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Efficacy of organic additive in EICP technique for soil improvement

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ABSTRACT

Application of enzyme as bio-catalysis in calcite precipitation technique has been confirmed as the potential soil improvement method (Yasuhara, 2012). In Enzymatically Induced Carbonate Precipitation (EICP) technique the calcite precipitation occurs after the chemical reaction between the urease enzyme mixed with urea and calcium chloride is induced in the soil. The precipitate of calcite has been used to fill up the void of sand, restricting their movement, and hence, improving the stiffness and the strength of soil. Although the calcium carbonate may improve the mechanical behavior of the soil when it precipitates on the surface of the soil particles by increasing inter-particle friction, the most significant impact on soil strength is induced when it precipitates at the particle on the contact point of the soil (Almajed, 2019). The utilization of non-fat milk powder added to the basic EICP solution is reported to result in larger calcium carbonate crystals and a higher proportion of calcium carbonate precipitation at inter-particle contact points (Almajed et al., 2019). In this experiment, the influence of adding dry milk as an organic additive material to the EICP solution was confirmed. Other organic material was also examined in this work. The various curing time with one cycle treatment were obtained. The unconfined compressive strength tests for enhanced strength of the soil, and the SEM tests for confirming the visualization of calcite precipitation were conducted.

1. INTRODUCTION

Application of bio-cementation in geotechnical and geo-environmental engineering currently being explored include sand cementation to improve mechanical properties and hence, to enhance bearing capacity and liquefaction resistance (Putra, 2017), and to control of soil erosion through surface stabilization (Harkes, 2010). A bio-mediated system generally refers to a series of chemical reactions that are conducted and controlled by biological activities. The most common method of bio-cementation is to utilize the formation of carbonate precipitation via hydrolysis of urea as the source of urease, and is referred to as microbial induced carbonate precipitation (MICP). The utilization of MICP method for soil improvement has been researched within geotechnical community for the past 15 years (Whiffin, 2004).

Recently, the urease enzyme from jack bean meal has been utilize for soil improvement, a process referred to enzyme induced carbonate precipitation (EICP). Application of powdered urease enzyme as bio-catalysis mixing with urea and calcium carbonate to produce calcite precipitation has been confirmed. (Neupane, 2013; Putra , 2016; Yasuhara, 2012; Almajed, 2019) used the powdered urease enzyme from jack bean and had been purified; calcite precipitation occurs after the chemical reaction between the urease enzyme mixing with urea and calcium chloride is induced in the soil. The reactions of the calcium carbonate formation are shown in the following eq. (1) – (3). The precipitated calcite may provide adhesive material between the grains of sand, restricting their movement, and hence, improving the stiffness and the strength of the soil (Yasuhara, 2011). Bio-cementation methods like EICP and MICP require less resources and release much less carbon emissions during application to soils than other existing methods of soil improvement such as the use of Portland cement.

$$CO(NH_2)_2 + 2H_2O \xrightarrow{\text{Urease}} 2NH_4{+} + CO_3^{2-} \quad (1)$$

$$CaCl_2 \longrightarrow Ca_2^{+} + 2Cl^{-} \quad (2)$$

$$Ca_2^{+} + CO_3^{2-} \longrightarrow CaCO_3 \text{ (precipitated)} \quad (3)$$

The calcium carbonate may improve the mechanical behavior of the soil when it precipitates on the surface of the soil particles by increasing inter-particle friction, the most significant impact on soil strength is induced when it precipitated at the particle on the contact point of the soil. (Almajed, 2019) reported that utilization of non-fat milk powder added to the basic EICP solution affects the size calcium carbonate crystals and a higher proportion of calcium carbonate precipitation at inter-particle contact points. In this experiment, the influence of adding dry milk powder as an organic additive material to the EICP solution was confirmed. The other organic material from soybeans powder was also conducted. The various curing time were obtained in the optimum treatment solution. The unconfined compressive strength tests with three different treatment solutions for enhanced strength of the soil were examined.

