EFFECT OF CURING TEMPERATURE AND PERIOD ON ENZYMATIC INDUCED CALCITE PRECIPITATION PROCESS FOR SUSTAINABLE SOIL IMPROVEMENT

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Abstract

Enzymatic induced calcite precipitation (EICP) is a novel, innovative, sustainable and environmentally friendly technique for soil improvement. EICP utilizes extracted free urease enzyme to induce calcite precipitation. Being enzymatic reaction, EICP process is susceptible to variation in pH, temperature, concentrations of reactants and urease enzyme content. This research studied the effect of curing temperature, ranging from 30 to 60°C on the shear strength and calcium carbonate content (CCC) of EICP treated residual soils cured at 3, 7, and 14 days. Based on the experimental results obtained, it was found that curing temperature has tremendous effect on the shear strength and CCC, while curing period has insignificant influence on the strength and CCC of EICP treated residual soils. The results have shown that at 3 days curing period, maximum shear strengths of 763.29 and 876.92 kPa were determined at optimum curing temperature of 50ºC when the residual soil was treated with 0.50 and 1.00 M, respectively. It was also found that the increased in shear strength and CCC of EICP treated residual soil was insignificant beyond 3 days curing period. The UCS values of 0.50 M treated samples cured under 50°C at 3, 7, and 14 days were 763.29, 781.15, and 790.99 kPa. In summary, curing temperature has tremendous effect on the shear strength and CCC of EICP treated residual soil and maximum efficiency of the EICP process could be attained at 50°C.

Keywords: **Sustainable soil improvement, Enzymatic Induced Calcite Precipitation (EICP), curing temperature, curing period**

Introduction

Enzymatic induced calcite precipitation (EICP) is a bio-inspired technique of improving engineering properties of soils. EICP is a sustainable and environmentally friendly process as it avoids the use of cements and chemical additives that are prone to the release of greenhouse gases or other toxic by-products. Instead, EICP exploits free urease enzyme - a biological product, to catalyze urea hydrolysis reactionwhich lead to the eventual formation of calcium carbonate precipitation - known as

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biocement or bio-grouts. Many researches have shown that EICP has great potential for soil improvement (Almajed *et al*., 2018; Yasuhara *et al*., 2021). However, these techniques cannot be attained, especially for successful field implementations without studying the influence of environmental factors such as pH, temperature, and concentrations of urease enzyme and reactants on the efficiency and effectiveness of the EICP process (Nemati and Voordouw, 2003; Miao *et al*., 2020a).

EICP, as a catalytic reaction, is prone to be affected by environmental conditions such as curing temperature, curing period and pH. Temperature and pH were found to affect the activity of the urease enzyme (Dilrukshi and Kawasaki, 2016). Uniformity of calcium carbonate precipitation in EICP treated soils were also reported to be influenced by temperature (Neupane *et al*., 2015). Miao *et al.* (2020a) studied the effect of temperature, pH and source of calcium on the strength of EICP treated sand soil. Miao *et al*. (2020b) determined that EICP can be effective at a wide range of temperature.

Dilrukshi and Kawasaki (2016) reported that the range of optimum temperature and pH for high urease activity extracted from jack bean fall between 65°C and 70°C and 6.0-7.0, respectively. They also reported that at a temperature beyond 70°C, the urease became inactive. Neupane *et al.* (2015) also studied the effect of temperature on the uniform distribution of calcium carbonate precipitations in EICP treated soils. They mixed two soil samples at two different temperatures, 5°C and 23.5°C, then cured at the same temperature 25°C in 100 cm column. They observed uniform distribution of calcite up to a distance of 90 cm in soils prepared at 5°C, while non uniform distribution of calcite was found in specimen mixed at 23.5°C. Miao *et al.* (2020b) subjected EICP treated desert soils to thawing temperature of up to 70°C. They determined that high precipitation ratio was achieved in the soils treated with up to 70°C.

It was also presented above that EICP being a chemical reaction is greatly influenced by many factors such as the concentration of cementation solutions, temperature, curing period and other environmental factors. However, the results found in literature were inconsistent, and thus insufficient to decide on the optimum environmental factors, especially temperature that yield maximum strength and calcium carbonate content via EICP technique. Therefore, this research is conducted to determine the effect of temperature and curing period on shear strength and $CaCO_3$ content of EICP treated residual soil.

