the distribution of precipitated minerals are considered essential for practical applications of calcite precipitation techniques (van Paassen *et al.* 2009). Upscale of bio-mediated soil improvement, from the laboratory to the field, and its homogeneous strengthening over a large scale are the main challenges (Martinez *et al.* 2013, van Paassen *et al.* 2009). The most important factor for achieving the uniformity of precipitated materials is the distribution of microbes (Martinez *et al.* 2013). Several techniques have been developed to improve the homogeneity of the mineral distribution, such as the fixation of the bacteria distribution, the control of the reaction and the injection rate, and the modification of the injection pattern (Chang *et al.* 2016, Harkes *et al.* 2010, Martinez *et al.* 2013, 2014, Neupane *et al.* 2015b, Whiffin *et al.* 2007).

Enzyme-mediated calcite precipitation (EMCP) may be an alternative methodology among calcite precipitation techniques (Neupane et al. 2013, 2015a, Yasuhara et al. 2012). Using an enzyme is more straightforward than using bacteria because biological treatments do not need to be considered, even the application of enzyme as bio-catalyst of urea could be too expensive for large-scale application (Ivanov and Stabnikov 2017, Yasuhara et al. 2012). Enzyme reagent mixed solutions, which produce the precipitated calcite, were injected into soil specimens. Hence, the cultivation and the fixation of the enzyme were not required (Neupane et al. 2013, Putra et al. 2016, Yasuhara et al. 2012). The efficacy of the EMCP technique on a small scale (50 mm in diameter and 100 mm in height) and on a relatively large scale (57 cm in diameter and 60 cm in height) has already been evaluated in previous works (Neupane et al. 2013, 2015a, Putra et al. 2015, Yasuhara et al. 2012). The unconfined compressive strength ranges from 400 kPa to 1.6 MPa, and the permeability of the improved samples is reduced by more than one order of magnitude (Yasuhara et al. 2012). Upscale of the EMCP technique on a long sample has already been reported in a previous work (Neupane et al. 2015b). A higher amount of precipitated minerals was observed close to the inlet; this might be attributed to the faster rate of precipitation (Neupane et al. 2015b). Therefore, the control of the precipitation rate is an important task for achieving the uniform distribution within a large volume of soil samples.

The addition of magnesium to delay the inorganic carbonation rates of calcite was reported (Apriliani *et al.* 2012, Deleuze and Brantley 1997). The precipitation rate of calcite decreases with the increase in dissolved magnesium concentration (Mucci and Morse 1983). The application of magnesium as the added material in EMCP has been reported by Putra *et al.* (2016). The magnesium was able to be used as a delaying agent for calcite precipitation. In this study, the efficacy of magnesium was examined to control the mineral precipitation rate and to achieve the homogeneous distribution of the precipitated minerals within the soil samples. In order to adopt the calcite precipitation for a grout solution was utilized to evaluate the distribution of precipitated minerals within the 1-m column experiments. The effects of the precipitated minerals on the mechanical and hydraulic properties were examined through unconfined compressive strength

804

(UCS) and permeability tests, respectively. Subsequently, the possibility of controlling the strength by increasing the mineral mass within the soil samples was investigated.

2. Materials and reagent selection

The mixed solutions of reagents, i.e., $CaCl_2$, $MgCl_2$, urea, and purified urease, were used as the grout materials in this work. $CaCl_2$, $MgCl_2$, and urea, with claimed purity levels greater than 95%, were obtained from Kanto Chemicals Co., Inc., Japan. The urease was obtained from jack bean meal, with the urease activity of 2950 U/g, from the Kishida Chemical Co., Inc., Japan and was utilized as the biocatalytic dissociation of urea. The expected reactions to producing precipitation in the existence of calcium ions (Ca^{2+}) and magnesium ions (Mg^{2+}) delivered in the solutions are shown in Eqs. (1)-(5). A schematic of the precipitation process and the grouting mechanism expected are illustrated in Fig. 1.

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

$$CaCl_2 \rightarrow Ca^{2+} + 2Cl^{-} \tag{2}$$

$$MgCl_2 \rightarrow Mg^{2+} + 2Cl^{-}$$
(3)

$$Ca^{2+} + CO_3^{2-} \rightarrow CaCO_3 \downarrow \tag{4}$$

$$Mg^{2+} + CO_3^{2-} \to MgCO_3 \downarrow$$
(5)



Fig. 1 Schematic of precipitation process and grouting mechanism

Heriansyah Putra, Hideaki Yasuhara, Naoki Kinoshita and Akira Hirata

Silica sand #6 was prepared to examine the distribution of precipitated materials within the sand samples and the evolution of the engineering properties. The physical properties of silica sand are shown in Table 1 and Fig. 2. (Neupane *et al.* 2015b, Putra *et al.* 2016, Yasuhara *et al.* 2014).

