

EFFECTS OF DEGREE OF SATURATION ON LIQUEFACTION RESISTANCE OF SAND IMPROVED WITH ENZYMATIC CALCITE PRECIPITATION

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ABSTRACT: In recent years innovative methods of environmentally friendly and economically technique for liquefaction remediation are being developed. One of those technologies is microbial induced calcite precipitation (MICP). In MICP the microorganisms like soil bacteria are used to improve the mechanical properties of soils. The urease enzyme catalyses the hydrolysis of urea ($\text{CO}(\text{NH}_2)_2$) in a solution, and the carbonate (CO_3^{2-}) produced precipitates with the calcium ion (Ca^{2+}) as calcium carbonate crystals (calcite). The improvement in the liquefaction resistance of Toyoura sands with calcite precipitated was investigated through a series of triaxial tests in this study. Liquefaction strength curves correlating corresponding to $\text{DA}=5\%$ for treated and untreated sand were compared. The results show that the MICP can improve the liquefaction resistance of sand. The lower the degree of saturation, the higher the liquefaction resistance of treated sand in the same amount of calcite. Besides that, the greater the amount of calcite in the improved sand the stronger is the sand created.

Keywords: soil improvement, degree of saturation, calcite content, sand, triaxial test, liquefaction resistance.

INTRODUCTION

Past occurrences of earthquake were often followed by the liquefaction of soil which had caused severe damage to a number of structures. Traditionally, liquefaction mitigation measures such as various grouting methods using chemical additives (e.g. portland cement, lime, fly ash, bitumen) and mechanical methods (e.g. providing drainage ability, densification, and mixing of soils) are costly, energy consuming and composed of environmentally unfriendly materials (DeJong et al., 2010; Khodadadi et al., 2012). In recent years innovative environmentally friendly methods are being developed that utilizes precipitated calcium carbonate as a cementing material. The precipitation of calcium carbonate may be induced by the microbial metabolism (Nemati and Voordouw, 2003; Whiffin et al., 2007; DeJong et al., 2010; Harkes et al., 2010; Al Qabany and Soga, 2013).

This method, using the microbial metabolism, may be effective to strength soils, however, the evolutions in the mechanical properties induced by the microbial metabolism may not be straightforward enough to be controlled, because it may be impossible to constrain the extinction and/or the generation of living bacteria in natural environments. The research on calcium carbonate precipitation by bacteria has been mainly conducted using ureolytic bacteria. These bacteria indirectly produce precipitated calcium carbonate by a urease enzyme.

Yasuhara et al. (2012), on the other hand, employed the urease enzyme instead of using bacteria as a promoter for the hydrolysis of urea, which causes Ca^{2+} and CO_3^{2-} to precipitate as CaCO_3 and form into the void spaces and/or the surfaces of grains. Utilizing the enzyme itself is more straightforward than using bacteria, since the cultivation and fixation of bacteria (i.e., biological treatment) do not need to be considered.

Hayashi et al. (2010) conducted triaxial test on calcite precipitated sand to study liquefaction resistances and confirmed that this soil improving method significantly enhances not only the strength and stiffness of sand but the liquefaction resistance as well. In their tests, the specimens were prepared at saturated condition and then calcite was precipitated in the specimens. They observed sand surface with SEM and found that the precipitated calcite distributed more or less uniformly on the surface of grains and only a limited amount of calcite on the surface contributed to bond between particles. It clearly suggested that most portion of chemical regents injected into the sand were used to generate calcite on the grain surface which had nothing to do with mechanical properties in interest and only a limited amount of regents were used to bond between particles which directly contribute to improve the properties.

