Evaluation of carbonate precipitation methods for improving the strength of peat soil

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Abstract. This research was carried out to evaluate the potential improvement of the strength of peat soil with the carbonate precipitation method. The grouting solution's optimum combination was determined and mixed to soil sample with a various density of 1.15, 1.20, and 1.25 g/cm³. The mass of precipitated carbonate within the soil and its effect on peat soil's increasing strength was evaluated by the unconfined compression (UCS) test. The mass of precipitated carbonate within peat soil was evaluated by acid leaching, respectively. The UCS test showed that the strength of treated peat soil had improved significantly. The treatment using carbonate precipitation brings about an improvement of 38% to 48% compared to the untreated sample. The maximum strength of 375.12 kPa was achieved at a density of 1.25 g/cm³. The result of this study also indicated that the initial density has no significant impact on the improvement of precipitated carbonate in peat soil. A relatively similar precipitated carbonate of 2-3% of soil mass was formed in the entire soil sample. This study elucidated that the carbonate precipitation method is a potential method to improve the soil's shear strength.

1. Introduction
Peatland in Indonesia spread over 21 million ha or 10.43% of Indonesia's land area [1]. This wide peatland has a significant issue and limitation for civil construction. Peat soil has a low shear strength, high compressibility, and long-term consolidation, which is resulting in massive deformation [2]. Several methods have been developed to improve the strength of peat soil, e.g., the cement grouting. It can improve the ultimate stress of loose sandy soil to 611 kN/m², which is around 27 times the ultimate stress of loose sandy soil state with 4% cement content [3]. However, this method is environmentally unfriendly because it can significantly reduce soil permeability [4]. One of the promising innovation methods of soil improvement that has great potential for application in the field and environmentally friendly is the carbonate precipitation method [5]. The precipitation of calcium carbonate can reduce compressibility and increase soil shear strength [6,7].

The carbonate precipitation method was induced by the reaction between urea and calcium chloride. Carbonate crystals are utilized as bridges between soil particles to prevent movement that increasing the strength and improved soil stiffness [8]. Microbial-Induced Calcite Precipitation (MICP) is one of the popular calcium carbonate precipitation methods. This technique uses bacteria such as Bacillus pasteurii to hydrolysis urea and promoted the precipitated calcite [9]. However, the use of bacteria has challenges, such as control the temperature, pH, and size of the bacteria itself should be considered [10]. Hence, the carbonate precipitation method without using bacteria such as Enzyme-Mediated Calcite Precipitation (EMCP) was developed as an alternative. EMCP method uses the urease enzyme to hydrolysis urea into carbonate ions (CO₃²⁻). Thus, promoted the calcite (CaCO₃) in the presence of calcium ions (Ca²⁺) [11]. The produced carbonate binds between soil particles and improves the soil strength and stiffness [12]. This method has been reported to improve the strength of sandy soil ranging from 200 kPa to 1.6 MPa [13]. The chemical reactions in promoting of calcite are shown in equation (1) - (3) [14].
CO(N\textsubscript{2}H\textsubscript{4})\textsubscript{2}+\textsubscript{2}H\textsubscript{2}O \xrightarrow{\text{urease}} 2NH\textsubscript{4}+CO\textsubscript{3}^{2-} (1)

CaCl\textsubscript{2} \rightarrow Ca^{2+}+2Cl^- (2)

Ca^{2+}+CO\textsubscript{3}^{2-} \rightarrow CaCO\textsubscript{3}↓(precipitated) (3)

The evaluation of the applicability calcite precipitation method on the peat soil was very limited. Hence, evaluating the effectiveness of the EMCP method in improving peat soils is a significant challenge to develop an effective and environmentally friendly method for improving peat soil strength. Soil density also needs to be concerned that it is related to soil subsidence that can damage the structure if the subsidence is not uniform [15]. The effect of soil density was evaluated by varying the soil density of the sample from the maximum dry density of the proctor test. This study evaluates the EMCP grouting solution's applicability to improve the shear strength of peat soil with a variety of density.

2. Material and Method

2.1. Material

The materials used in this study were urea (CO(NH\textsubscript{2})\textsubscript{2}, purity 95%) and calcium chloride (CaCl\textsubscript{2}, purity 95%) produced by Ajax Finechem Pty. Ltd., magnesium sulfate heptahydrate (MgSO\textsubscript{4}.7H\textsubscript{2}O, purity 99.5%) produced by Loba Chemie Pvt. Ltd., urease enzymes, hydrochloric acid (HCl), and peat soil. Peat soil used in this study is collected from Riau Province, Indonesia, with the properties and size distribution are presented in Table 1 and Figure 1, respectively. The peat soil sample classified as SM based on USCS Classification.

