Influence of relative density on microbial carbonate precipitation and mechanical properties of sand

L'influence que la densité relative du sol donne dans précipitation du carbonate microbienne et propriétés de la mécanique

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ABSTRACT: There exists a ground improvement technology that uses calcium carbonate precipitated from carbon dioxide generated by microbial metabolism and calcium sources in the pores of soil. It is known that the mechanical properties of the improved grounds correlate with the amount of calcium carbonate precipitation, but it is unclear how soil density influences calcium carbonate precipitation and the mechanical properties of the improved soil. Toyoura sand specimens of three relative densities are used to precipitate calcium carbonate through microbial metabolism. The injection-improved test and the triaxial test (consolidated-drained condition) are conducted to investigate calcium carbonate precipitation and the mechanical properties of the soil. The results show clearly that more calcium carbonate precipitation occurs in soil with lower relative density, but that in soil with higher relative density, the mechanical properties strengthen as calcium carbonate precipitation increases.

RÉSUMÉ : Dans la nature, il existe des micro-organismes qui capturent le dioxyde de carbone et les ions calcium présents dans la terre pour ensuite rejeter du carbonate de calcium. Ces dernières années, en s'appuyant sur le fonctionnement de ces micro-organismes, une technique renforçant la résistance du sol a été développée. Dans le cadre du développement d'une technologie qui renforce le sol à l'aide de métabolisme microbien, la présente étude a mis en évidence expérimentalement que l'influence de la densité relative du sol exerce un effet bénéfique. Tout d'abord, à travers une série de tests de cisaillement, le comportement au cisaillement du sol renforcé par le métabolisme de ces micro-organismes a été mis en avant. Ensuite, il s'est avéré que plus la densité relative du sol était petite, plus la quantité de carbonate de calcium déposée était importante. Cependant, nous avons aussi compris que plus la densité relative du sol était grande, plus les effets renforçant la résistance du sol étaient visibles.

KEYWORDS: micro-organism, ureolysis, soil improvement, mechanical properties, triaxial test, calcium carbonate

1 INTRODUCTION

To ensure the efficient maintenance of civil engineering structures, which is an issue, ground improvement technologies can be applied to reinforce existing structures. Ground improvement technology that uses calcium carbonate precipitated from carbon dioxide generated by microbial metabolism and calcium sources in the pores of soil is expected to be applicable to ground directly under existing structures because the viscosity of the injected grout is low (Wiffen et al. 2007). It is known that the mechanical properties of grounds improved through the use of this method correlate with the amount of calcium carbonate precipitation (Inagaki et al. 2011), but it is unclear how the relative density of the soil influences the precipitation and the mechanical properties of the improved soil.

In this study, we used *Sporosarcina pasteurii* (ATCC11859) to stimulate microbial metabolism via the following reactions. Our aim was to investigate the relationship between the soil's mechanical properties and calcium carbonate precipitation in three types of Toyoura sands that were compacted by microbial metabolism.

(Ureolysis)

$$CO(NH_2)_2 + 3H_2O \rightarrow 2NH_4^+ + 2OH^- + CO_2 \tag{1}$$

(Calcium carbonate precipitation)

$$CO_2 + H_2O \rightarrow HCO_3^- + H^+ \tag{2}$$

$$HCO_3^- + Ca^+ + OH^- \rightarrow CaCO_3 + H_2O \tag{3}$$

In the tests, microbial broth and a nutrient mixture were injected into the Toyoura sand specimens with three relative densities to improve compaction. In addition, we carried out a triaxial test (consolidated-drained [CD] condition) on the improved specimens. Then, we examined the influence of the relative density of the soil on calcium carbonate precipitation and the soil's mechanical properties.

