Effect of confining pressures on the shear modulus of sand treated with enzymatically induced calcite precipitation

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Abstract. Enzymatically induced calcite precipitation (EICP) is a new breakthrough for liquefaction prevention. This approach uses urease enzyme directly instead of bacteria to hydrolyze urea that precipitated as calcite crystal by the availability of calcium ion. Cyclic triaxial shear test under undrained condition incorporated with bender element test were conducted conscientiously on the EICP-treated sands. The parameters study reviewed includes: size of the sand particle, confining pressure, calcite contents, and saturation degrees along curing. The effects of those factors on the shear modulus are systematically investigated and compared. It was revealed that the precipitated calcite wraps the sand particles, which supports simultaneously to the mechanical properties’ improvement. The sum of materials needed of urea and CaCl₂ to reach a target of maximum shear modulus can be diminished prominently by reducing the saturation degree along curing. It is also revealed that the formation of the precipitated calcite is more significant than its amount on the strength improvement. The maximum shear modulus ($G_{\text{max}}$) of the sands treated with EICP increases with increasing in the calcite content, confining pressure, and decreasing in the saturation degrees during curing but the influence of the sands grain size is insignificant.

Keywords: shear modulus, confining pressures, saturation degrees, calcite content, grain size.

1. Introduction
Enzymatically induced calcite precipitation (EICP) method is a new innovation for liquefaction countermeasure. This new approach applies directly urease enzyme to hydrolyze urea becoming carbonate ion. With the present of calcium ion, it reacts and precipitates as calcium carbonate and bind soil particles. Their bond would improve the strength of the soil and restrict not only pore pressures generation but stiffness degradation of the soil as well.

The usage development of the EICP approach as a grouting material for liquefaction mitigation requires understanding on some parameters. One of them is the shear modulus, both at very small shear strain and at the others. At very small shear strain level, shear modulus generally remains stable and maximum shear modulus is stated in this range. Maximum shear modulus can be benefitted for liquefaction prediction of soil in site by using of liquefaction approximation curve correlating liquefaction resistance and shear wave velocity prepared by researcher [1–5]. At a higher strain level,
shear modulus decreases with strain. The enormity of the strain depends mostly on the rigidity of the soil and pore pressure generation during earthquake. The variation of shear modulus with strain under undrained condition is very important for ground movement prediction. Many research dealing with shear modulus of sands treated with any kinds of grouting materials have been prepared [6–9]. However, research on the deformation characteristic of the soils improved with EICP is relatively sparse.

The quantity of material used in the EICP approach plays an important role on its mechanical behaviour. However, the more the material used, the higher the cost performed. On that reason, reduction in the amount of its forming materials is worth considering. It has been proved using scanning electron microscopy (SEM) images that in a lower saturation degree along curing, the calcite precipitation tends to conglomerate at the connection surface among soil particles which directly contribute to the strength improvement [10–12]. Research on mechanical properties of EICP treated sands show that at the same calcite content, sands treated at a lower saturation degree along curing had a bigger mechanical property than that cured at a 100% saturation condition. It reveals that the sum of substances used can be diminished for attaining a specific strength of treated soil by reducing saturation degree along curing [11, 13].

This study presents the deformation characteristic of EICP treated sands by performing a sequence of cyclic triaxial shear test under undrained condition integrated with bender element test in the laboratory. Testing parameters cover: confining pressure (CP), calcite content (CC), saturation degree along curing ($S_r$), and grain size of sand. Bender element test was conducted shortly before deformation characteristic test for determining shear modulus at a very small shear strain.

