Effect of Biocementation via Enzymatic Induced Calcium Carbonate Precipitation (EICP) on the Shear Strength of Compacted Clay Liner

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Abstract. The strength of residual soil treated via biocementation means by employing enzymatic induced calcium carbonate precipitation (EICP) was assessed based on the standard recommended for compacted clay liner. EICP treated samples were prepared at four different concentrations of cementation solution (urea-CaCl\textsubscript{2}) (0.25, 0.50, 0.75 and 1.00 M) and at various moulding water content (-2, 0, +2 and +4\% OMC) using reduced British standard light compaction effort (RBSL). The result obtained has shown UCS values of untreated natural soil at the four molding water contents were less 200 kPa, the minimum standard recommended for compacted clay liner. Upon EICP treatment, it was determined that the UCS values increase with the increase in the concentration of cementation solution. The treated soils at all the cementation solutions and molding water contents have UCS values greater than 200 kPa. The lowest strength of the treated soils was 468.5 kPa determined at 0.25 M cementation solution and +2\% OMC molding water content. The maximum UCS (643.5 kPa) value was determined at 1.00 M urea-CaCl\textsubscript{2} and -2\% OMC. The results also reveal that calcium carbonate content in the treated soil increases with the increase in the concentration of cementation solution. Microstructural analysis on the treated soil indicated the presence of white precipitation within the pore space of the soil, and the mineral was confirmed to be calcite through XRD analysis.

1. Introduction

Compacted clay liner is one of the most essential components of engineered sanitary landfills provided to protect groundwater and native soil from the harmful effect of decomposed leachate [1]. Constructability in terms of shear strength is one of the four main criteria considered in the selection of suitable material for the construction of a liner [2]. Compacted clay liner should have adequate strength that is capable to sustain its own self-weight and expected solid waste. The shear strength of compacted clay liner should be able to withstand stresses that could develop from wet-dry or freeze-thaw cycles [3]. A value of 200 kPa unconfined compressive strength (UCS) had been recommended in literature by Daniel and Wu [4] as minimum strength suitable for compacted clay liner.

Not all natural soil meets the engineering requirements for the intended use. Thus, soil improvement is one of the most significant tasks of geotechnical engineering [5]. Considerable
conventional materials (cement, lime and bentonite) and alternative materials (fly ash, rice husk ash, palm fuel ash) were investigated in the past to either substitute or amend natural native soil for the construction of compacted clay liner [1]. Although these materials were found to be effective in soil improvement. But, the majority of the investigated materials are threat to the environment due to either emission of greenhouse gases or release toxic by-product [6–8].

Accordingly, a new soil improvement technique known as bio-mediated soil improvement technique is being developed recently to reduce the negative environmental impacts posed by cement and other chemical stabilizers employed in soil improvement [9–11]. The technique uses either microorganisms or biological products to produce a chemical substance that would eventually improve the engineering properties of soil [10,12].

In EICP, free urease enzyme is used instead to trigger the formation of calcium carbonate precipitation via ureolysis [13]. The free urease in EICP can be extracted from an agricultural, microbial and fungal source. Agricultural urease is occurring in many high order plants such as sword beans, jack beans (Canavalia ensiformis), soybeans (Glycine max), melons, pine family, mulberry leaf, and pigweed [11].

Research into the use of EICP for civil engineering applications can be traced back to 2003 on the work conducted by Nemati and Voordouw [14]. They investigated the influence varying the strength of urease enzyme, urea and calcium chloride on the hydraulic conductivity of sandstone. Recent studies conducted on the improvement of shear strength of sandy soils using EICP process includes [15,16].

Hitherto, limited studies have been conducted on the application of MICP and EICP in fine-grained clay soils, specifically in improving engineering properties of compacted clay liner. Among the few works conducted on MICP with residual soils is that of [17,18]. This study is intended to improve the strength of residual clay soil for application in compacted clay liner at reduced British standard compactive effort.

2. Materials and Methods
2.1 Materials
The natural soil utilized in this work was sampled using a disturbed sampling method below a depth of about 1.5 m at Universiti Teknologi Malaysia (UTM) JB. The soil was reddish-brown and dominated by kaolinite clay minerals as revealed by XRD analysis.

