

EFFECT ON UNCONFINED COMPRESSIVE STRENGTH OF SAND TEST PIECES CEMENTED WITH CALCIUM PHOSPHATE COMPOUND

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ABSTRACT: Chemical grout is composed of a calcium phosphate compound (CPC) which develops to form calcium carbonate (CC) precipitation throughout the soil and leading to an increase in soil strength. In this paper, initially the condition for CPC precipitation by using different mixtures of calcium and phosphate stock solutions were investigated and analyzed. For that, Toyoura sand test pieces were cemented by CPC solutions and cured up to 28 days and carried out unconfined compressive strength (UCS) test. Moreover, Toyoura sand test pieces were cemented by CPCs with scallop shell (SS) powder and cured and these specimens also analyzed with UCS tests. The UCS of the sand test pieces cemented by CPC with SS powder was larger than that of the test pieces with no added powders. The UCS of Toyoura sand test piece cemented with the CPC-SS powder method increased to a maximum of 156.9 kPa. Moreover, the best CPC-Chem mixture for cementation is CA: DPP with the concentration of Ca/P ratio is 0.5. In addition pH concentration, scanning electron microscope (SEM), and density before and after curing were observed. The results indicate that the density and the pH concentration of the sand test pieces cemented by CPCs with SS powder were larger than that of the sand test pieces with no added powders. SEM images of test pieces cemented with CA: DPP mixture by addition of SS powders not clearly identified a crystal formation among particles of Toyoura sand.

Keywords: Calcium Carbonate; Calcium Phosphate Compound; Ground Improvement; Unconfined Compressive Strength

1. INTRODUCTION

Cement grouting is used as a ground improvement method for countermeasure against soil liquefaction during an earthquake [1]. However, cement grouting comprises several environmental problems such as high CO₂ emissions during cement production, high energy cost for re-excavation and hard to recycling the improved ground. Hence in present, grout materials produce by microorganism have been developed for ground permeability control and reinforcement [2]-[6]. Biogrout is the process of ground improvement by biological action [7].

A novel ground stabilizer was developed to increase the number of options available among cementing mechanisms based on microorganisms [8],[9]. In addition, it is reported on a CPC chemical grout (CPC-Chem) that utilizes self-setting CPC mechanisms [10] and on a CPC biogrout (CPC-Bio) whose solubility is dependent on its pH [10] (Fig. 1), which can be increased by a microbial reaction.

CPC-Chem is easy to obtain, safe to handle, non-toxic, and recyclable, advantages that make it suitable for geotechnical application [8]. The maximum unconfined compressive strength (UCS) of sand test pieces cemented with CPC-Chem was found to be 63.5 kPa [8]. Our aim is to achieve a UCS value of

100 kPa, which is needed to avoid ground liquefaction during earthquakes [11]. This implies that the UCS of CPC-Chem is not sufficient for use as a ground stabilizer, necessity of a preferable mechanism for further increase in UCS.

An earlier research was said, the UCS of the test pieces with TCP and CC additives exceeded the targeted value of 100 kPa and increased to a maximum of 261.4 kPa and 209.7 kPa respectively [12]. This observations indicates that the existence of CC seed crystals can reinforce the strength of CPC grouts. The main component of scallop shells is CC, which are disposed of in large quantities as marine industrial waste (410,000 tons/year in Japan) [13]. Scallop shells are non-toxic to handle and inexpensive to obtain. Hence, scallop shells can be used for CC which is a favorable material in the geotechnical field from the view point of waste utilization and costeffectiveness.

In the present study, our aim was to improve strength by adding CPC with scallop shell powder. This study aims to exceed a maximum UCS of 100 kPa after 28 days of curing, which is the strength required to use the CPC and scallop shell powder combination as a countermeasure against soil liquefaction during earthquake. UCS tests and scanning electron microscopy (SEM) observations

were conducted on sand test pieces as a function of time. In addition, pH value and wet density were measured. Based on the results, we discuss the effect of the amount of added powders, variation in wet density (ρ_i), pH and crystal form on the UCS.