The precipitated amount and the evolution of pH with time, corresponding to the different combinations of CaCl₂-MgCl₂, have been evaluated in our previous work. The precipitation of the minerals was evaluated directly in transparent test tubes. The total concentration of CaCl₂-MgCl₂ was fixed at 0.5 mol/L. The concentrations of MgCl₂, varying from 0.05 to 0.25 mol/L, were added to obtain the optimum combination based on the amount of precipitated minerals and the reaction rate. 1.0 g/L of urease was used to dissociate 0.5 mol/L of urea. The experimental conditions for the test tube experiments are listed in Table 2 (Putra *et al.* 2016). The evolving pH was also measured to examine the effect of magnesium on the rate of urea hydrolysis. The evolution of pH with time indicates the reaction rates of urea dissociation (Yasuhara *et al.* 2012). In the hydrolysis process, urease dissociates urea and produces ammonium, resulting in increases in the pH of the solution (see Eq. (1)). The pH meter, SATO SK-620, was used to measure the evolving pH 0, 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, and 12 hrs after mixing.

Properties	Keisha #6	
D ₁₀ [mm]	0.200	
D ₅₀ [mm]	0.340	
e_{\max}	0.899	
e_{\min}	0.549	
C_u	1.550	
G_s	2.653	
$ ho_{d\max}$	1.713	
$ ho_{d\min}$	1.397	
<i>k</i> [cm/s]	0.043	

Table 1 Properties of sand (Neupane et al. 2015b, Yasuhara et al. 2014)



Fig. 2 Grain size distribution of silica sand #6 (Putra et al. 2016, Yasuhara et al. 2014)

806

Case –	Reagent concentration [mol/L]		
	CaCl ₂	MgCl ₂	Urea
M0	0.50	0.00	0.50
M1	0.45	0.05	0.50
M2	0.40	0.10	0.50
M3	0.35	0.15	0.50
M4	0.30	0.20	0.50
M5	0.25	0.25	0.50

Table 2 Experimental conditions for test tube experiments (Putra et al. 2016)

A summary of the test tube experiments of the various combinations of CaCl₂-MgCl₂ is shown in Fig. 3 (Putra *et al.* 2016). Fig. 3(a) shows. The MgCl₂ with concentrations of 0.05, 0.10, and 0.15 mol/L (i.e., M1, M2, and M3, respectively) obtained higher precipitation ratios than that without magnesium (i.e., M0). The 20% increase in precipitated minerals was achieved by the addition of magnesium. The maximum precipitation ratio without magnesium was roughly 70%. The amount of precipitated minerals increased rapidly and approached the maximum precipitation ratio of 90% when 0.05 and 0.10 mol/L of magnesium were added. Subsequently, the mass decreased gradually as the concentration of magnesium was further increased. It is shown that the addition of a small amount of magnesium brings about a significant improvement in the precipitated mass. The results indicated that the addition of the appropriate amount of magnesium is essential to enhancing the mineral mass.

The evolution of the pH measurements is shown in Fig. 3(b). All the measured pH levels increased significantly when the reagent and the urease were mixed. In case M0, the maximum pH was obtained in 1 hr, then the pH decreased gradually and approached a steady state after 3 hrs. In cases M1, M2, and M3, the decrease in pH started after 2 hrs and reached the steady state after 8 hrs. In cases M4 and M5, the pH decreased after 4 hrs. The decrease in pH indicates that precipitated materials were formed (Handley-Sidhu *et al.* 2013). The increase in the elapsed time to reach the maximum pH when the magnesium was added indicates that the presence of Mg²⁺



Fig. 3 Results of test tube experiments: (a) Effect of addition of MgCl₂ on precipitation ratio; and (b) Evolution of pH with time (Putra *et al.* 2016)

ions delays the reaction rate. In case M0, the CO_3^{2-} ions bound the Ca^{2+} ion directly to produce $CaCO_3$. In other words, as magnesium is added, Mg^{2+} may inhibit $CaCO_3$ nucleation. This inhibition may be related to the high hydration energy of Mg^{2+} and to the calcite structure which allows for the incorporation of Mg^{2+} (Deleuze and Brantley 1997). This result indicates the possibility of using magnesium as a delaying agent in the precipitation process. In order to evaluate the applicability of magnesium to control the mineral precipitation rate and to achieve the homogeneous distribution of the precipitated minerals and to adopt the calcite precipitation technique in the real field, 1-m column experiments were conducted.

3. 1-m column experiments

1-m PVC cylinders (5 cm in diameter) were used for preparing the sand specimens to examine the distribution of the precipitated materials within the sand samples and their effects on the mechanical properties. For each experiment, firstly, dry silica sand #6 was poured into the PVC cylinder to obtain a relative density of 50% (Fig. 4). Distilled water was injected from the bottom to the top to remove air from the specimen. Afterward, two pore volumes of the selected grout solution were injected in the same way at certain curing time intervals. The curing time was determined by the observation of the test tube experiments. The solution was allowed to permeate throughout the porous media. A tubing pump was utilized to inject the grout solution with a constant rate of 40 mL/minute through the sand specimen.