Table 1. Peat soil properties

| Soil properties          | Values | Units |
|--------------------------|--------|-------|
| Specific gravity (Gs)    | 2.35   |       |
| Ash content              | 7.04   | %     |
| Organic content          | 92.96  | %     |
| Plastic limit            | 42.00  | %     |
| Liquid limit             | 49.00  | %     |
| Plasticity Index         | 7.00   | %     |
| pH                       | 3.51   |       |

Figure 1. The grain size distribution of peat soil sample
2.2. Precipitation test
Test-tube experiments were conducted to determine the optimum composition of reagents (urea and CaCl$_2$) and the enzyme urease that formed the mass of carbonate (CaCO$_3$). Calcite precipitation was evaluated by precipitation test with the test-tube method. This method used a transparent polypropylene (PP) tube to observe the calcite precipitation of several reagents (urea-CaCl$_2$) and the urease enzyme. The selected grouting solution is determined based on the mass and its effect on treated soil's strength.

2.3. Soil preparation and UCS test
The peat soil was prepared in various densities based on the maximum dry density obtained from the compaction test. The compaction test was conducted based on ASTM D698 about Standard Test Methods for Laboratory Compaction of Soil Using Standard Effort. Thus, the various density of 50%, 70%, and 90% of the maximum dry density was selected as the soil sample. Thus, the selected grouting solution was prepared and mixed through the soil. The treated soil was prepared in a mold with 5 cm in diameter and 10 in height. The 50 g treated soil was compacted to make a sample height of 2 cm for every layer in five layers. Thus, the treated sample was cured for three days of curing time. The procedure of treated soil preparation is presented in Figure 2.

![Figure 2. Soil sample preparation for UCS tests](image)

UCS tests were performed to evaluate the effect of the precipitated carbonate in soil samples on the treated sample's strength. Samples that have been cured for three days are released from the mold tube and then tested with the UCS test. The UCS test is carried out based on ASTM D7012 about Standard Test Methods for Compressive Strength and Elastic Moduli of Intact Rock Core Specimens under Varying States of Stress and Temperatures and the study from [16].

2.4. Carbonate quantification
The carbonate quantification tests were conducted to evaluate the mass of precipitated carbonate formed in the soil sample. This test is carried out using the acid leaching method developed by Putra et al. 2016. Peat samples that through the curing time were taken as much as 50 g, washed with distilled water to dissolve NH$_4$Cl salt, and dried in the oven for 24 hours. A 50 g treated sample is collected and washed with HCl several times until air bubbles no longer appear. Thus, the sample was dried in an oven and measured its dry mass. During acid leaching, dry weight loss is evaluated and assumed as the mass of mineral mass [17]. The acid leaching process is shown in Figure 3.
3. Results and Discussion

The selected grouting solution was evaluated by the precipitated ratio of the test-tube method results. The selected grouting solution for peat soil improvement also needs concern for the soil's strength has been improved. [16,18] reported the results of carbonate precipitated by the test-tube method that mixed enzyme urease and reagent (CO(NH$_2$)$_2$; CaCl$_2$; MgCl$_2$; and MgSO$_4$). The precipitated ratio is obtained from the test-tube method. The amount of carbonate is formed carbonate in the soil and compared with the soil sample's mass. The UCS value showed the strength of the soil achieved. The selected grouting solution for peat soil improvement is presented in Table 2.

| Case | Urease (g/L) | Urea (mol/L) | CaCl$_2$ (mol/L) | MgCl$_2$ (mol/L) | MgSO$_4$ (mol/L) | Precipitated ratio (%) | Amount of carbonate (%) | UCS Value (kPa) | References |
|------|--------------|--------------|------------------|------------------|-----------------|-----------------------|------------------------|-------------------|------------|
| S1   | 2.00         | 1.00         | 0.96             | -                | 0.04            | 66.00                 | 10.00                  | 555.00           | Putra et al.[16] |
| S2   | 2.00         | 1.00         | 0.90             | -                | 0.10            | 113.17                | 8.00                   | 300.00           |            |
| C1   | 1.00         | 0.50         | 0.45             | 0.05             | -               | 90.19                 | 3.00                   | 70.00            |            |
| C2   | 1.00         | 0.50         | 0.40             | 0.10             | -               | 87.51                 | 3.00                   | 120.00           |            |
| U1   | 2.00         | 1.00         | 1.00             | -                | -               | 57.65                 | 9.50                   | 231.00           | Putra et al.[18] |
| U2   | 2.00         | 1.00         | 0.98             | -                | 0.02            | 60.34                 | 10.00                  | 508.00           |            |

The selected grouting solution is the S1 case. Based on the data in Table 2, the S1 solution improved soil strength to 555 kPa even though the test-tube precipitated ratio was only 66%. The S1 solution is better in improving sand soil than the S2 solution that improved soil strength to 300 kPa even though a precipitated ratio is 113.17%. The consideration of addition MgSO$_4$ over MgCl$_2$ is the expected reaction between CaCl$_2$ and MgSO$_4$ that formed gypsum, besides MgSO$_4$ (especially Mg$^{2+}$ ions) initiated aragonite formation. The formed gypsum can contribute to the strength of the soil. Gypsum reaction is present in equation (4) - (5) [18].