The EICP solutions employed for this study are made up of free urease enzyme, urea and calcium chloride dihydrate. Four different equimolar concentration of urea-CaCl$_2$ (0.25, 0.5, 0.75 and 1.00 M) were used in the treatment process.

2.2 Methods
In order to determine the index properties of the natural soil, the laboratory tests that include particle size analysis, specific gravity and Atterberg limits (liquid limit, plastic limit, linear shrinkage, and plasticity index) were conducted based [19]. Reduced British standard light (RBSL) is selected in this study for the compaction test and subsequent unconfined compressive strength test. Compaction test using RBSL involves impacting 2.5 kg rammer falling from 300 mm on three layers of soil, each of which receives 15 number of blows. The test was performed on the natural residual soil sample that passed through a 20 mm size sieve as described in BS 1377 [20].

The EICP solution for the treatment was first made by mixing urea (CO(NH$_2$)$_2$) and calcium chloride dihydrate (CaCl$_2$.2H$_2$O) at various equimolar concentrations (0.25, 0.50, 0.75 and 1.00) with distilled and de-ionized water until all the solutes were dissolved. The free urease enzyme at 3 g/L was then added to the prepared urea-CaCl$_2$ solution and stirred for about 5 minutes to form EICP Solution. The EICP solutions were used to prepare UCS samples at various molding water contents relative to the optimum moisture content determined in the previous section. The mix and compact method was employed in preparing the UCS sample. The dried soils were mixed with EICP solutions and then
compacted to various densities corresponding to the molding water contents -2%, 0%, +2% and +4% using static compaction machine. The extruded were then cured for 3 days in a humidity chamber that was operating at 99 RH and 25 °C.

The Unconfined Compressive Strength, UCS test was conducted by following the procedure described in [19]. The unconfined compressive test was performed by mounting the cured samples prepared in section 2.2.3 on the triaxial compression machine. The samples were compressed uniaxially until failure at a rate of 1.52 mm/min.

About 20 g of dried soil specimens, obtained from failed UCS samples were used for the determination of calcium carbonate content. The dried soils were soaked in a 4 M HCl acid for 24 hours. The mixture of the soil dissolved CaCO₃ and acid were washed thoroughly with distilled by using a 63 µm sieve. A filter paper was then used to separate the soil residue and the liquid. The soil residue was then dried in an oven and then weighed. The amount of calcium carbonate content in the treated soil was computed using the formula shown in equation 1, as adopted from Choi et. al [21].

\[
CCC = 100 - \left( \frac{B}{A} \right) \times 100
\]  

(1)

CCC = Calcium carbonate content (%)  
B = Mass of oven dried soil post washing  
A = Mass of oven dried treated soil before washing

The scanning electron microscope (SEM) and X-ray diffraction (XRD) analyses were conducted on the natural and EICP treated soil in order to investigate the change in morphology and phases due to the formation of calcium carbonate precipitations and also to confirm the mineral content of the precipitation formed.

3. Results and Discussion

3.1 Index Properties of the Natural Soil

The physical and microstructural properties of the natural soil are presented in table 1. The soil is classified as sandy silts of high plasticity according to BSCS. The dominant clay mineral in the soil is kaolinite as revealed by XRD analysis on the natural soil. The soil was found to have a high liquid limit, plastic limit and plasticity index of 79, 30 and 49% respectively. The values of maximum dry density and optimum moisture content were 1.36 Mg/m³ and 32% respectively. These values are later used in soil preparation for the unconfined compressive strength test.

| Property                          | Quantity     |
|-----------------------------------|--------------|
| Natural Moisture Content (%)      | 32.72        |
| Percentage Passing 63 µm Sieve (%)| 57           |
| Gravel Fraction (%)               | 24.16        |
| Sand Fraction                     | 17.16        |
| Liquid Limit (%)                  | 79           |
| Plastic Limit (%)                 | 30           |
| Plasticity Index (%)              | 49           |
| Linear Shrinkage (%)              | 16           |
| Specific Gravity                  | 2.63         |
| BSCS Classification               | Sandy silts soil of high plasticity |
| Maximum Dry Density (Mg/m³)       | 1.36         |
| Optimum Moisture Content (%)      | 32           |
3.2 Unconfined Compressive Strength (UCS)

The relationship between UCS and molding water contents for all the natural and treated soils are shown in figure 1. There is a general decrease in UCS with molding water in both natural soil and enzymatic-induced calcium carbonate (EICP) treated soils. At higher molding water contents, the soil structures are deflocculated due to the loss of cementation between soil particles, thus reduction in shear strength. A similar pattern of results was determined by Eberemu [1].