2. Materials and methods

The substances used in this research were: urea, CaCl$_2$, and urease enzyme which have a function as cementation materials. The expected reactions for precipitating calcite in the form of calcite crystal are as shown in equation (1) to equation (3).

$$\text{CO(NH}_2\text{)}_2 + 2\text{H}_2\text{O} \xrightarrow{\text{Urease enzyme}} 2\text{NH}_4^+ + \text{CO}_3^{2-} \quad (1)$$

$$\text{CaCl}_2 \rightarrow \text{Ca}^{2+} + 2\text{Cl}^- \quad (2)$$

$$\text{Ca}^{2+} + \text{CO}_3^{2-} \rightarrow \text{CaCO}_3 \downarrow \text{(precipitation)} \quad (3)$$

CO(NH$_2$)$_2$ and CaCl$_2$ are the chemical formula for urea and calcium chloride, respectively.

![Grain size distribution of sands used](image.png)

**Figure 1.** Grain size distribution of sands used [13].

Those materials would be combined with water as a solution at an own predetermined amount to achieve the expected of precipitated calcite at 100% efficiency of either 0.4% or 0.8%. This solution
was mixed with sands of either Keisha No. 4 (Kitanihon Sangyo Co. Ltd.) [14] or Toyoura sand, with the grain size distribution chart and physical properties as presented in figure 1 and table 1 respectively.

Table 1. Physical properties of sands used [13].

|                        | Keisha No.4 | Toyoura sand |
|------------------------|-------------|--------------|
| Specific Gravity G_s   | 2.65        | 2.64         |
| Maximum void ratio e_{max} | 0.804     | 0.973        |
| Minimum void ratio e_{min} | 0.605     | 0.609        |
| Mean grain size D_{50}  | 0.825       | 0.17         |

The predetermined amount of materials used is as presented in table 2. Dry sand was mixed with solution at Laboratory before it was tamped directly on pedestal to form a specimen with target relative density of around 50%. The time consuming at this stage was attempted to be less than 30 minutes for reducing the detriment of calcite bonding during sample preparation. After tamping, specimen was cured for around six hours, the time needed for attaining 100% calcite precipitation, and loaded isotropically to a CP of 50, 100, or 200 kPa in the the cell of the triaxial. During curing, the saturation degree of specimen was expected maintain stable of either 30% or 97%. Specimen was then saturated fully by flowing tap water from the bottom pedestal to the top until the B-value at least 0,95. In the next sequences, bender element test was performed for measuring shear modulus at the small shear strain just prior to undrained cyclic shear test. Deformation characteristic test was conducted at a condition of undrained to find out the changing on shear modulus at other shear strain levels. For that objective, cyclic stress ratio (CSR) was changed gradually step by step to a higher value on the same specimen and at the same frequency of 0.1 Hz until specimen was liquefied, as depicted in figure 2.

Table 2. The predetermined amount of materials used.

| S_c (%) | Dry sand (g) | Solution volume (ml) |
|---------|--------------|----------------------|
|         | Keisha No.4  | Toyoura              | Keisha No.4 | Toyoura |
| 30      | 315          | 300                  | 25          | 27      |
| 97      | 315          | 84                   |             |         |

At the end of the stages, real count of the calcite precipitated in the specimen was determined using acid leaching method. More detail information relating to this approach is available in Simatupang and Okamura (2017) [15].
3. Test circumstances and proceeds
Tests circumstances as prepared in table 3 was applied to study the influences of CP, CC, $S_{rc}$, and particle size of sand on the mechanical properties of sands. The test proceeds of the maximum shear modulus by utilizing bender element are also included.