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Figure 3. Acid leaching process
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\[ \text{MgSO}_4 \rightarrow \text{Mg}^{2+} + \text{SO}_4^{2-} \quad (4) \]

\[ \text{Ca}^{2+} + \text{SO}_4^{2-} \rightarrow \text{CaSO}_4 \text{ (precipitated)} \quad (5) \]

The addition of Mg\(^{2+}\) ions promotes the formation of aragonite, which is a polymorph of calcium carbonate calcite. Aragonite with a hardness of 3.5 - 4 Mohs and solubility of \(6 \times 10^{-9}\) higher than those calcite with a hardness of 3 Mohs solubilities of \(3.36 \times 10^{-9}\) [19]. Aragonite can be formed at room temperature only if dissolved additives are Mg\(^{2+}\) ions [20].

Soil samples were made with various densities based on the results of the Proctor test. The maximum dry density of peat soil samples is 1.27 g/cm\(^3\). The optimum water content was obtained at 24.5%. The results of the Proctor test are presented in Figure 4. Peat soil samples varied with a density of 50%, 70%, and 90% of the Proctor test's maximum dry density. The dry density for each variation of 50%, 70%, and 90% is 1.15, 1.20, and 1.25 g/cm\(^3\). The optimum water content for each variation of 50%, 70%, and 90% density of the Proctor test is 22%, 19%, and 16%. Peat soil samples that were repaired by the EMCP method were made with selected grouting solutions. The conditioning experiments on peat soil sample variations are presented in Table 3.

![Figure 4. The result of the Proctor test](image)

**Table 3. Experimental condition of peat soil samples**

| Case | Density (%Proctor) | Dry density (g/cm\(^3\)) | Water content (%) | Urea (g/L) | Urease (mol/L) | CaCl\(_2\) (mol/L) | MgSO\(_4\) (mol/L) |
|------|--------------------|--------------------------|------------------|-----------|---------------|-----------------|-----------------|
| T90  | 90.00              | 1.25                     | 22.00            | 2.00      | 1.00          | 0.96            | 0.04            |
| T70  | 70.00              | 1.20                     | 19.00            | 2.00      | 1.00          | 0.96            | 0.04            |
| T50  | 50.00              | 1.15                     | 16.00            | 2.00      | 1.00          | 0.96            | 0.04            |
| U90  | 90.00              | 1.25                     | 22.00            | -         | -             | -               | -               |
| U70  | 70.00              | 1.20                     | 19.00            | -         | -             | -               | -               |
| U50  | 50.00              | 1.15                     | 16.00            | -         | -             | -               | -               |

Soil samples that were improved by the EMCP method showed the increasing strength for each varied density. The increasing strength of peat soil samples was observed from the UCS value change from untreated peat soils to treated samples. The UCS of untreated samples at variations of 1.15, 1.20,
and 1.25 g/cm$^3$ density is 114.63 kPa, 190.98 kPa, and 270.96 kPa. The UCS of treated samples at variations density of 1.15, 1.20, and 1.25 g/cm$^3$ density are 170.06 kPa, 270.66 kPa, and 375.12 kPa. The strength of untreated and treated peat samples at variations density of 1.15, 1.20, and 1.25 g/cm$^3$ density are 170.06 kPa, 270.66 kPa, and 375.12 kPa.

The strength of untreated and treated peat samples is presented in Figure 5. Also, the increasing strength of soil samples at variations density of 1.15, 1.20, and 1.25 g/cm$^3$ density are 48%, 42%, and 38%.

Figure 5. The increasing strength of peat soil samples

The EMCP method's application to improve peat soil was evaluated by the increased strength of the peat soil with an increase in density. The treated sample's increased strength was compared with the untreated sample at the same increase in density. Increasing density from 1.15 to 1.20 g/cm$^3$ has increased the strength of untreated soil by 76.35 kPa and treated soil by 100.6 kPa. Similarly, the increasing proctor density from 1.20 to 1.25 g/cm$^3$ density were increased the strength of untreated soil by 79.98 kPa and treated soil by 104.46 kPa. The carbonate formed in the treated soil could have a higher strength than the untreated soil. The increasing density of 20% increased the treated sample's strength to 100 kPa while the increasing density of 20% in untreated soil only increased the strength to 75 kPa. Therefore, the EMCP method has succeeded in increasing the strength better than the soil without the EMCP method treatment at the same increase in density.