On the other hand, Okamura et al. (2012) has developed a technic to effectively desaturate a potentially liquefiable sand by injecting air directly into ground. They indicated that degree of saturation of sand around air injector lowered down to some 40% where sand was considered to be in the condition of the pendular saturation. In this unsaturated

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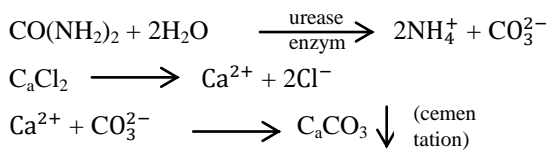
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condition, pore water exists mostly at the contacts between particles. Based on these facts, one of the author, Okamura, came up with an idea that calcite precipitation at lowered degree of saturation may enhance efficiency of soil improvement. Much smaller amount of calcite precipitation may be enough to improve soil properties as compared to the previous studies. In this method, liquefiable ground may be desaturated by injecting air first and chemical reagents are then injected. After allowing ample time for precipitation, air injection may be halted and degree of saturation becomes high again.

In this study, degree of saturation during calcite precipitation on the liquefaction strength of partially and fully saturated sand was investigated through a series of triaxial tests.

EXPERIMENTS

Reagent (consisted of calcium chloride- CaCl_2 and urea- $\text{CO}(\text{NH}_2)_2$) and urease enzyme were used in this experiment. The expected reactions for obtaining the precipitation of calcium carbonate- CaCO_3 (calcite) are as follows.



Precipitation Test in Test Tube

Precipitation tests with test tube were conducted to know appropriate amount of both the urease and the reagent to obtain desired calcite precipitated, as well as curing time needed to reach 100% precipitation of calcite. The combinations of the reagent concentrations and the amount of urease tested in this study are shown in Table 1.

Table 1 The Reagent Concentrations, the Amount of Urease in the Solutions, and the Expected Precipitation

Reagent Concentration (m/l)	Urease Amount (g/l)	Expected Precipitation (g/30ml solution)
0.2	2	0.6
0.3	2	0.9
0.5	3	1.5
	10	
1	3	3.0
	10	
	20	
1.5	25	4.5
	35	

Relationship between calcite precipitation ratio and curing time is plotted in Figure 1 at various content of urease enzyme (g/l) and concentration of reagent (m/l) consisted of urea and calcium chloride. For making sure the reproducibility of the materials used, two samples were prepared for each test condition. An increase in the urease amount effectively enhanced the precipitation ratio and accelerated the reactions. For example, 10 g/l urease content can only produce around 50% the precipitation ratio of 1 m/l reagent concentration along the curing time, while for 20 g/l urease level the 100% precipitation ratio had already been occurred nearby 36 hours of curing time. Contrastingly, the more the reagent concentration in the same urease level will produce the less the calcite precipitated. This is consistent with the result of the research conducted by Neupane, D, et al. (2013). The high concentration of reagent relative to urease content may restrict the performance of urease enzyme which may give a reduction in the amount of calcium carbonate precipitation. Urease amount implemented for the next experiment is based on the value resulting 100% or close to 100% precipitation of calcite with specific concentration of reagent.

Especially for urease amount of 2 g/l with reagent concentration of 0.2m/l and 0.3m/l, the observation was only conducted once when the curing time had already been three days.