After the solution injection and another curing time, the treated sand was removed from the PVC cylinder. The specimen was divided into five parts (Fig. 4), and the strength was evaluated through UCS tests. The tests were performed under wet conditions to avoid any unexpected precipitation that may occur when samples are intentionally dried out. a percentage error of 1.8%



Fig. 4 Schematic of 1-m column experiments

808

(Putra *et al.* 2016) was conducted to evaluate the distribution of precipitated minerals in the 1-m column experiments. About 50 g of treated sand were collected from each part, washed with distilled water to dissolve the product of NH_4Cl , and dried in an oven for 24 hrs. The dried sand was weighed and washed with 0.1 mol/L of HCl several times until air bubbles no longer appeared. The sample was dried again, and the final weight was taken. The dry weight lost during the acid leaching was evaluated and was assumed to be the weight of the mineral mass.

M1 and M2 were selected as the optimum grout solutions to be carried out in the 1-m column experiments as DM1 and DM2, respectively. The vertical distribution of the precipitated minerals within the sand specimen is depicted in Fig. 5. The mineral content is the ratio of the mass of the precipitated minerals to the mass of sand, expressed as a percentage. The precipitated minerals were concentrated close to the inlet. In DM1, 5% precipitated materials were obtained around the inlet; this amount then decreased significantly as the distance of the materials from the inlet increased. A relatively uniform distribution was achieved at a distance of 50 cm from the injection point. In DM2, the higher uniformity of the mineral distribution was achieved at a distance of 30 to 90 cm from the inlet. In comparison to the previous study, in which only the calcite precipitation (Neupane *et al.* 2015b) was addressed, the distribution of precipitated minerals in this study was more homogenous. The deviation of the mineral distribution without magnesium was roughly 2.042. The uniformity increased significantly and achieved the varied of 1.057 when 0.10 mol/L of magnesium was added. It was possible to reduce the deviation of the mineral distribution within the 1-m sand specimen up to 48%. The results indicated that magnesium can be used to enhance the uniform distribution of precipitated mineral distribution within a 1-m sand column.



Fig. 5 Vertical distribution of precipitated materials within 1-m sand column

Heriansyah Putra, Hideaki Yasuhara, Naoki Kinoshita and Akira Hirata

810

UCS tests were conducted to examine the effect of the precipitated minerals within the sand specimens on the mechanical properties. The relation between the mineral content and UCS is depicted in Fig. 6. A similar trend to that found in previous studies was obtained. The soil strength was seen to increase gradually as the mineral content was increased (Cheng *et al.* 2013, van Paassen *et al.* 2009, Whiffin *et al.* 2007). It was revealed that a lower concentration of magnesium in the grout solution may not influence the strength of treated sand. The results confirmed that it is possible to control the strength of treated sand by adjusting the mineral mass. The mineralogical substances were also observed throughout SEM and XRD analysis. Fig. 7(a) shows the SEM result of the treated sand. As is apparent, the precipitated materials are situated onto the free surface and at boundaries of grains. These materials are verified to be calcite by X-ray diffraction (XRD) (see Fig. 7(b)).



Fig. 6 Relation between mineral content and strength of treated sand



Fig. 7 The result of mineralogical analysis; (a) SEM; (b) X-ray diffraction

4. Permeability test

Permeability tests were also conducted to examine the effect of the precipitated minerals on the hydraulic conductivity of the treated sand and the possibility of controlling the amount of precipitated materials. An acrylic cylinder (5 cm in diameter and 10 cm in height) was used to conduct the tests. The fixed volume of the grout solution was injected into the prepared sand specimen. The procedure of the sample preparation for the permeability tests was equivalent to that for the 1-m column experiments. It was the same for the density of sand and the concentration of urease, urea, calcium chloride, and magnesium chloride. The procedure for the permeability test is illustrated in Fig. 8.

M2 was selected as the optimum grout solution based on the uniformity in the 1-m column experiments. Fig. 9 shows that the increase in precipitated minerals decreased the hydraulic conductivity significantly. As compared with the previous study, a similar trend was obtained. The hydraulic conductivity was approximately constant in the presence of a 6% mineral mass (Yasuhara *et al.* 2012). The permeability of 1×10^{-2} cm/s was obtained when 8% precipitated minerals existed within the sand sample. However, it is conceivable that the permeability could be reduced further with additional treatment. The amount of precipitated materials was able to be increased as long as the injected solution was allowed to permeate within the soil.



Fig. 8 Schematic of permeability test



Fig. 9 Effect of grouting materials in hydraulic conductivity

5. Conclusions

Achieving a uniform distribution of precipitated minerals is an essential issue in insitu calcite precipitation techniques. In this study, magnesium was newly added to the injected solutions to delay the reaction rate and to improve the homogenous distribution of precipitated minerals within soil sample. The addition of magnesium was found to be effective in delaying the reaction rate by more than one hour. The uniform distribution of the precipitated minerals within the 1-m sand column was obtained when 0.1 mol/L of magnesium was added. These results confirmed that the presence of magnesium delayed the reaction time, and hence, improved the homogenous distribution of the precipitated materials within the 1-m sand specimens. The strength increased gradually as the mineral content was further increased. It was possible to control the strength of the treated sand by adjusting the amount of minerals precipitated. The permeability test results indicated that the treated sand still permitted the addition of the solution. The strength of the improved sand was able to be controlled as long as the amount of precipitated materials could be increased.

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