Figure 1 also revealed that the samples of the natural soil compacted at RBSL compaction energy for all the molding water content (-2% to +4% relative to OMC) have UCS less than 200 kPa, the minimum standard required for compacted clay liner. The compacted soils have attained the minimum value of shear strength (>200 kPa) upon treatment with EICP solution at 0.25 M to 1.00 M cementation solution. The increase in UCS due to EICP treatment is attributed to the formation of bio-cement in form of calcite carbonate precipitates between soil particles pore spaces [22,23]. As also depicted in figure 3, UCS increases with the increment in the concentration of cementation solution at any particular molding water content. Thus, the highest values of shear strength of 643.5, 573, 563.5, and 533 kPa are obtained at 1.00 M concentration of cementation solution when the samples were prepared at -2, 0, +2 and +4% moulding water content relative to OMC, respectively. The highest UCS values of 2.61±0.55 Mpa were also obtained at 1.0 M cementation solution by Deng and Wang [24] when sandy soil were treated by the MICP technique.

![UCS at RBSL](image-url)

**Figure 1.** Variation of Unconfined Compressive Strength with Moulding Water Content in the Natural Soil and EICP Treated Soil

Figure 2 was plotted to explain the increase in calcium carbonate content in the EICP treated soil with the increment in cementation solution. As seen in the figure, calcium carbonate precipitations increase with a higher cementation solution from 0.25 to 1.0 M urea-CaCl$_2$. The figure also shows
that; calcium carbonate content reduces with the increase in molding water content relative to the OMC. As explained by Okwadha and Li [25], the increase in calcium carbonate precipitation with the increment cementation solution is due to the increase in urea available for conversion to carbonate ions and also the presence of the high amount of calcium ions from calcium chloride. While the reduction in calcium carbonate content with the increment in molding water content relative to OMC is ascribed to the high amount of water that led to the less absorption of calcium carbonate in higher molding water content [26]. Osinubi et al. [27] reported a similar pattern of results.

![Graph showing the effect of concentration of cementation solution on calcium carbonate content in the soil.](image)

**Figure 2.** Effect Concentration of Cementation Solution on Calcium Carbonate Content in the Soil

3.3 Microstructure of the Treated Soil

The SEM and XRD analyses were performed on both natural and EICP treated soil in order to confirm the formation of bio-calcite within the treated soil phase. Figure 3 shows the SEM analysis of the natural and treated soil. Formations of white precipitations can be observed in the SEM image of the treated soil. XRD analysis conducted on the treated soil indicated the presence of calcite mineral as can be seen in figure 4.
Figure 3. SEM Images of (a) Natural and (b) EICP Treated Soil

Figure 4. XRD of EICP Treated Soil

4. Conclusion and Recommendation
This study evaluated the strength of EICP treated residual clay soil relevant to compacted clay liner. The residual clay soil was treated with varying concentrations of cementation solution ranging from 0.25 to 1.00 M and the samples for unconfined compressive strength tests were prepared at 4 four different molding water contents relative to OMC (-2, 0, +2 and +4% OMC) using RBSL compaction effort. The results showed that the shear strengths of untreated residual soil 143.7 – 68.3 kPa were
found to be less than 200 kPa recommended for CCL, but upon EICP treatment the shear strengths were found to be well above 200 kPa. The shear strengths were seen to increase with increment in the molarity of urea-CaCl$_2$ solution. The highest strength of 643.5 kPa was obtained at 1.00 M cementation solution when the sample was prepared at -2% moulding water content relative to OMC.

Finally, in order to recommend the utilization of EICP treated residual clay soil for the construction of liner or cover in a hydraulic barrier, other parameters such as hydraulic conductivity, volumetric shrinkage strength and leachate compatibility of the treated soil should be investigated.

Acknowledgement
The authors hereby recognize the financial support from grants Q.J130000.2522.20H21-GUP from UTM, R.J130000.7851.5F256-FRGS from Ministry of Higher education and Q.J130000.2451.04G57 from UTM. The first and third author also appreciate scholarship support from TETFUND-Nigeria.

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