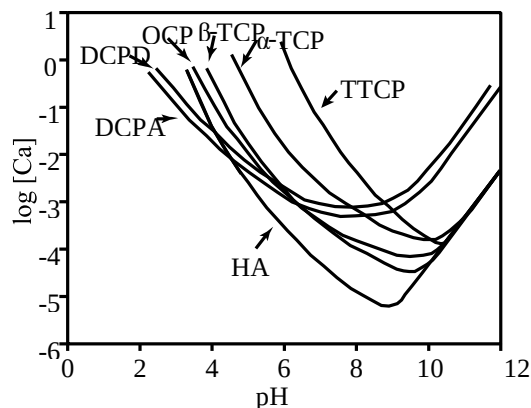


Fig. 1 Solubility phase diagrams for the ternary system, $\text{Ca}(\text{OH})_2\text{-H}_3\text{PO}_4\text{-H}_2\text{O}$, at 25 °C, showing the solubility isotherms of CaHPO_4 (DCPA), $\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$ (DCPD), $\text{Ca}_8\text{H}_2(\text{PO}_4)_6 \cdot 5\text{H}_2\text{O}$ (OCP), $\alpha\text{-Ca}_3(\text{PO}_4)_2$ (α -TCP), $\beta\text{-Ca}_3(\text{PO}_4)_2$ (β -TCP), $\text{Ca}_4(\text{PO}_4)_2\text{O}$ (TTCP), and $\text{Ca}_{10}(\text{PO}_4)_6 \cdot (\text{OH})_2$ (HA). The figure is adapted from Tung [10].

2. METHODOLOGY

In this study, for the CPC-Chem, calcium acetate (CA) was used as a calcium solution and diammonium phosphate (DAP) and dipotassium phosphate (DPP) were used as phosphate solutions. The concentration of CA was varied from 1.5 M to 0.5 M and the concentration of DAP and DPP was constant as 3.0 M for prepare the CPC-Chem solution with two different Ca/P ratios (0.5 and 0.25). Then, Toyoura sand test pieces were cemented with CPC-Chem only and cured up to 28 days and analyzed with UCS tests. For this case, a standard sand test piece was made from 320.09 g of Toyoura sand (mean diameter $D_{50} = 170 \mu\text{m}$, 15% diameter $D_{15} = 150 \mu\text{m}$) and 73.3 mL of CPC-Chem according to the previous report [8].

In addition, Toyoura sand test pieces were cemented with CPC-Chem (CA: DPP = 0.6 M: 1.2 M) by addition of scallop shell (SS) powder and cured up to 56 days and these specimens also analyzed with UCS tests. In this case, a standard sand test piece was made as same as previous case and it is called as 'CPC-Cont' sample. The examined test pieces were made with the combination ratios shown in Fig. 2. 1% (3.2 g) (Case SS-01), 5% (16.0 g) (Case SS-05), and 10% (32.0 g) (Case SS-10) of SS (mean diameter $D_{50} = 25.12 \mu\text{m}$) were mixed with 72.21 mL, 67.84 mL, and 62.38 mL of CPC-Chem respectively and added

to weight of a standard sand test piece of 320.09 g for prepare CPC-SS samples.

It was uniformly mixed in a stainless-steel ball for 2 min and the mixture was divided into quarters, each of which was placed into a plastic mold container ($\phi = 5 \text{ cm}$, $h = 10 \text{ cm}$). The sand in the mold container was tamped down 30 times by a hand rammer after each of the four quarters was placed in the mold. The molded test pieces were subsequently cured in an airtight container at a high humidity for 28 days and 56 days at 20°C.

The UCS of the test pieces removed from the mold container after curing was measured at an axial strain rate of 1%/min with the UCS apparatus T266-31100 (Seikensha Co., Ltd., Japan). In all cases two test pieces were tested. The pH of the test pieces was calculated as an average of three measurements (top, bottom, and middle of each test pieces) using pH Spear (Eutech Instruments Pte., Ltd., Singapore). Segments of the UCS test pieces were observed by an SEM. The segments were naturally dried at 20°C for a few days and carbon-coated with a carbon coater. SEM observations were carried out at an accelerating voltage of 15 kV and at x 1000 magnification.