| Table 3. Test circumstances and proceeds of the maximum shear modulus [13]. |
|---------------------------------|------------------|------------------|------------------|------------------|------------------|------------------|------------------|
| Saturation degree along curing | Effective | Concentration of reagent | Relative density | Observed | Maximum shear modulus | Relative density | Observed | Maximum shear modulus |
| $S_{rc}$ (%) | CP | $\sigma'_c$ (kPa) | CC | $D_r$ (%) | $CC_{obs.}$ (%) | $G_{max}$ (MPa) | $D_r$ (%) | $CC_{obs.}$ (%) | $G_{max}$ (kPa) |
| 30 | 50 | 50.3 | 0 | 54 | 49.5 | 0 | 53 |
| 0.5 | 53.0 | 0.38 | 154 | 49.9 | 0.39 | 161 |
| 1 | 52.9 | 0.79 | 201 | 51.9 | 0.80 | 204 |
| 100 | 0 | 50.5 | 0 | 77 | 49.7 | 0 | 72 |
| 0.5 | 51.6 | 0.39 | 195 | 51.5 | 0.41 | 210 |
| 1 | 50.1 | 0.81 | 259 | 47.7 | 0.79 | 263 |
| 200 | 0 | 51.3 | 0 | 114 | 51.6 | 0 | 104 |
| 0.5 | 52.6 | 0.41 | 231 | 48.2 | 0.39 | 230 |
| 1 | 50.1 | 0.79 | 288 | 48.6 | 0.81 | 283 |
| 97 | 50 | 50.3 | 0 | 54 | |
| 100 | 0 | 50.5 | 0 | 77 | |
| 1 | 51.5 | 0.79 | 207 | |
| 200 | 0 | 51.3 | 0 | 114 | |
| 1 | 53.0 | 0.79 | 235 | |
4. Bender element (BE) test
BE test was conducted shortly before characteristic test of deformation for determination of $G_{\text{max}}$ based on shear wave velocity $V_s$ measurement, according to:

$$G_{\text{max}} = \rho_{\text{sat}} V_s$$  \hspace{1cm} (4)

where, $\rho_{\text{sat}}$ denotes mass saturated density of the soil, and $V_s$ is expressed as the comparison of travel distance ($d$) to travel time ($t$).

$$V_s = \frac{d}{t}$$  \hspace{1cm} (5)

Travel distance, in this study $d = 9.6$ cm as shown at figure 3, is measured as the space between midpoint of the protrusions of the embedded BE in the specimen of height 4 mm [16, 17]. Input signal using sine waves with frequency of 10 kHz with an oscilloscope was applied during the test. Furthermore, stacking approach was applied for averaging in 10 times of the input and received signals.

![Figure 3. Travel distance measurement using BE.](image)

4.1. Maximum shear modulus
Equation (4) was applied for determining maximum shear modulus. The results of the test presented in table 2 and redrawn collectively in figure 4 give interesting information relating to the testing parameters. First of all, maximum shear modulus, $G_{\text{max}}$, increases with CP for both sands tested, Keisha No.4 and Toyoura sand, both in cases of untreated and treated sands. This information agrees with the formula

$$G_{\text{max}} \sim \left(\sigma_c'\right)^n$$  \hspace{1cm} (6)

postulated by researchers, where $G_{\text{max}}$ is comparable to the power n of 0.5 of the effective CP especially for untreated sands [18–21]. This value of “slope n” shown in figure 4(a) however decreases sharply for treated sands and stays in a lower value in a higher CC showing strength improvement. Lin et al. (2016) [22] and Montoya et al. (2013) [23] were also reported the same trend. Montoya further said that the addition of CC, it would then reach a steady state condition where there was no change in slope “n=0” due to an increase in CP. Second, $G_{\text{max}}$ of EICP treated sands increases sharply if compared to that of untreated sands. However, the improvement on that slowdown in a higher CC. Third, unlike with CP and CC, $G_{\text{max}}$ decreases with $S_{sc}$ in all cases of CP. In this case, lowering $S_{sc}$ becomes a choice for ameliorating $G_{\text{max}}$ as proved by researcher using SEM image [10–13]. At low $S_{sc}$, calcite tends to precipitate at contact point of grain directly relating to strength improvement. Fourth, figure 3(a) shows...
clearly that $G_{\text{max}}$ of sands tested, Keisha No.4 and Toyoura sand, is almost coincided each other both untreated and treated sands. This information gives indication that grain size effect on changing $G_{\text{max}}$ value is very small that can be avoided. Research prepared by Youn et al. (2008) [24] using Toyoura and silica sand with the same relative density $Dr$ of 50% shown the same trend. $G_{\text{max}}$ obtained on their study using bender element test agrees with that on this study.