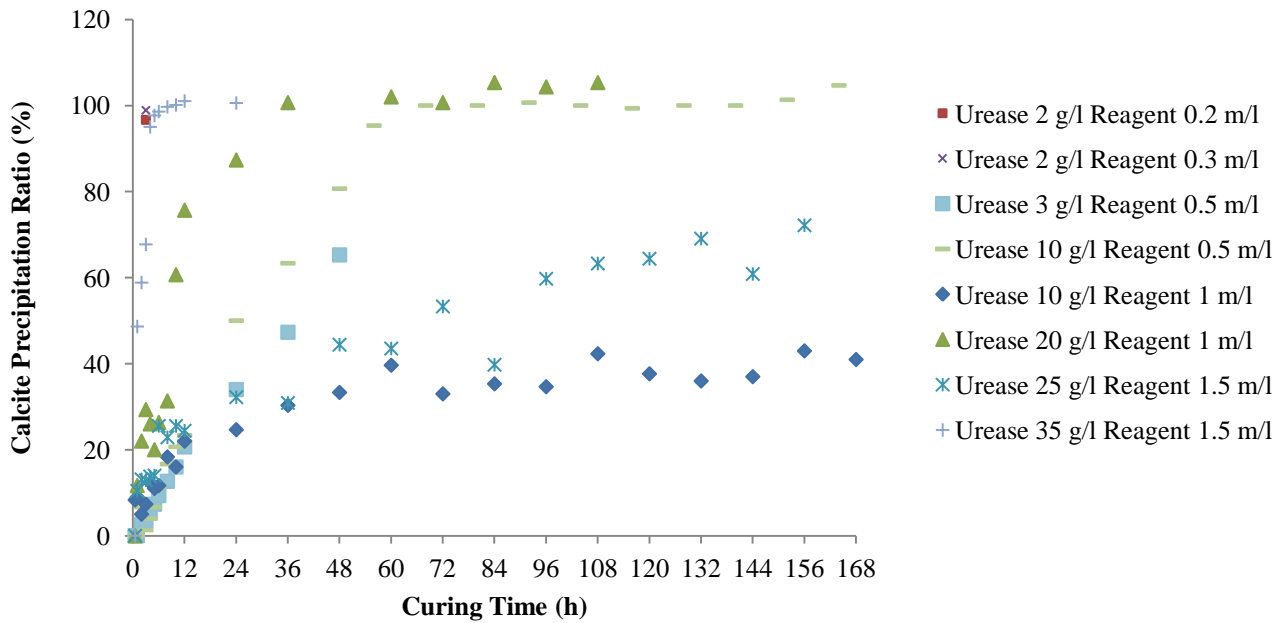


Figure 1 The Relationship Between Calcite Precipitation Ratio and Curing Time at Various Content of Urease Enzyme and Concentration of Reagent

Triaxial Test

The material used during the test performed in this experiment was Toyoura sand. It is a uniform fine sand composed mainly of quartz with the specific gravity (G_s) = 2.64, the minimum and the maximum void ratios $e_{min} = 0.609$ and $e_{max} = 0.973$ respectively.

Sample Preparation

All specimens were prepared using wet tamping method. Before tamping, predetermined quantity of dry Toyoura sand of around 60 gram for each layer was mixed in a transparent plastic bag with a certain amount of solution consisted of calcium chloride, urea, and urease (depended on saturation level needed). The quantities of material used are shown in Table 2. The mixed sand was then placed in a mould with internal dimension of 50 mm in diameter and 100 mm in high and tamped to the target relative density of 50% with a predetermined degree of saturation, either 40% or 70%. This procedure was repeated five time to build a 100 mm high sample in a short duration, typically 30 minutes, to minimize chemical reaction during tamping. The specimen was then subjected to a vertical pressure of 50 kPa for three days which duration is long enough to make the calcite precipitation ration of 100%.

The specimen was removed from the mold to a triaxial apparatus and was fully saturated with water. In this saturation process, more than two time the volume of water was flowed through the specimen to flush all the byproducts (ammonia) and possible chemical reagent remaining out from the specimen. Confirming that the Skempton's B-value higher than 0.95, the specimen was consolidated isotopically at an effective pressure of 50 kPa. The shear modulus at an axial strain level of 10^{-5} was measured and cyclic undrained shearing with a loading frequency of 0.1 Hz was performed.

Table 2 The Amount of Materials Used and the Expected Precipitation

Sr (%)	Solution Concentration			Dry Toyoura Sand (gr)	Water Volume (ml)	Expected C_aCO_3 Prec.		Solution Weight (gr)		
	urea (m/l)	CaCl ₂ (m/l)	Urease (gr/l)			Weight (gr)	Percentage (%)	Urea	CaCl ₂	Urease
40	0	0	0	300	36	0	0	0	0	0
	0.5	0.5	10	300	36	1.74	0.60	1.08	2.00	0.36
	0.25	0.25	2	300	36	0.87	0.30	0.54	1.00	0.07
70	0	0	0	300	63	0	0	0	0	0
	0.29	0.29	2	300	63	1.74	0.60	1.08	2.00	0.13

After the test, amount of calcite precipitated was measured. The specimen was oven dried and divided into three parts and weighed. Hydrochloric acid (0.1 mol/l) was used to rinse that sample. This acid leaching procedures was done three times for each sample to dissolve all the precipitated calcite followed by applying distilled water in every process

to clean both HCl and calcite that might be left in the sample. The disaggregated sand was dried again, and the loss in the dry weight was assumed to be the weight of the precipitated calcite.