3. RESULTS AND DISCUSSION

3.1 UCS Test

In this study, Toyoura sand test pieces cemented by four reaction mixture sets of CPC-Chem were chosen. The measured UCS in this study ranged from 26.1 to 143.6 kPa. The maximum value was measured when the sand test pieces cemented with CA: DPP=1.5M: 3.0M mixture and Ca/P ratio was 0.5 (Fig. 3 (a)). The UCS is tended to increase with the curing time for 28 days for sample prepared with CA: DPP=1.5M: 3.0M mixture but the test piece at 14 days of curing time shows that the UCS is tended to decrease. It is assumed that some error could occur when preparing the sample. To clarify this result, further examination of the test pieces is needed in the future. There is no significant variation of UCS in the test pieces prepared with CA: DPP=0.75 M: 3.0 M mixture. Also, the UCS value of the test pieces cemented with CA: DPP=1.5M: 3.0M mixture (CA: DPP=0.5) is larger than the UCS value of the test pieces with CA: DPP=0.75M: 3.0M mixture (CA: DPP=0.25).

According to Fig. 3(b), the value of UCS is tended to constant for both CA: DAP=0.5 and CA: DAP=0.25. Also, the UCS value of the test pieces with CA: DAP=0.5 is larger than the UCS value of the test pieces with CA: DAP=0.25. Moreover, from the Figs. 3(a) and 3 (b), the UCS value of the test pieces with CA: DPP=0.5 is larger than the UCS value of the test pieces with CA: DAP=0.5.

Case Name	Control	CPC-SS Method		
	CPC-Cont	SS-01	SS-05	SS-10
Sand Weight (g)	Sand 320.09	Sand 320.09	Sand 320.09	Sand 320.09
Weight of adding powders (g)	Without powder	+	+	+
		SS 3.2	SS 16.0	SS 32.0
Volume of CPC- Chem (mL)	73.30	72.21	67.84	62.38

Fig.2 Conceptual image of the contents.

Figs. 4(a) and 4(b) show the relationship between UCS and Ca/P ratio for the sand test pieces cemented with CPC-Chem only. The results are given considering after 1 day and 28 days curing period. It comprises that the UCS value (Ca/P=0.5) is larger than the UCS value (Ca/P=0.25) for both testing cases such as CA: DPP and CA: DAP. The UCS value is larger after 28 days curing time than 1 day curing time. However, the rate of increase of UCS of sand test pieces cemented with CA: DPP mixture is larger in 28 days curing period than 1 day curing period (Fig. 4(a)).

In Fig. 4(b), when the test pieces cemented with CA: DAP mixture, the UCS values are nearly same after 1 days and 28 days curing period. Therefore, the rate of increase of UCS of sand test pieces cemented with CA: DAP solution is larger in 1 day curing period than 28 day curing period. However, it is assumed as an error and to get more precise result, further testing is required.

In addition, Toyoura sand test pieces cemented by four reaction mixture sets were chosen (adding CA: DPP = 0.6M: 1.2M mixture with no adding powders

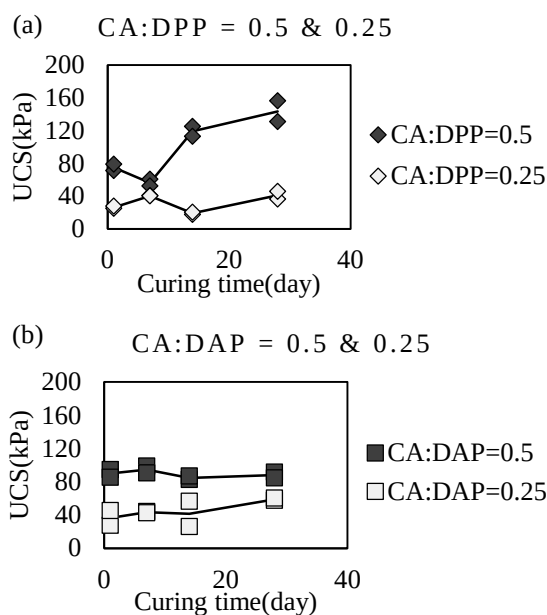


Fig. 3 (a) UCS of Toyoura sand test pieces cemented by CA: DPP=0.5 and 0.25. (b) UCS of Toyoura sand test pieces cemented by CA: DAP=0.5 and 0.25.

