TITLE

Improvement in the unconfined compressive strength of sand test pieces cemented with calcium phosphate compound by addition of calcium carbonate

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ABSTRACT

We added calcium carbonate (CC) powder to a novel grout made from calcium phosphate compound (CPC-chem) to increase the ground strength improvement afforded by CPC-chem. We conducted the unconfined compressive strength (UCS) test and scanning electron microscopy (SEM) observation on test pieces cemented with CPC-chem and CC powder. The UCS of test pieces cemented with CPC-chem and CC powder was significantly higher than that of test pieces cemented without CC powder, and it reached a maximum of 209.7 kPa. The UCS of test pieces cemented with CC powder and deionized water was 12.5 kPa, which was similar to that of test pieces cemented with deionized water only (10 kPa). SEM observation revealed mesh-like and three-dimensional structures in the segment of the test piece cemented with CPC-chem and 1 wt% of CC, which showed UCS of over 200 kPa and the minimum axial strain rate among all cases in this study. These results suggest that the addition of CC powder significantly enhances the ground improvement afforded by CPC-chem.

KEYWORDS

Biogrout, Calcium carbonate, Calcium phosphate compound, Ground improvement, Seed crystal, Unconfined compressive strength

1. INTRODUCTION

Cement-based hardener is a common material for ground improvement by consolidation and is available in several different varieties (Karol, 2003). On the other hand, in recent years, a new geotechnical method has been developed that involves the use of microorganisms for ground permeability control and reinforcement (e.g. Harkes et al., 2010). The process of ground improvement by biological action is called "biogrouting" (Van Paassen et al., 2009). We are developing a novel ground stabilizer to increase the number of options available among cementing mechanisms based on microorganisms (Akiyama and Kawasaki, 2012a, 2012b).

Further, we have reported on a CPC chemical grout (CPC-chem) that utilizes self-setting CPC mechanisms (Tung, 1998), and on a CPC biogrout (CPC-bio) whose solubility is dependent on its pH (Tung, 1998), which can be increased by a microbial reaction. Our aim was to achieve a UCS value of 100 kPa, which is needed to avoid liquefaction during earthquakes (Yamazaki et al., 1998). CPC-chem is easy to obtain,

safe to handle, non-toxic, and recyclable, advantages that make it suitable for geotechnical application (Akiyama and Kawasaki, 2012a). The maximum unconfined compressive strength (UCS) of sand test pieces cemented with CPC-chem was 63.5 kPa (Akiyama and Kawasaki, 2012a). When the CPC-chem was converted to CPC-bio by the addition of microorganisms and an ammonia source, the UCS increased from 42.9 kPa to 57.6 kPa (Akiyama and Kawasaki, 2012b). These results imply that the UCS of both CPC-chem and CPC-bio is sufficient to enable their use as a ground stabilizer, although a further increase in UCS would be preferable.

In the field of medical and dental science, CPC paste is used as a bone graft material and for hardening teeth on the basis of the mechanism of hydroxyapatite (HA) precipitation (Chow, 1991). Previous research on CPC paste has showed that the compressive strength of a mixture paste of dicalcium phosphate and α-tricalcium phosphate reached can be increase from 35 MPa to a maximum of 56 MPa by using calcium carbonate (CC) as the seed crystal (Fernández et al., 1998). This observation indicates that the existence of CC seed crystals can reinforce the strength of CPC grouts, such as the grout used in this study. CC is the main component of oyster and scallop shells, which are disposed of in large quantities as marine industrial waste (410,000 tons/year in Japan; Ports and Harbours Bureau, 2004). CC is also the main component

of limestone. Moreover, it is non-toxic to handle and inexpensive to obtain. Thus, CC is a promising material in the geotechnical field from the viewpoint of waste utilization and cost effectiveness.

Therefore, we sought to improve the performance of CPC-chem by mixing it with CC powder. This study aims to exceed a maximum UCS of 100 kPa, which is the UCS required to enable the use of CPC-chem as a countermeasure against ground liquefaction (Yamazaki et al., 1998).

2. MATERIALS AND METHODS

The CPC-chem used in this study was a 1.5 M:0.75 M mixture of diammonium phosphate (DAP) and calcium acetate (CA); we used this mixture because it has previously been reported that this mixture yields the highest UCS among all combination ratios of DAP with calcium nitrate or CA (Akiyama and Kawasaki, 2012a).