Materials

The soil sample used in this research is a natural tropical residual soil collected at a depth of 1.5 m from block P16, Electrical Engineering department, UTM, JB (1°33′35″N, 103°38′38″E) Johor Malaysia. The physical properties tests that include particle size distribution, Atterberg limits, and specific gravity of the natural untreated soil was determined according to BS 1377-2 (1990). Table 1 shows the summary of the index properties of the natural residual soil. As can be seen in the table, the natural soil contains 58% fines, about 24.16% and 17.16% gravel and sand fractions, respectively. The natural soil's liquid limit, plastic limit, linear shrinkage limit, and plasticity index are 79, 30, 16, and 49, respectively. The soil is classified following the British Standard Classification System (BSCS) as sandy silts of very high plasticity (MVS). While based on the physical inspection, the soil was fine-grained in texture and reddish-brown in color.

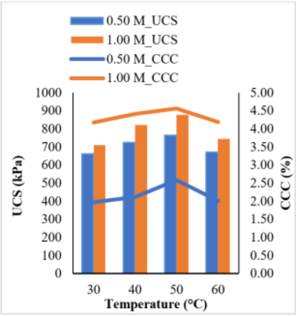
Table 1. Index Properties of the Natural Soil

Property	Quantity
Natural Moisture Content (%)	32.72
Percentage Passing 63 μm Sieve (%)	58
Gravel Fraction (%)	16
Sand Fraction (%)	26
Silt Fraction (%)	40.5
Clay Fraction (%)	17.5
Liquid Limit, LL (%)	79
Plastic Limit, PL (%)	30
Plasticity Index, PI (%)	49
Linear Shrinkage, LS (%)	16
Specific Gravity, G_s	2.63
Loss on Ignition, LOI (%)	12.28
Soil Classification (BSCS)	MVS (Sandy SILTS of Very High Plasticity)
Color	Reddish Brown
Clay Minerals	Kaolinte

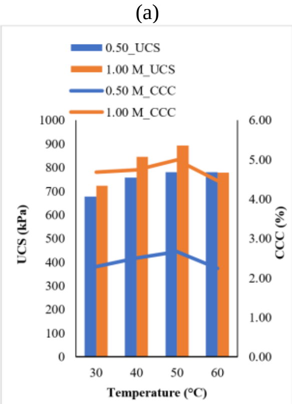
Methods

Sample Preparation

The EICP solutions for soil treatment were prepared at 0.50 M and 1.00 M equimolar concentrations of urea and $CaCl_2$. The prepared solutions were mixed with dried soil that passed through 2 mm BS sieve. The soil-EICP mixtures were compacted using both static compaction machine in order to form UCS samples of size 38 mm diameter by 76 mm height (length to diameter ratio equals to two). The prepared soil columns were then incubated at various curing temperatures (30°C, 40°C, 50°C, and 60°C) in



(a)



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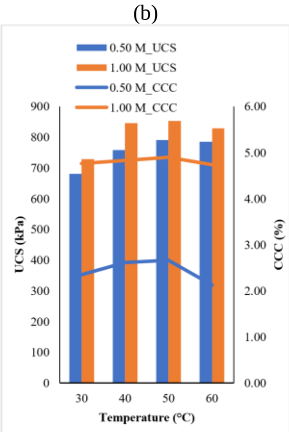


(c)

Figure 1. Effect of curing temperature at on shear strength and CCC of EICP treated soils cured for (a) 3 days, (b) 7 days and (14) 14 days

a humidity chamber for 3, 7-, and 14-days curing periods.

UCS Test

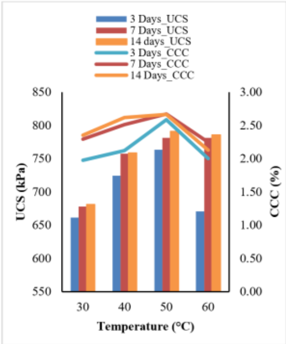
The untreated and EICP treated samples prepared and cured as described in above were subjected to unconfined compressive strength tests to determine their shear strength according to the procedure stipulated in (BS 1377-7, 1990). The prepared 38 mm diameter × 76 mm height cylindrical samples were mounted on machine pedestal between the upper and lower platens. The samples were crushed gradually at a rate of 2% per minute until the samples were failed. The applied axial forces and axial deformation were recorded automatically and saved at the end of the times. The reading was analysed to determine the maximum strength at which the soils fail.