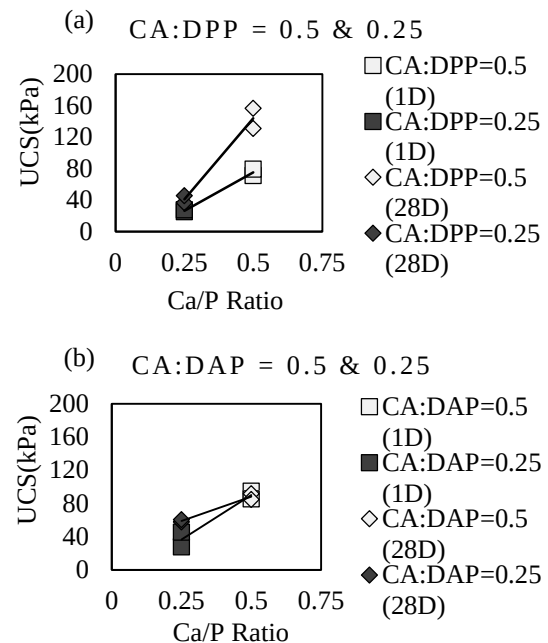


Fig. 4 Relationship between UCS and Ca/P ratio of Toyoura sand test pieces cemented by (a) CA: DPP=0.5 and 0.25. (b) CA: DAP=0.5 and 0.25.

and adding SS powder with different percentages: 1%, 5% and 10%). The measured UCS in this study ranged from 49.9 to 156.9 kPa.

The maximum value was measured when the sand test pieces cemented with CA: DPP=1.2M: 0.6M mixture by adding SS powder 10% (Fig. 5). The UCS of sand test pieces cemented by CPC with SS powder was larger than that of the test pieces with no added powders when considering curing time until 28 days. However, the UCS values of sand test pieces cemented by CPC with SS-1% and SS-5% after 56 days curing period is less than that of the CPC-Cont sample.

The test pieces with SS-10% show that the UCS is tended to decrease at 14 days curing period, it is assumed that some error could occur when preparing sample. To clarify this result, further examination of the test pieces is needed in the future.

3.2 Effect of pH on UCS

Figs. 6(a) and 6(b) illustrate the effect of pH on UCS for the test pieces cemented by CPC-Chem (CA: DPP and CA: DAP). The pH of the test pieces range from acidic to weakly alkaline (6.4-7.5) for CA: DAP and the pH range from weakly alkaline to strong alkaline (7.0-8.7) for CA: DPP. Moreover the UCS is tended to increase with the increase of pH for CA: DPP (Fig 6 (a)) while the UCS was nearly constant when increase of pH for CA: DAP (Fig. 6(b)).

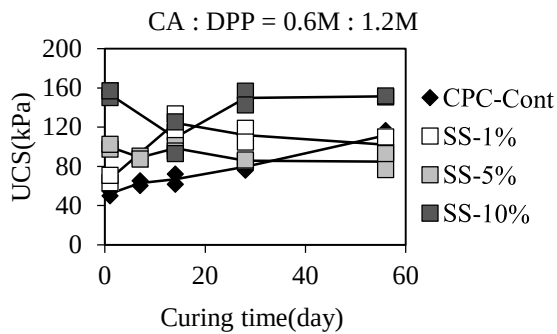


Fig. 5 Unconfined compressive strength (UCS) of sand test pieces cemented by CPC with SS.

According to Fig. 7, the pH is tended to increase with the time. Moreover, the pH value of CPC-SS sample is larger than pH values of CPC-Cont sample. When the concentration of SS powder is increased, pH value is tended to increase. Although, when pH is increased, UCS is tended to increase.

3.3 SEM Observation

Fig. 8 shows SEM images of sand test pieces cemented with four reaction mixture set of CPC-Chem (CA: DPP=0.5 and 0.25 and CA: DAP=0.5 and 0.25) and the curing time was 14 days. Wisker-like crystal structure was observed in the sample prepared

with CA: DPP=0.5 mixture and other samples were not clearly observed any crystal formation.

SEM images of test pieces subjected to CA: DPP treatment with SS powders were not clearly identify a crystal formation among particles of Toyoura sand (Fig. 9). The increase in UCS seemed to be because of the binding of the sand particles by the precipitated CPC that enveloped the CC particles.