2. MATERIAL AND METHOD

2.1 Material

The mixed solution of reagent, i.e., $CaCl_2$, urea, and purified urease were used as the grouting material in this study. $CaCl_2$, and urea with purity levels greater than 95% were obtained from Kanto Chemicals Co., Inc., Tokyo, Japan and urease from jack bean meal, with urease activity of 2650 Units/g, was obtained from the Kishida Chemical Co. Ltd., Osaka, Japan. Non-fat dry milk, and soybean powder as organic stabilizer was used in the EICP modified solution. The experiment involves two series conditions of reagent solutions. The first test series involves an examination of the effect of non-fat dry milk, whereas the second test was examined the new candidate for organic stabilizer from soybean powder.

2.2 Test-tube experiment

The precipitation of calcite was evaluated directly in the transparent polypropylene (pp) tubes. The experimental procedures developed by Neupane (2013) were adopted in this study. The organic stabilizer was added for the injecting solution composed of CaCl2 and urea. Non-fat dry milk and soybean powder as organic stabilizer with the concentration of 4.0 g/L were added to obtain the total concentration of CaCl2-organic stabilizer of 1.0 mol/L, respectively. Purified urease with concentration 3.0 g/L was utilized to dissociate 1.0 mol/L of urea, and was mixed with distilled water for 2 mins. Therefore, the organic stabilizer-CaCl2, urea, and urease were thoroughly mixed with distilled water with a total solution volume is 30 mL. The curing time was allowed to react for 5 days curing time. (Putra, 2016) evaluated about after 5 days curing times the amount of precipitation is constant using urease enzyme from jack bean. The test tubes were cured at a room temperature of 20°C, while the sample was kept in a box without shaking. The particles deposited on the paper and the particles remaining in the tubes were dried at 60°C for 24 hours. The procedures for test-tube experiments are shown in fig. 1.

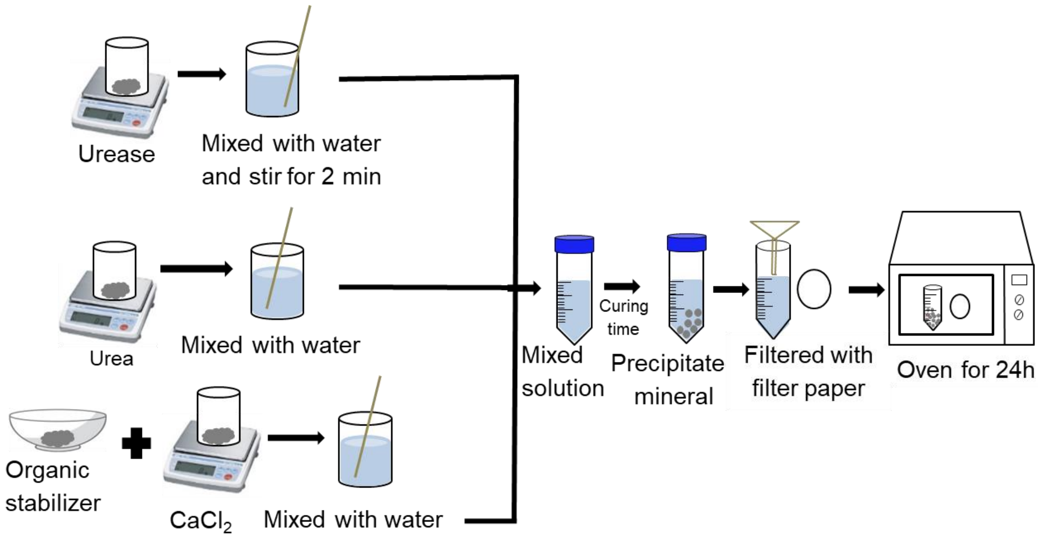

Fig. 1 Procedure for test-tube experiments

The precipitation ratio, which is the ratio of the actual mass of the precipitated materials to the theoretical mass of the maximum precipitation of calcium carbonate, given by eq. (4) (5):