Previous studies related to the peat soil improvement by carbonate precipitation method have been reported by Canakci et al. [21]. It showed an increase in peat soil strength who improved by the Microbial Induced Calcite Precipitation (MICP) method. Canakci et al. [21] have also reported that the peat cohesion was 8.4 kPa for untreated samples and 11 kPa for treated samples, which is the carbonate content in the soil was 19% [21]. The studies showed an increase in peat cohesion due to the carbonate precipitation method was 31%. The increasing strength of peat soils by the MICP method from the studies of Canakci et al. [21] compared with the increase in peat soil strength by the EMCP method.

The increasing strength of peat soil samples by the EMCP method in this study is higher than the study of Canakci et al. [21]. A low increasing strength of peat soil sample in the study of Canakci et al. [21] is caused by the MICP method's weakness that is bacterial activity. The bacterial activity is influenced by the physical and environmental conditions of bacteria [22]. Peat soils have a low pH; for example, the peat soils used in this study have a pH of 3.51. These bacteria cannot reproduce except at their optimum pH, which is pH 9 [23]. The consequence of that is the reproduction of bacteria in peat soils will be obstructed. Therefore, the EMCP method has an advantage in its possible application in peat soil because it used purified enzyme urease that is not from bacterial activity. Also, the addition of Mg$^{2+}$ ions in this study is indicated to promote aragonite, which increases the bonding strength is between soil particles because it has a hardness level of 3.5-4 Mohs [19].

Figure 5. The increasing strength of peat soil samples
The carbonate content is the mass of carbonate in the soil compared to the soil sample’s mass. The carbonate content of 1.15, 1.20, and 1.25 g/cm³ density were 1.5%, 1.9%, and 2.7% with the strengths was 170.06 kPa, 270.66 kPa, 375.12 kPa. Correlation between the mass of calcite formed and increased strength are presented in Figure 6. The increasing value of carbonate content can increase the strength of the improved soil sample. Carbonate content of 1.5%, 1.9%, and 2.7% can increase peat soil strength from untreated to treated samples by 48%, 42%, and 38%. The effectiveness of the carbonate content in increasing strength is better at low density than at high density. This result was observed from the sample with a 1.15 g/cm³ density of the Proctor test, which has a carbonate content of only 1.5% but can produce the highest strength increase of 48%. The high-density sample (1.25 g/cm³ density) only produced a smaller increase in 38% strength even though the carbonate content was the highest at 2.7%. It may be caused by the contribution of adding strength already fulfilled by the compaction effort itself. Therefore, the role of carbonate as a binder only has a small contribution to the increase in the high-density peat sample’s strength.

![Figure 6. Mass of precipitated calcite and strength in various of density](image)

The carbonate content formed in the soil for each density is close to the theoretical maximum carbonate content. The carbonate content formed in peat soil samples at 1.15, 1.20, and 1.25 g/cm³ density was 1.5%, 1.9%, and 2.7% compared to the theoretical maximum carbonate content that should be formed at 1.15, 1.20, and 1.25 g/cm³ density was 1.9%, 2.0%, and 2.2%. The carbonate content formed in the soil sample at 1.25 g/cm³ density of the Proctor test exceeds the maximum carbonate content that should be theoretically formed. This result is indicated by other minerals formed besides carbonate, specifically gypsum. Putra et al. [18] reported that the addition of MgSO₄ to the EMCP grouting solution was confirmed to produce gypsum based on the results of X-Ray Diffraction (XRD) analysis and Scanning Electron Microscopy (SEM) test. Gypsum can be formed from the reaction between the Ca²⁺ ion and the SO₄²⁻ ions [18]. Gypsum increased the measured mineral mass when acid leaching is done. Therefore, it is necessary to do a Scanning Electron Microscopy (SEM) test to see the shape of the minerals formed to justify gypsum. SEM test results can show minerals between peat soil particles. Also, the shape of peat soil particles and the distribution of carbonates formed can be seen to be identified [21].

4. Conclusions
The applicability of the calcite precipitation method as a peat soil improvement technique has been evaluated. The various density of peat soil is prepared and thus, is treated with the selected grouting
solution composed of reagent and urease enzyme. The impact of grouting solution on shear strength and formation of calcite was evaluated through the UCS and acid leaching test, respectively.

The selected grouting solution used for peat soil improvement consists of 2 g/l urea, 0.96 mol/l CaCl$_2$, and 0.04 mol/l MgSO$_4$. The UCS test result elucidated that the calcite precipitation method significantly affects the improvement of strength of peat soil samples. The strength improvement of 48% was achieved with a maximum strength of 375 kPa by applying the calcite precipitation method. Also, the increase in density had a significant effect on the UCS values of the treated samples than on the carbonate content. The maximum strength of 270 kPa was obtained without grouting solution.

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