A standard sand test piece was made from 320.09 g of Toyoura sand (mean diameter D_{50} = 170 μm, 15% diameter D_{15} = 150 μm) and 73.3 mL of CPC-chem according to the previous report (Akiyama and Kawasaki, 2012a), and the examined test pieces were made with the combination ratios and wet density shown in Fig. 1. 1% (3.2 g) (Case CC-01), 5% (16.0 g) (Case CC-05), and 10% (32.0 g) (Case CC-10) of CC (mean

diameter D_{50} = 17.1 μm, 85% diameter D_{85} = 23.9 μm) was added to 316.89 g, 304.09 g, and 288.09 g of Toyoura sand, respectively, each time yielding the weight of a standard sand test piece of 320.09 g. Furthermore, CPC-chem was added to the mixture of sand and CC. 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 (φ = 5 cm, h = 10 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 at 20 °C. The control samples were test pieces cemented with only deionized water (Case DW-Cont), with only CPC-chem (Case CPC-Cont), and with deionized water and CC (Case CC-Cont). Hereafter, the method of improving ground strength by adding CC powder to CPC-chem is referred to as the CPC-CC method. 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 DW-Cont and CC-Cont, only one test piece was tested, whereas in the other 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 pHSpear (Eutech Instruments Pte., Ltd., Singapore).

Segments of the UCS test pieces were observed by an SEM (SuperScan SS-550, Shimadzu Corporation, Kyoto). The segments were naturally dried at 20 °C for a few days and carbon-coated with a carbon coater (Quick Carbon Coater SC-701C, Sanyu Electron Co., Ltd., Tokyo).

3. RESULTS AND DISCUSSION

3.1. Unconfined compressive strength

The results for the UCS test are shown in Fig. 2. The UCS values of the test pieces treated with the CPC-CC method were larger than those of the controls (DW-Cont, CPC-Cont, and CC-Cont). In particular, the UCS values of CC-01 and CC-10 exceeded 200 kPa, which is four times that CPC-Cont. This means that the addition of CC powder achieved the goal of this study.

The stress (σ)–strain (ε) curves of all cases are shown in Fig. 3. All the test pieces to which CC powder was added (CC-01, CC-05, and CC-10) showed a UCS greater than 150 kPa (Fig. 2); moreover, as the CC content increased, the compressive strain increased and the Young's modulus decreased (Fig. 3). This observation suggests that through the control of the CC content, the CPC-CC method would allow the adjustment

of hardness according to the required mechanical/deformation property of the ground while maintaining the UCS at over 150 kPa.

3.2. SEM and EDX observations

The crystal structures observed in CPC-Cont and CC-Cont were whisker-like and cuboid-like, respectively. In contrast, CC-01 had a mesh-like three-dimensional structure, in which the precipitated CPC that enveloped the CC particles bonded with the surface of the sand particles; such bonding was also observed in CC-05 and CC-10, but without the formation of any crystal structure. The increase in UCS seemed to be because of the binding of the sand particles by the precipitated CPC that enveloped the CC particles. The difference between the failure strain (Fig. 3) of CC-01, with its mesh-like structure (Fig. 4C), and CC-05/CC-10, which did not show any crystal structure (Fig. 4D and 4E), suggests that crystal structure of the CPC-CC in the sand test pieces affects the mechanical/deformation property of the test pieces.

Elemental mappings of Ca and P showed similar distributions on a background of Si (mainly sand particles) (Fig. 4). The analysis revealed that the improvement in UCS afforded by the CPC-CC method was because of the filling to the voids between sand particles and the uniting of the particles of cement material comprising Ca and P.

3.3. Wet density and cement material mass

The wet density of the test pieces is provided at the bottom of Fig. 1. Since the density of CC powder (2.93 g/cm^3, Chemical Book Web site, 2012) was greater than that of Toyoura sand (1.65g/cm^3), the density of the test pieces would increase with the mass% of CC powder in the test pieces; we expected that the increase in density would result in an improvement in UCS. However, CC-Cont (ρ_t = 1.936 g/cm^3) test pieces showed a UCS of 12.5 kPa, which was similar to that (10.0 kPa) of DW-Cont test pieces (ρ_t = 1.831 g/cm^3) (Fig. 1). In the case of the test pieces treated by the CPC-CC method, the increase in CC content increased the filling of voids between sand particles because of the increase in wet density; however, even in this case, there was no increase in the UCS with wet density.