Test Conditions

The test program was designed for assessing liquefaction resistance of improved sand which is cured at different degrees of saturations (S_r) and calcite contents. The concentration of the reagents was determined so that calcite precipitation ratio (CC) was either 0.3% or 0.6% at the degree of saturations as shown in Table 2. A total of 20 specimens were tested and the resultant ratios of calcite precipitation observed with the acid leaching are shown in Table 3.

It should be mentioned that the specimens were cured at different degree of saturations but fully saturated at the time of the cyclic shearing to avoid any possible effects of existence of air in the specimen. The comparisons of test results for specimens with the same CC but different S_r indicates effects of calcite precipitation characteristics, i.e. location of precipitation at sand grain surface.

Table 3 Testing Program and Testing Condition

Degree of Saturation S_r (%)	Reagent Concentration (m/l)	Calcite Content CC (%)	Relatif Density D_r (%)	CSR $\sigma_d/2\sigma'_c$	Number of Cycles (N)	
40	0	0	48.1	0.260	4	
			49.1	0.216	8	
			52.8	0.174	16	
			53.3	0.333	2	
			53.1	0.151	23	
	0.50	0.58	0.58	51.3	0.309	24
			0.58	52.1	0.363	11
			0.55	53.9	0.433	4
			0.61	52.9	0.211	41
			0.58	53.7	0.232	53
	0.25	0.30	0.30	52.7	0.262	4
			0.30	53.2	0.201	7
			0.20	52.0	0.146	44
	70	0	0	51.8	0.260	4
53.2				0.188	21	
54.1				0.140	26	
0.29		0.64	0.64	51.1	0.306	7
			0.63	53.5	0.260	12
			0.68	53.2	0.380	6
			0.67	53.6	0.174	145