The effectiveness of the EICP treated sands based on their improvement ratio of $G_{\text{max}}$ is drawn in figure 4(b). It is clearly shown that the improvement ratio increases with CC but decreases with CP and $S_{\text{rc}}$ irrespective grain size of the sands used. The effectiveness of the EICP treated sands is higher at lower CP as well as $S_{\text{rc}}$. Even though $G_{\text{max}}$ increases with CP as aforementioned, it is slow down in a higher CP. This fact is more significant particularly for treated sands. As a consequence, their improvement ratio is lower at higher CP. The improvement ratio of $G_{\text{max}}$ of EICP treated sands performed in this study achieves the value of around 4 particularly at low CP and $S_{\text{rc}}$. It means that $G_{\text{max}}$ of EICP lightly treated sands of less than 1% is 4 times higher than that of untreated sands. This achievement agrees with the results on $G_{\text{max}}$ test of treated sands presented by Delfosse-Ribay et al. (2004) [8]. Their value on the materials of silica, micro fine cement, and mineral grout are 3, 4, and 5 respectively.

| $S_{\text{rc}}$ (%) | 10 | 50 | 90 |
|--------------------|----|----|----|
| $S_c$ (%)          | 0  | 0  | 0  |
| $C_{\text{c}}$ (%) | 0  | 0  | 0  |
| $C_{\text{p}}$ (%) | 0  | 0  | 0  |
| $Src$ (%)          | 0  | 0  | 0  |
| $G_{\text{max}}$ (MPa) | 50 | 100 | 150 |
| $G_{\text{max}}/G_{\text{max}}(CP=0)$ | 1  | 2  | 3  |

**Figure 4.** Maximum shear modulus and improvement ratio.

5. **Deformation characteristic test**

The shear modulus evolution with strain of sands investigated for both cases of untreated and treated sands are shown at figure 5. Those graphs presented in figure 5 are clearly figuring that shear modulus depends mostly on strain. Like untreated sands, shear modulus of treated sands decreases along with increasing in shear strain and comes close to that of untreated sands at a higher shear strain [8, 22, 25, 26]. On the other hand, normalized shear modulus of sands treated with EICP approach is higher at bigger CC as a consequence of bond deterioration of the specimen due to shear strain elongation. The comparison with other test result of untreated sands prepared by Kokusho (1980) [20] has been performed and showing a good agreement. His test was conducted under the almost same condition in some cases with this study, such as: CP of 98 kN/m² and $Dr$ around 50%.
6. Conclusions

Research prepared using bender element test and undrained cyclic triaxial shear test give some information relating to test parameter reviewed. Some of them are as follows.

Maximum shear modulus of EICP treated sands increases with increasing in CP. That is proportional to the power “n” function of effective confining pressures which is slope of the line connecting log $G_{\text{max}}$ and log effective confining pressures. That “n” of EICP treated sands is smaller at higher CC is showing strength improvement until it reaches a steady state of “n=0”. Slope “n” of EICP treated sands decreases sharply to around 50% of that of untreated sands showing that little quantity of precipitated calcite can upgrade $G_{\text{max}}$ prominently.

Strength improvement of EICP treated sands can also be found by decreasing $S_{\text{cc}}$ expressing the potency of calcite position at low $S_{\text{cc}}$ rather than its quantity. In other words, the amount of forming material of calcite those are urea and calcium chloride can be reduced for attaining specific strength by reducing $S_{\text{cc}}$. On the other sides, $G_{\text{max}}$ of different sands is almost coincided each other at any cases of CP, and CC meant that grain size is insensitive for $G_{\text{max}}$ determination.

Shear modulus of EICP treated sands is strain dependent. It decreases with strain and decreases sharply at higher strain. At higher strain level of around $10^{-2}$, it comes close to that of untreated sands showing bond deterioration. Shear modulus reduction curve of EICP treated sands at strain level of more than $10^{-5}$ is shift below that of untreated sands. At this strain level, calcite bonding is predicted starting to deteriorate.

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