$$precipitation\ ratio\ (\%) = \frac{actual\ mass\ (a_m)}{theoretical\ mass\ of\ CaCO_3 (t_m)} \tag{4}$$

$$t_m = C.V.M \tag{5}$$

Where a_m is the mass of the precipitated materials obtained from the test (g), t_m is the theoretical mass of $CaCO_3$ (g), C is concentration of the solution (mol/L), V is the volume of the solution (L), and M is the molar mass of $CaCO_3$ (100.087 g/mol).

A comparison of the precipitation ratios between basic EICP solutions with EICP modified was conducted, to evaluate the effect of adding dry milk powder, and soybean powder as organic stabilizer. The amount and the characteristic of the precipitated materials was obtained from the test-tube experiments, corresponding to the treatment solution, were also evaluated in this experiment. The material precipitation was crushed into a fine powder for XRD test to analyze and examine the mineralogical substances.

2.3 Unconfined compressive strength tests

Unconfined compressive strength tests were carried out to evaluate the improvement in stiffness and strength of the sand sample. PVC cylinders (5 cm in diameter and 10 cm in height) were used to prepare the sand samples. The reagent solution with concentration 1 mol/L, Urease enzyme 3 g/L, and organic additive 4 g/L was injected with one cycle treatment. The injected volume was controlled by the number of pore volumes plus 3 mm from the top of the sample, being ~80 mL (soaked condition).

First, 324 g of dry silica sand was poured into the PVC cylinders to obtain a relative density of 50%. Second, 80 mL of the treated solution, obtained from the test-tube experiments, were poured into the PVC from the top. The various curing time of the PVC cylinder test was conducted to obtain the optimum curing time for the specimens. After the curing time, the treated specimens were removed from the PVC cylinders. The surface of the treated specimens was flattened before the UCS tests were conducted. Two tests were performed for each condition to check the reproducibility.

3. RESULTS AND DISCUSSION

3.1 Test-tube experiment

The precipitated ratio for three conditions of treatment solution using basic EICP solution, and EICP modified with added dry milk, and soybean powder respectively, with 1.0 mol/L reagent solution and 3.0 g/L urease for five days curing time were evaluated. A result of the precipitation ratio after five days for the various condition of treatment solution is shown in Fig. 1. As is apparent in the figure, the amount of the precipitation almost same in various conditions were 100%. Thus, the added dry milk and soybean powder in EICP solution respectively did not influence the amount of precipitation.

The addition of organic stabilizer affected the characteristic of the precipitated materials. The XRD results Fig. 2 shows the impact of two various organic stabilizer on the crystalline material. The main material was calcite. The added of organic stabilizer promoted the vaterite peak. Table. 1 shows the characterization and percentage amount of mineral precipitation. The basic EICP solution results show that 100% of the mineral precipitation is calcite. While the EICP modified with added dry milk and soybean powder as organic stabilizer produce 9.7% and 13.1% of vaterite respectively.