3.4. pH of test pieces

The test pieces with added CC showed a pH of around 8 (Fig. 2). Considering that the solubility of CPC is dependent on its pH (Tung, 1998), we can utilize the mechanism of CPC-bio (Akiyama and Kawasaki, 2012b) to increase the CPC precipitation; this would be achieved by using microorganisms and ammonia sources to increase the pH to 9 via

a pH-increasing reaction and would result in a further increase in UCS. In a future study, we intend to report on the effect of CC addition on the UCS of sand test pieces cemented with CPC-bio.

3.5. Potential applications of the CPC-CC method

The CPC-chem used in this study has a viscosity similar to grouts with a high concentration of silicate and might show effective penetrability for soil types ranging from medium sand to fine gravel (Karol, 2003; Akiyama and Kawasaki, 2012a). In addition, the groutability (N) (Akbulut and Saglamer, 2002) of CC powder in relation to Toyoura sand was estimated to be $N = (D_{15})_{\text{Toyoura sand}} / (D_{85})_{\text{CC powder}} = 4.3 < 11$. Although the CC powder (Chemicals, 2010) used in this study did not satisfy the groutability requirements ($N > 25$, sufficiently injected; $N < 11$, not sufficiently injected) (Akbulut and Saglamer, 2002), the CPC-CC method is expected to be practicable if CC powder with a smaller particle size is used for sandy ground that has particles larger than those of Toyoura sand.

In addition, the CPC-CC method can be applied to the sand compaction pile (SCP) method and the deep mixing method intended for soft ground. For example, simply considering the application to SCP, the results of this study mean that the ground

improvement using CPC-CC method can be 20 times of UCS in comparison to that of sand only, which is same situation of DW-Cont in this study.

Furthermore, unlike ground treated with a cement-based hardener, ground treated with the CPC-CC method can be re-excavated and recycled. This means that the CPC-CC method can be used as a temporary supplemental hardening method. For conducting a practical-scale experiment on actual ground, we plan to investigate the temporal variation in the UCS of test pieces, the relation between the crystal form and the mechanical/deformation property of the test pieces, and other parameters for evaluating the ground reinforcement.

4. CONCLUSIONS

We observed the effect of the addition of CC to CPC-chem on the UCS of test pieces cemented with CPC-chem. The results confirm the prospect that the addition of CC increases the UCS of the test pieces and may apply to CPC-bio.

Furthermore, CC is a promising material in the geotechnical field from the viewpoint of waste utilization and cost effectiveness. Hence, the CPC-CC method has the potential to be utilized as a non-contaminating and recyclable method for ground reinforcement that can satisfy the strength requirements for actual ground while avoiding the problems

of existing cement-based hardener.

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FIGURE CAPTIONS

Figure 1. Conceptual image of the contents and wet density of test pieces.

Figure 2. UCS and pH of test pieces.

Figure 3. Stress (σ)–strain (ε) curves of all cases.

Figure 4. SEM and EDX images of test pieces (2000×). White dots in the elemental mapping represent the distribution of Si, Ca, and P in each EDX image.

Figure 1

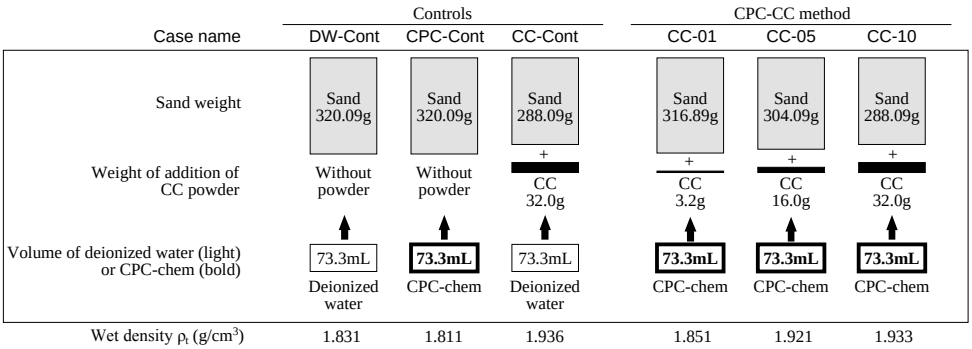

Controls
CPC-CC method
Case name
DW-Cont
CPC-Cont
CC-Cont
CC-01
CC-05
CC-10
Sand weight
Sand 320.09g
Sand 320.09g
Sand 288.09g
Sand 316.89g
Sand 304.09g
Sand 288.09g
Weight of addition of CC powder
Without powder
Without powder
+ CC 32.0g
+ CC 3.2g
+ CC 16.0g
+ CC 32.0g
Volume of deionized water (light) or CPC-chem (bold)
73.3mL
73.3mL
73.3mL
73.3mL
73.3mL
73.3mL
Deionized water
CPC-chem
Deionized water
CPC-chem
CPC-chem
CPC-chem
Wet density ρt (g/cm3)
1.831
1.811
1.936
1.851
1.921
1.933