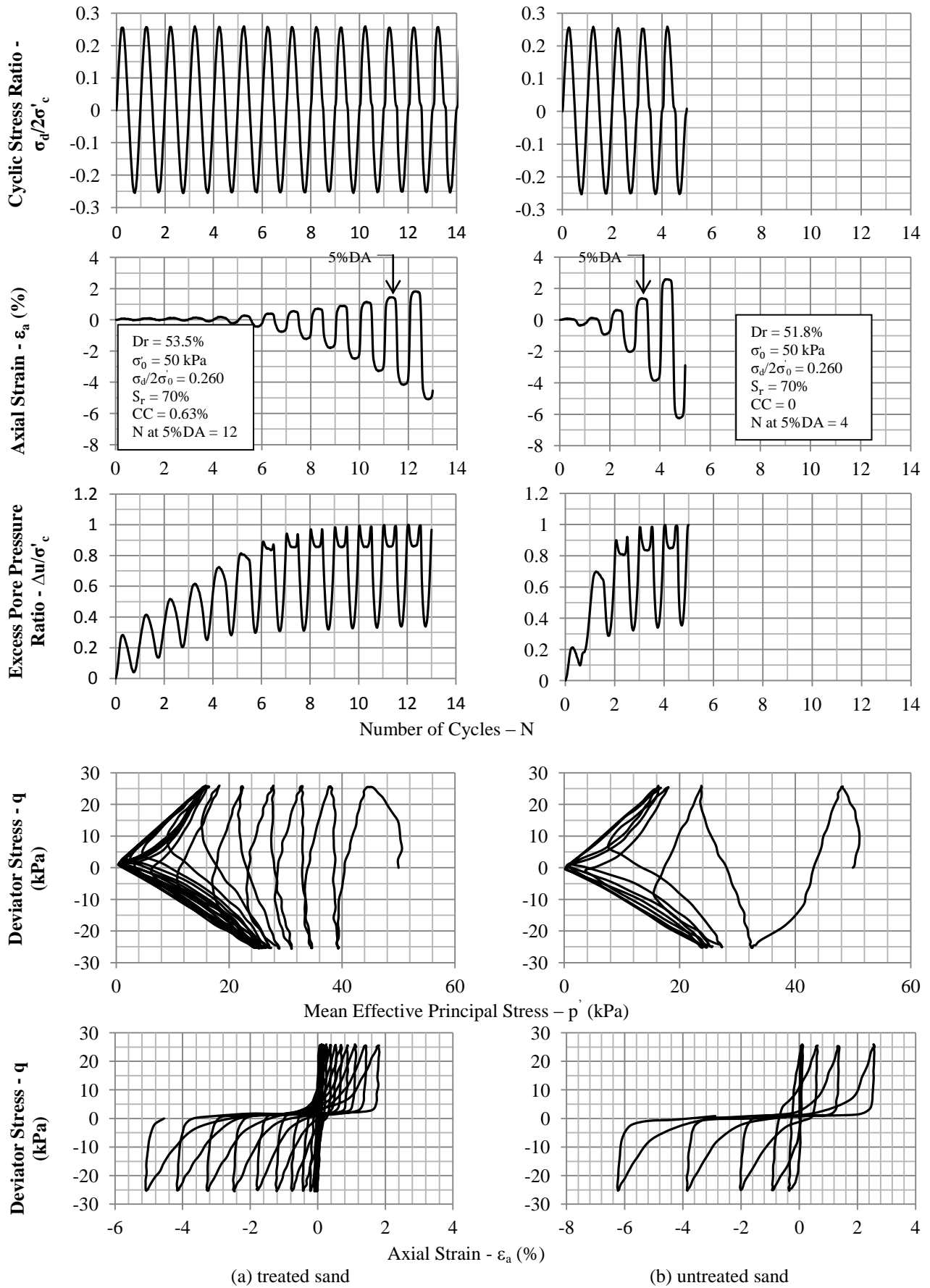


Figure 2 Typical Records of Cyclic Triaxial Test on Treated and Untreated Sand

RESULTS OF TRIAXIAL TEST AND DISCUSSIONS

Figure 2 displays typical records of cyclic triaxial test on both improved sand with calcite content (CC) = 0.63% cured at Sr= 70% and unimproved sand. The number of cycles needed to attain a 5% double amplitude axial strain were 4 for unimproved sand and 12 for improved sand, indicating clearly the effect of calcite precipitation on the liquefaction resistance of sand. Figure 3 shows relationships between cyclic stress ratio (CSR) and the number of cycles for double amplitude axial strain DA=5%. For cases of unimproved samples (CC=0), almost the same CSR was observed irrespective of Sr at sample preparation. CSR for improved sand significantly increased with increasing CC. In order to see effects of Sr at sample preparation and curing, results of the tests on specimen with similar CC but prepared at different Sr are compared in Fig. 3(c). Although the specimens of Sr=40% had slightly lower CC than specimens of Sr=70%, the specimens at lower Sr exhibited apparently higher resistance to liquefaction. The liquefaction resistances of both the improved and unimproved specimens are shown in Fig. 4, together with that obtained by Hayashi et al. (2010) who conducted similar test on specimens with CC=1% and Sr=100% during the curing. It was found that improved liquefaction resistance of approximately 0.3 was obtained with much smaller CC by reducing Sr during the curing. This is probably due to the fact that precipitated calcite tends to concentrate to the contact points between sand grains as degree of saturation decreases. This hypothesis, however, have to be confirmed by observing grain surface. Detailed observation with SEM will be reported elsewhere in the near future.