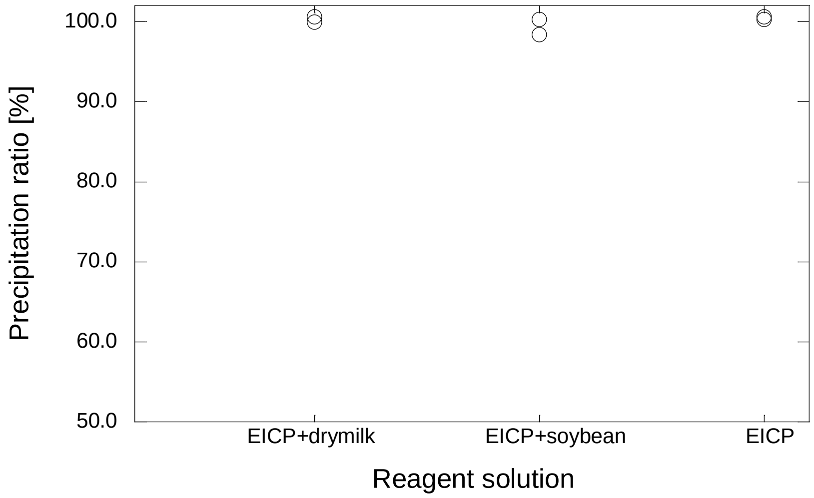

Fig.1 The comparison of precipitation test results using basic EICP and EICP modified

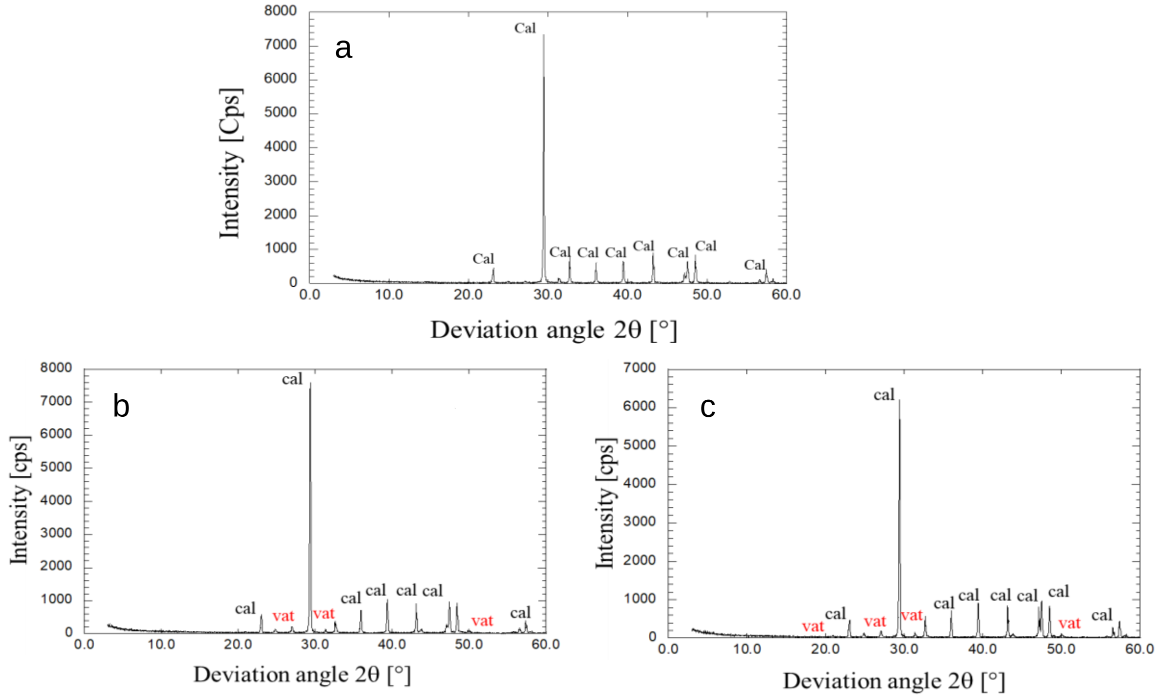

Fig.2 X-Ray Diffraction results of precipitated material (a) EICP solution (b) EICP modified with dry milk (c) EICP modified with soybean powder

Table. 1 Characterization and estimate amount (%) of mineral in material precipitation

Parameter	EICP solution	EICP modified	
		Dry milk	Soybean
Calcite Vaterite	100.0 0.0	90.3 9.7	86.9 13.1

3.2 Unconfined compressive strength (UCS)

The relation between the content of the precipitated carbonate and UCS of the various treated specimens after 3 days of curing time is presented in Fig. 1. The comparison of the compressive strength of the specimens treated adding dry milk and soybean powder to the EICP solution resulted in UCS between 120 kPa and 140 kPa, and the specimens without organic stabilizer had unconfined compressive strength between 54 kPa and 69 kPa at similar carbonate content.