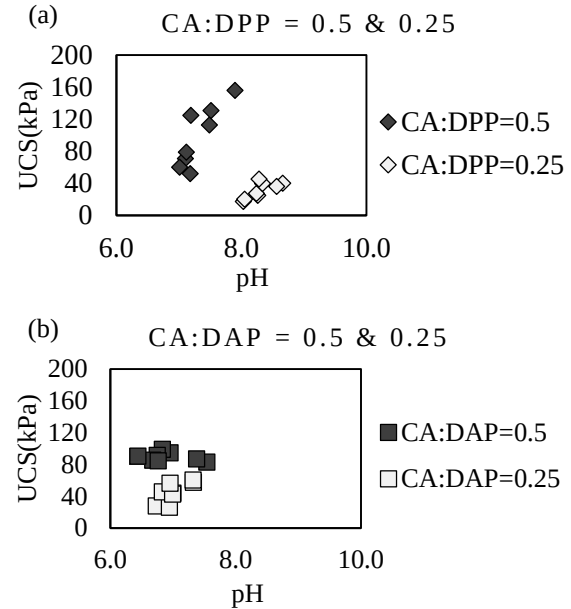


Fig. 6 (a) Relationship between pH and UCS for CA: DPP=0.5 and 0.25 (b) Relationship between pH and UCS for CA: DAP=0.5 and 0.25.

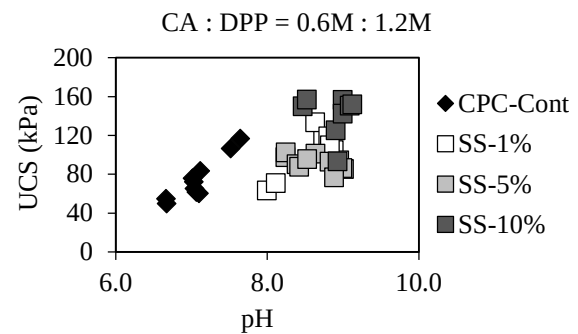


Fig. 7 Relationship between pH and UCS for CPC-Cont, and CPC-SS test pieces.

3.4 Effect of Wet Density on UCS

The estimated wet density of the test pieces is shown in Figs. 10 and 11. Wet density (before) was calculated by measuring mold weight immediately after being produced and wet density (after) is defined as wet density immediately after cured. Fig. 10 shows wet density of the test pieces cemented with CPC-Chem only. The results shows, UCS value is increased with increase of density only in CA: DPP=0.5. In addition, when curing time is increased

the UCS is tended to increase. In other cases, the UCS value neither increase nor decrease with increase of wet density.

There is no significant difference between wet density, before and after the test. That is because there was no volume change such as expansion or shrinkage after curing.

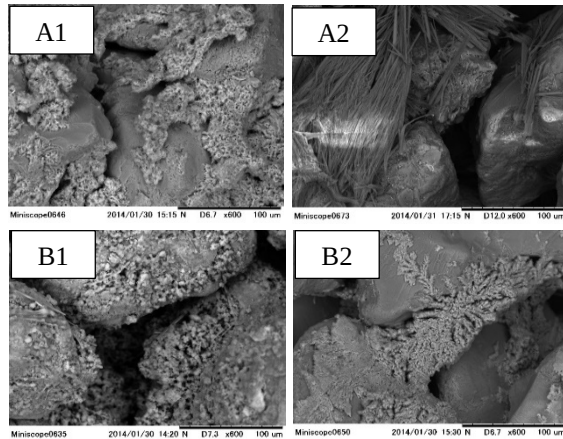


Fig.8 SEM images for test samples after 14 days curing period (600 X). (A1) CA: DPP=0.25, (A2) CA: DPP=0.5, (B1) CA: DAP=0.25 and (B2) CA: DAP=0.5.