Figure 2

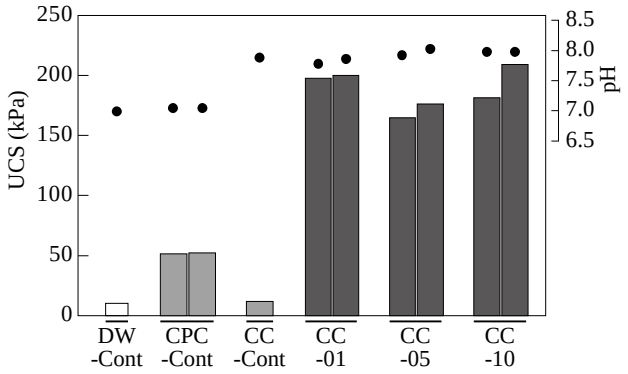

250
200
150
100
50
0
UCS (kPa)
8.5
8.0
7.5
7.0
6.5
pH
DW -Cont
CPC -Cont
CC -Cont
CC -01
CC -05
CC -10

Figure 3

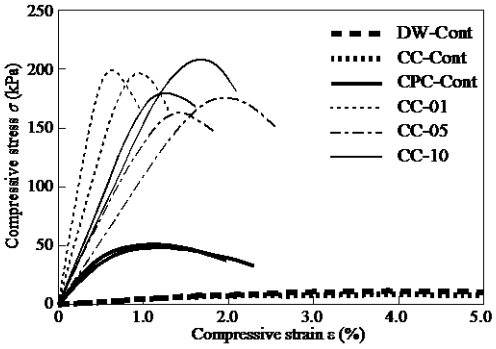
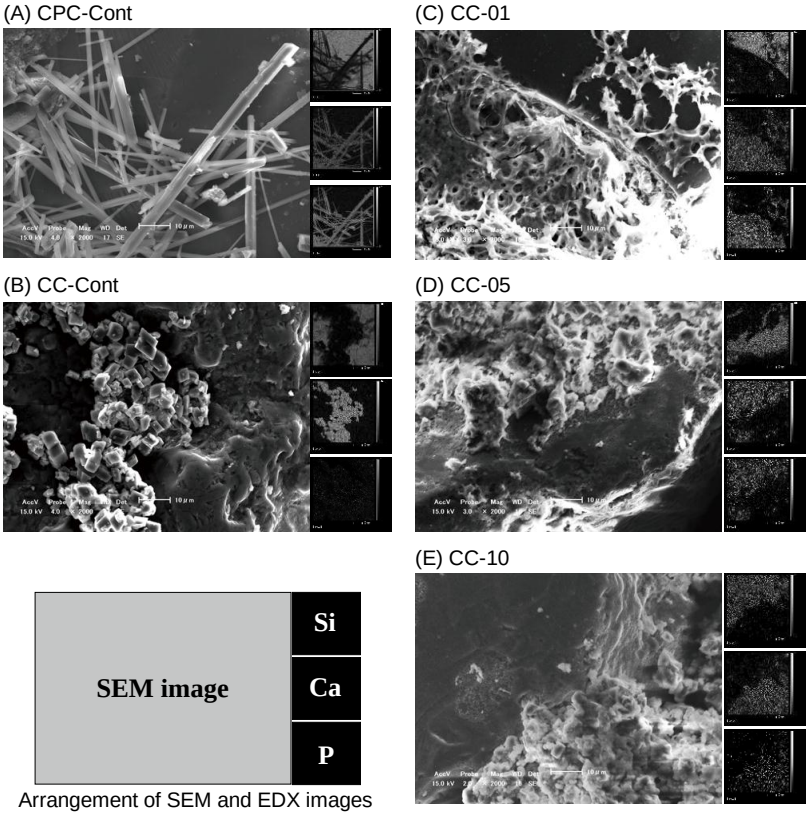

250
200
150
100
50
0
Compressive stress σ (kPa)
0
1.0
2.0
3.0
4.0
5.0
Compressive strain ε (%)
DW-Cont
CC-Cont
CPC-Cont
CC-01
CC-05
CC-10

Figure 4


(A) CPC-Cont
(B) CC-Cont
(C) CC-01
(D) CC-05
(E) CC-10
SEM image
Si
Ca
P
Arrangement of SEM and EDX images