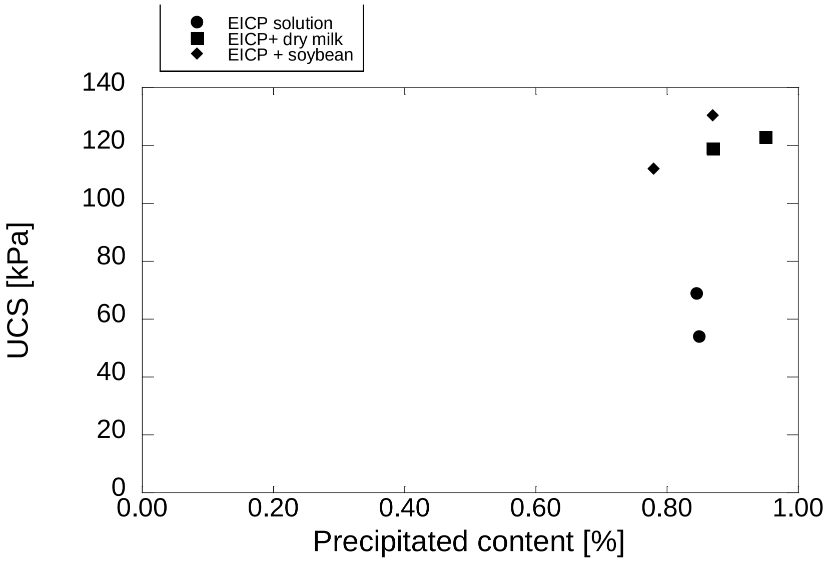

Fig. 3 Relationship between UCS results with precipitated content for various treatment solution

The investigated the effect of curing time for EICP modified treated sample, sample were left to cure 3, 7, and 14 days at room temperature after one cycle injection with EICP modified solution. The sample were flushed with distilled water and tested. Fig. 4 shows the relationship of UCS results with various curing time. The treated specimen using EICP modified with dry milk powder, left to cure for 14 days showed a peak strength of about 465 kPa with an increase of about 3 times compare to the samples left to cure only 3 days. This indicates that the curing time for 3 days were not enough for the compressive strength of treated soil to reach the maximum strength. This probably that the reaction was not concluded in three days and the increase in curing time allowed for an increase in calcium carbonate precipitation. Meanwhile, the treated of soil with EICP modified with soybean powder has maximum strength in curing time 7 days a peak strength of about 203.5 kPa with an increase of about 2 times compare to the samples left to cure 3 days. The higher variability in the UCS results may be indicates non-uniform distribution of the carbonate precipitation, which cause of the lower concentration of reagent solution (Rohy, 2019).