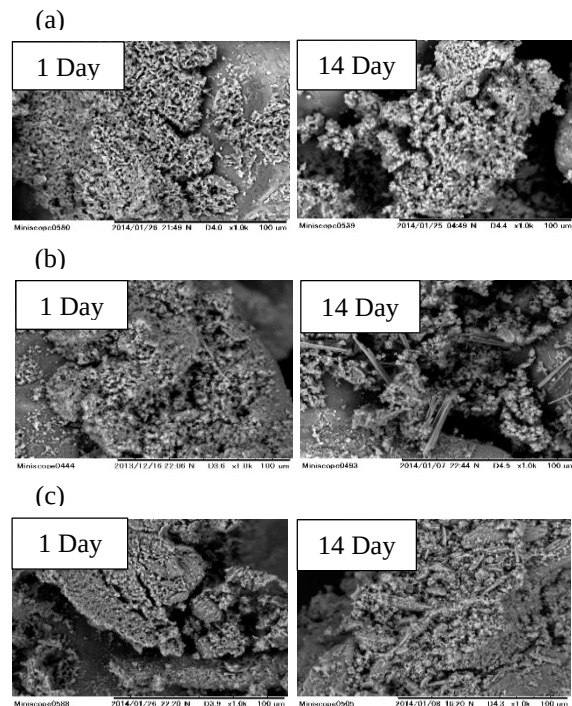


Fig.9 SEM images for test samples cemented with CPC by adding SS powder after 1 day and 14 days curing period (1000 X). (a) SS-1%, (b) SS-5% and (c) SS-10%.

In the case of the test pieces treated by the CPC-SS powder method, the wet density is larger than the test pieces cemented with CPC-Chem only. (Fig. 11). Also, it comprises that the UCS is tended to increase with increase of density. In addition, when the

increase of SS and CC powders %, wet density is increased.

3.5 Effect on Addition of SS Powder on the UCS

Figs. 3 and 5 show the UCS test results for Toyoura sand test pieces cemented with CPC-Chem only and CPC-SS powder respectively. It comprises that the UCS of test pieces cemented with CPC-SS is larger than that for CPC-Chem alone. Practically, the test pieces to which the SS powder with 10% was added show a UCS is larger than 100 kPa. This statement recommends that through control of the CC content, the CPC-SS and CPC-CC method would allow for adjustment of strength according to the required strength properties of the ground while maintaining a UCS of over 100 kPa. Considering results of UCS, pH, wet density and SEM images in CPC-SS powder method, the governing factors for increase the strength of the sample are pH and wet density.

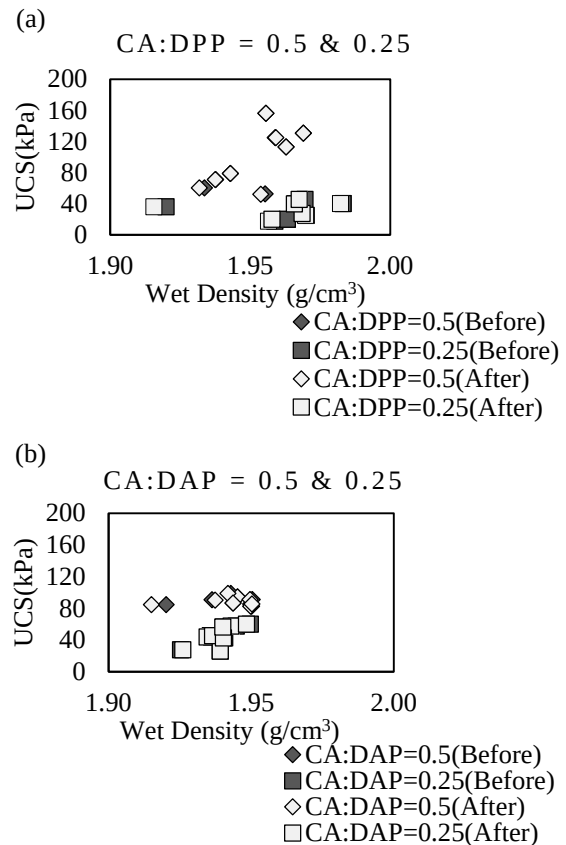


Fig. 10 Relationship between wet density and UCS (a) CA: DPP=0.5 and 0.25 (b) CA: DAP=0.5 and 0.25.

4. CONCLUSION

In this study, initially the condition for CPC precipitation by using different mixtures of calcium and phosphate stock solutions was investigated and analyzed. It was concluded that the best CPC-Chem

mixture was CA: DPP with the concentration of Ca/P ratio is 0.5.

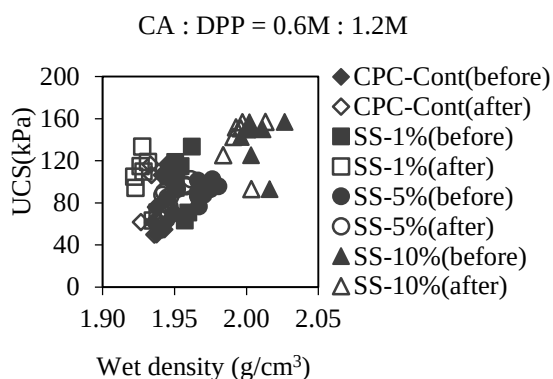


Fig. 11 Wet density of Toyoura sand test pieces cemented by CPC with SS.