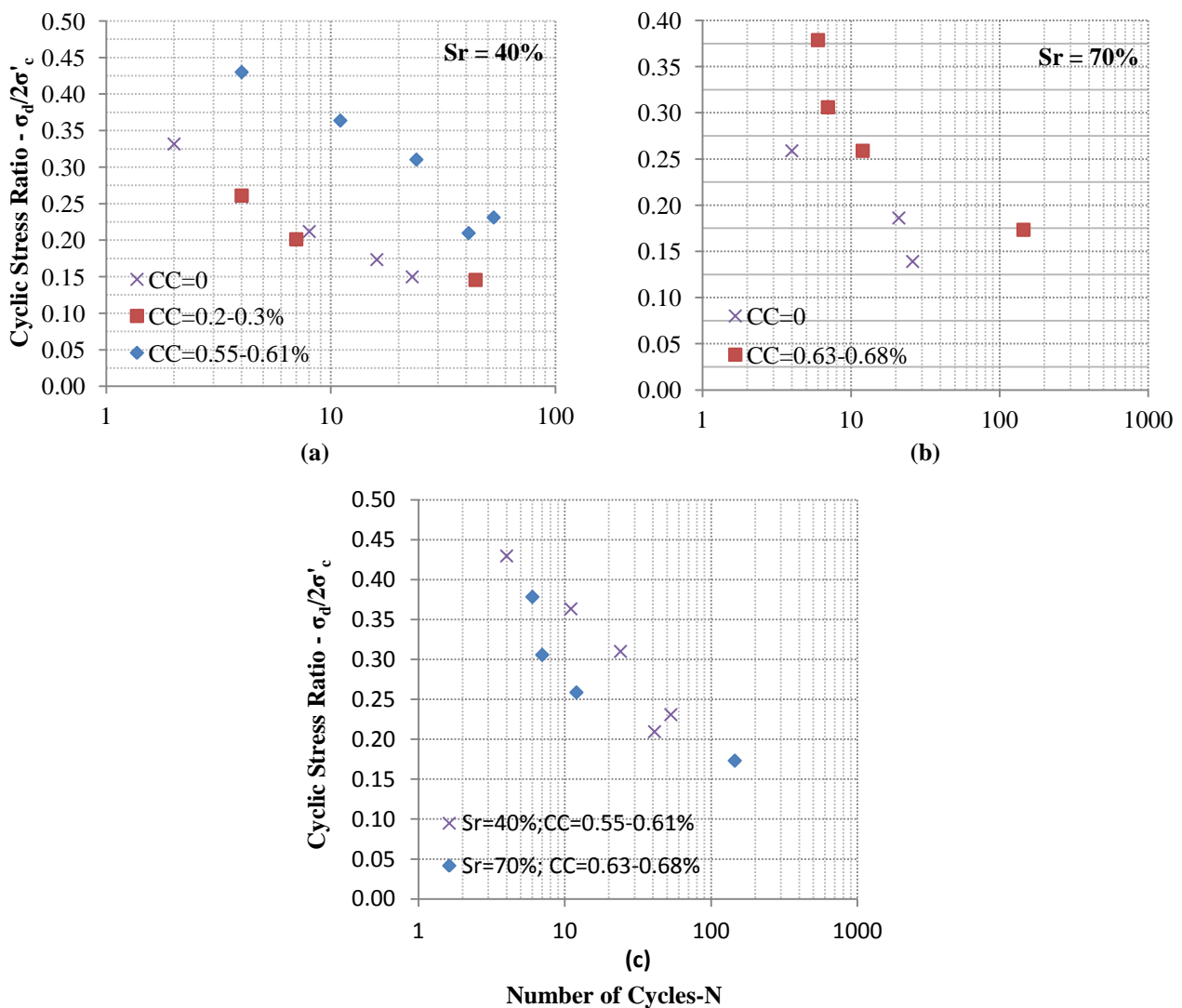


Figure 3 Liquefaction Strength Curve of Treated Sand

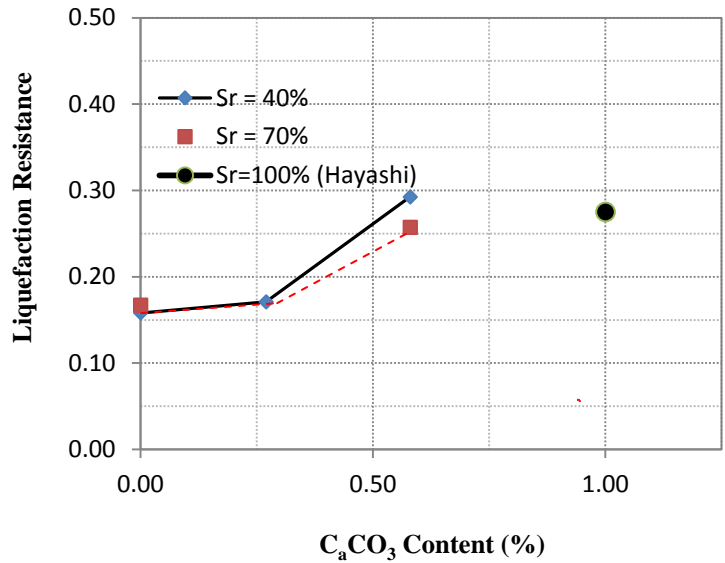


Figure 4 Alteration of Liquefaction Resistance due to the Change in Calcite Content