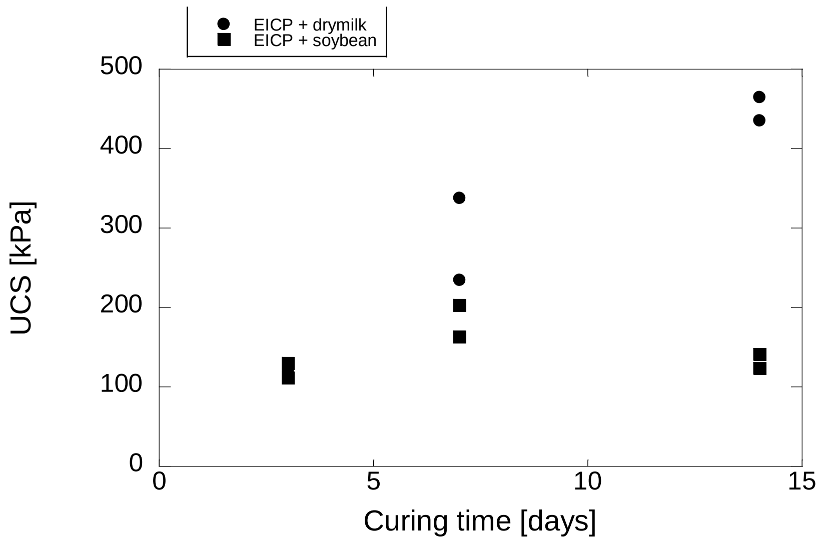

Fig. 3 Relationship between UCS results with curing time for EICP modified

The crystal shapes obtained from the SEM analysis is shown in Fig. 7. The crystal position and structure image of calcite using EICP modified treatment with dry milk powder is shown in Fig. 7 (a) and (b). SEM picture in Fig. 7 (c) and (d) show the soil treatment using EICP modified with soybean powder. In this picture, shows the sand particle with the calcium carbonate precipitation at the soil surface and inter-particle of soil.

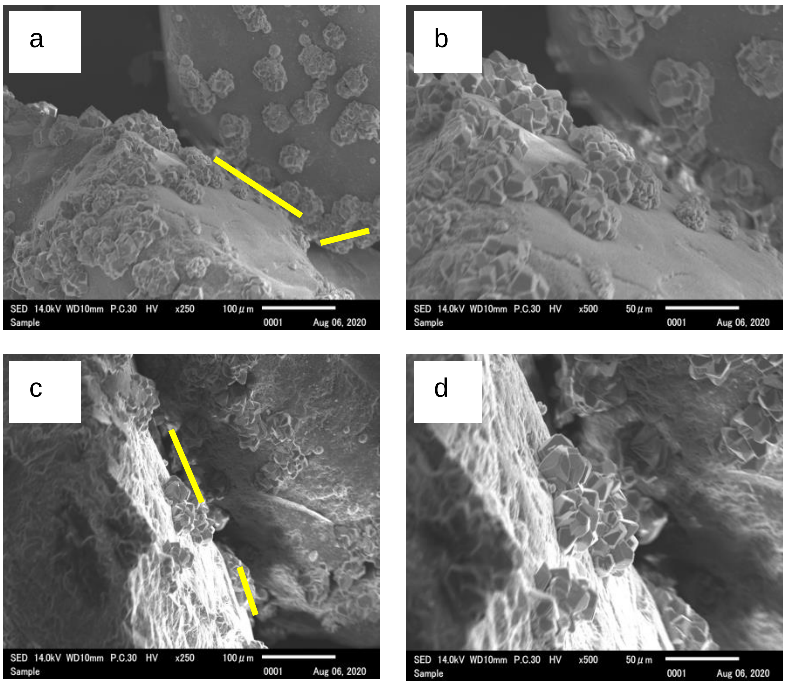

Fig. 4 SEM picture of a treated specimen with EICP modified (a) and (b) EICP modified with dry milk powder (c) and (d) EICP modified with soybean powder

4. CONCLUSIONS

This paper evaluated the efficacy of added organic stabilizers in the EICP method using one cycle treatment. Three different EICP solutions were investigated. The precipitation results indicate that the addition of organic stabilizer does not affect the amount of material precipitation. The utilization of dry milk and soybean powder as additives affects the strength of the soil stabilized using the EICP method. The comparison of compressive strength of soil with the same concentration of reagent solution in 3 days curing time shows, the EICP method with added the organic stabilizer has a higher strength than using the basic EICP method. XRD analysis has confirmed the characteristic of crystal in three conditions of treatment solution.

Higher variability is shown in UCS results, which may indicate the non-uniform distribution of calcium carbonate precipitation. An increased concentration of reagent solution may be required to increase the uniform distribution of carbonate precipitation. SEM analysis has confirmed that the calcium carbonate precipitation at the soil, and inter-particle of soil.

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