Further, the study was expanded to CPC powder method with using scallop shell powder. From this study, it was concluded that CPC-SS powder method had a significant potential for strength of the soil. The UCS of a Toyoura sand test piece cemented with the CPC-SS powder method increased to a maximum of 156.9 kPa. The aim of this study was to use CPC-Chem to achieve a maximum UCS of over 100 kPa, which was the strength required to prevent ground liquefaction. Using the CPC-SS powder method, we far exceeded this objective by achieving a UCS of over 150 kPa.

Considering cost effectiveness of preparing samples, CPC solutions are very expensive. When the samples prepared with only adding CPC-Chem, it was need to increase the concentration of calcium and phosphate solutions (CA: DPP=1.5M: 3.0M) to achieve our aim. However, we got the more than targeted strength by using smaller amount (CA: DPP=0.6M: 1.2M) of CPC by addition of SS powder and the cost was low. Furthermore, scallop shells can be found very easily.

Finally, considering above viewpoints, CPC-SS powder method is more effective and reliable than CPC-Chem method for achieve a maximum UCS of over 100 kPa to prevent the ground liquefaction.

5. ACKNOWLEDGEMENT

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6. REFERENCES

- [1] Karol RH, "Chemical grouting and soil stabilization", 3rd ed., CRC Press, Boca Raton, FL, 2003.
- [2] Whiffin VS, Van Paassen LA and Harkes MP, "Geomicrobiology Journal", Vol. 24, 2007, pp. 417-423.

- [3] De Muynck W, De Belie N, and Verstraete W, "Ecological Engineering", Vol. 36, 2010, pp. 118-136.
- [4] DeJong JT, Mortensen BM, Martinez BC and Nelson DC, "Ecological Engineering", Vol. 36, 2010, pp. 197-210.
- [5] Harkes MP, Van Paassen LA, Booster JL, Whiffin VS and Van Loosdrecht MCM, "Ecological Engineering", Vol. 36, 2010, pp. 112-117.
- [6] Kawasaki S, Ogata S, Hiroyoshi N, Tsunekawa M, Kaneko K. and Terajima R, "Effect of temperature on precipitation of calcium carbonate using soil microorganisms", Journal of the Japan Society of Engineering Geology, Vol. 51, 2010, pp. 10-18 (in Japanese with English abstract).
- [7] Van Paassen LA, Harkes MP, Van Zwieten GA, Van der Zon WH, Van der Star WRL and Van Loosdrecht MCM, "Conference Proceedings", in Proc. 17th Int. Conf. on Soil Mechanics and Geotechnical Engineering, 2009, pp. 2328-2333.
- [8] Kawasaki S. and Akiyama M., "Unique grout material composed of calcium phosphate compounds", Int. Journal of GEOMATE, Vol. 4, No.1, 2013, pp. 429-435.
- [9] Akiyama M. and Kawasaki S, "Microbially mediated sand solidification using calcium phosphate compounds", Journal of the Japan Society of Engineering Geology, Vol. 137-138, 2012, pp. 29-39.
- [10] Tung MS, "Calcium phosphates: structure, composition, solubility, and stability, in: A. Zahid (Ed.), Calcium phosphates in biological and industrial systems", Kluwer Academic Publishers, Norwell, 1998, pp. 1-19.
- [11] Yamazaki H, Maeda K, Takahashi K, Zen K and Hayashi K, "Technical Note of Port and Harbour Research Institute", Vol. 905, 1998, pp. 1-29 (in Japanese).
- [12] Kawasaki S. and Akiyama M, "Enhancement of unconfined compressive strength of sand test pieces cemented with calcium phosphate compound by addition of various powders", Journal of Soils and Foundations, Vol. 53-(6), 2013, pp. 966-976
- [13] "Recycling Technology Guidelines for Harbor and Airport Construction and Maintenance", Edited in 2004, Ports and Harbours Bureau. <http://www.mlit.go.jp/kowan/recycle/>.

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