Shear Modulus at Small Strain Level

Shear modulus at small strain level was observed in prior to the liquefaction tests. Fig. 5 depicts shear modulus (G) for all the specimens tested in this study. Also shown in the figure is the relationship between G and shear strain of unimproved Toyoura sand at $Dr = 70\%$. Shear modulus of sand is approximately constant in the strain level of 10^{-6} and 10^{-5} . Although the range of shear strain observed in this study was 10^{-5} , the shear modulus was significantly varied. Similar to the liquefaction resistances, the shear modulus increased with increasing CC and decreasing Sr.

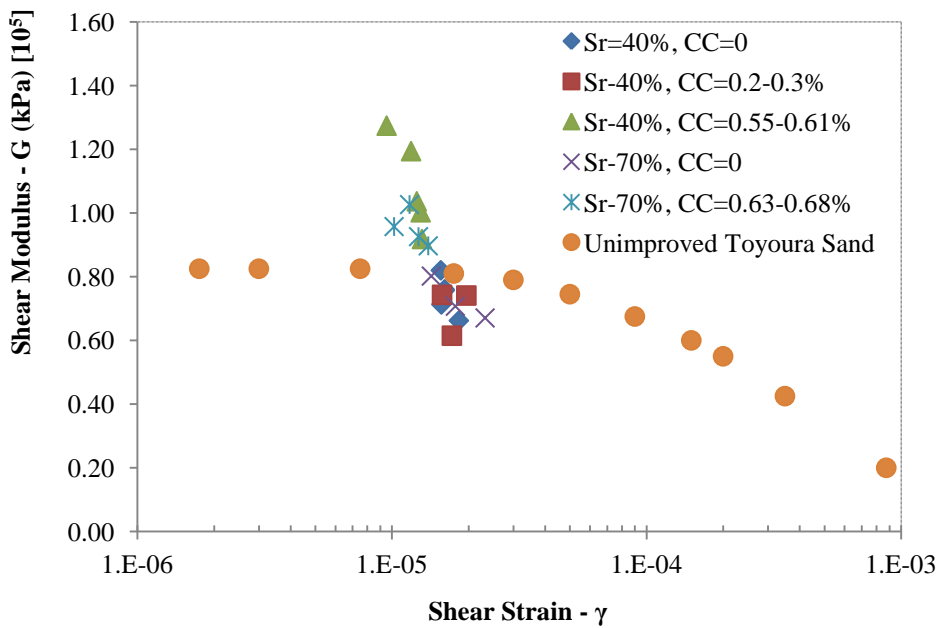


Figure 5 Shear Modulus Variations with Shear Strain

CONCLUSION

A series of undrained cyclic triaxial tests on sand improved with calcite precipitation has been conducted to study effects of calcite precipitation on dynamic properties of sand. Special attention was paid to the degree of saturation during curing the specimens. Two parameters selected as testing variables in this study are degree of saturation and calcite content. The main finding from the experiment are summarized as follows:

- a. The existence of calcite content in sand sample enhance the liquefaction resistance as well as shear modulus at small strain level. This is occurred due to the restriction of buildup excess pore water pressure leading to liquefaction.

- b. Cyclic stress ratio grows up due to the compound of calcite content and the decrease of saturation level in the sand sample. The bigger level of calcite and the lower the degree of saturation in the sand samples bring them stronger. This is probably due to the fact that precipitated calcite tends to concentrate to the contact points between sand grains as degree of saturation decreases. This hypothesis, however, have to be confirmed by observing grain surface. Detailed observation with SEM will be reported elsewhere in the near future.

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