Results and Discussion

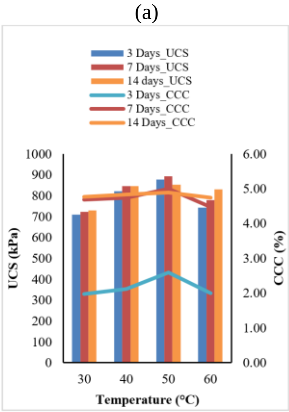
Effect of Curing Temperature on Unconfined Compressive Strength of EICP Treated Residual Soil

EICP as a chemical and enzymatic reaction was found to be influenced by curing temperature as reported by (Dilrukshi and Kawasaki, 2016). The effects of curing temperature on unconfined compressive strength and calcium carbonate content were investigated in this study. Figures 1(a)-1(c) illustrate the influence of curing temperature on the UCS of EICP treated residual soils prepared at two different concentration of cementation solution (0.50 and 1.00 M) and cured at various ages (3, 7, and 14 days). As can be seen in the figures, the change in curing temperature from 30°C to 60°C lead to variation in UCS. Regardless of the CCS and curing ages, the UCS raised with the curing temperature from 30°C to 50°C, and the UCS values drop at 60°C. In other words, maximum UCS values are obtained at 50°C. For example, in EICP treated soils prepared at 0.50 M cementation and cured for three days, the shear strength improvement ratio raised from 2.51 to 2.89 while the CCC increased from 1.98% to 2.59% due to variation in curing temperature of 30°C to 50°C. Similar trends were obtained in 1.00 M EICP treated soils as well as other curing periods. As illustrated in Figure 1, the increase in UCS with a temperature rise is attributed to the formation of more $CaCO_3$ content (Miao *et al*., 2020a). The reason for maximum UCS values and CCC can also be attributed to increase in urease enzyme activity at optimum temperature. As reported by Dilrukshi and Kawasaki (2016), maximum urease activity of jack bean enzyme occurred at high temperature in the range of 50°C-65°C.

The influence of curing temperature on the UCS and CCC was also studied by Nemati and Voordouw (2003); Neupane *et al.* (2015) and Miao *et al.* (2020a, 2020b). Miao *et al.* (2020b) determined that maximum calcium carbonate precipitations occurred in EICP treated soils at high temperatures close to 70°C. Optimum temperature close to 50°C as obtained in this study was also reported in the MICP treatment technique. For example, Cheng



(a)



(b)

Figure 2. Effect of curing age on shear strength and CCC of EICP treated at (a) 0.50 M and (b) 1.00 M cementation solution

et al. (2014) and Umar *et al.* (2016) found that higher strength gain and calcite precipitations are obtained at 50°C curing temperature. Keykha *et al.* (2017) found that the highest UCS was achieved when the MICP treated samples were cured at 40°C.

Effect of Curing Period on Shear Strength of EICP Treated Residual Soil

The influence of curing period on the shear strength and CCC were investigated by curing the EICP treated soils at various temperatures (30, 40, 50°C, and 60°C) for 3, 7, and 14 days. Figures 2(a) and 2(b) depicts the curing period's influence on the UCS values of EICP treated soils. There is little difference in UCS and CCC obtained at 3, 7-, and 14-days curing periods for a given curing temperature and concentration of cementation solution. As an example, the UCS of EICP treated soils cured at 40°C as shown in Figure 2(a) were 724.23, 757.56, and 758.59 kPa after 3, 7-, and 14-days curing periods. This represents a gain in strength of 4.60% and 0.14% between 3-7 days and 7-14 days, respectively. Similarly, the CCC corresponding to those 3, 7-, and 14-days curing periods are 2.21, 2.51, and 2.62%.

The results obtained are consistent with the findings (Lee and Kim, 2020; Miao *et al*., 2020b). Although increased in UCS were observed with the curing time in the works (Lee and Kim, 2020), the increase in CCC was insignificant. The increase of UCS in the work of (Lee and Kim, 2020), was attributed to the reduction in moisture content in the soils, which was not observed in this research. However, Miao *et al.* (2020b) found that EICP treated soils' hardness values remained constant after 7 days curing period.

Besides, the finding of this study agrees with the results obtained by (Nemati and Voordouw, 2003; Oliveira *et al*., 2017). For example, Nemati and Voordouw (2003) observed when the urease enzyme content was 0.1 g/L, the enzymatic reaction did not exceed 24 h to complete. Carmona *et al*. (2017) also observed a slight increase in strength and CCC for EICP treated soils cured beyond seven days.

Conclusions

The following conclusions were drawn based on the results obtained from the research.

i. It has been seen that curing temperature has significant influence on the strength and calcium carbonate content of EICP treated residual soils, while curing period has insignificant influence on both strength and calcium carbonate content.
ii. Regardless of the concentration of cementation solution as well as curing period, the strengths of EICP treated soils increased with the variation in curing temperature from 30°C to 50°C, then the strengths reduced with further increased in temperature to 60°C. maximum shear strengths were obtained at optimum curing temperature of 50°C.
iii. The results obtained have shown that irrespective of curing temperature and concentration of cementation solution, there were marginal increase in shear strengths of EICP treated with further increase in curing age beyond 3 days. This indicates that EICP process completed within short time, averagely 3 days.

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