2 TEST METHODS

2.1 *Method for production of specimens*

A half-split mold made of PVC, 15 cm in height and 5 cm in diameter, was used to create the specimens (Fig. 1 and Photo 1). Silicone grease was applied on the internal surface of the mold to prevent the generation of water paths along the wall. Toyoura sands having the physical properties listed in Table 1 were used to create the specimens, and the molds were filled with sand using the air-drop method. The specimens had a relative density of Dr = 15% (ρ_d = 1.372 g/cm^3), Dr = 60% (ρ_d = 1.504 g/cm^3), and Dr = 85% (ρ_d = 1.589 g/cm^3). The specimens were checked for weight and density, fitted with a collar on top, and saturated with distilled water supplied from the bottom. The surface of the specimen of Dr = 15% sank significantly during the hydraulic filling. This settlement was measured using vernier calipers, and it was confirmed that the relative density after hydraulic filling was about Dr = 30%.

2.2 *Curing method (nutrient injection process)*

After saturation, 250 ml of microbial broth was injected into each specimen. The microbial broth was made by planting *Sporosarcina pasteurii* in the medium described in Table 2.

After confirming that the broth had completely permeated the specimen, 200 ml of the nutrient mixture described in Table 3 was injected into the specimen at 12 h intervals for specified cycles.

Table 1. Physical properties of Toyoura sand

Soil particle density ρ_s (g/cm^3)	Water content (%)	Max. grain diameter (mm)	50% diameter on the grain size diagram D_{50} (mm)	Fine fraction content (%)	Max. dry density $\rho_d$max (g/cm^3)	Min. dry density $\rho_d$min (g/cm^3)	Soil suspension pH
2.623	0.0	0.425	0.177	0.6	1.645	1.333	6.3

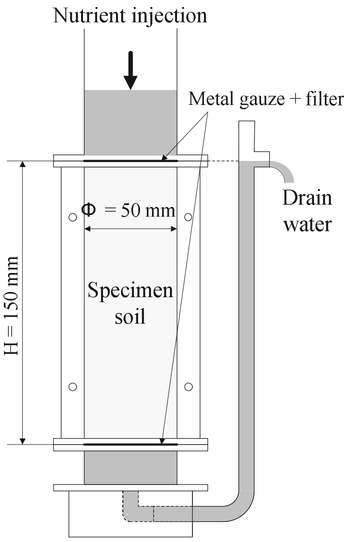

Figure 1. Schematic diagram of the mold

Photo 1. Photo of the mold

Table 2. Composition of medium

Name of reagent	Added amount
0.13 M Tris (pH = 9)	0.13 mol = 15.75 g
Yeast extract	20 g
(NH4)$_2$SO$_4$	10 g
Distilled water	1 L

Table 3. Composition of nutrient mixture

Name of reagent	Added amount
Nutrient broth	3 g
NH_4Cl	10 g
$NaHCO_3$	2.12 g
Urea	0.5 mol = 30.03 g
$CaCl_2$	0.5 mol = 55.49 g
Distilled water	1 L

The mold was cured in a room with a constant temperature set at 22°C. The nutrient mixture that had been injected previously and that remained in the pore was pushed out and drained to maintain the saturated state of the specimen in the mold.

Approximately 12 h after the specified cycles of nutrient injection were completed, 300 ml of distilled water was injected to wash away the nutrient mixture remaining in the pore.

As Table 4 shows, 15 specimens were made. The nutrient mixture was injected into these specimens at various frequencies in order to diversify the amount of $CaCO_3$ precipitation at each relative density. In addition, three specimens were only saturated with distilled water, and not injected with the broth and nutrient mixture, in order to examine the strength of the Toyoura sand itself.

Table 4. Test cases

Case	Specimen No.	Frequency of nutrient injection (Total injection amount ml)	Curing time (hours)	Initial dry density • $_d$ (g/cm^3)	Initial relative density Dr (%)	$CaCO_3$ Precipitation (kg/m^3)
Dr30	Dr30-N	—	—	1.423	33.2	—
	Dr30-P1	2(400)	24	1.428	35.2	32.24
	Dr30-P2	4(800)	48	1.414	30.2	63.72
	Dr30-P3	8(1600)	96	1.416	30.8	141.92
	Dr30-P4	12(2400)	144	1.414	30.4	225.53
Dr60	Dr60-N	—	—	1.513	62.6	—
	Dr60-P1	2(400)	24	1.504	59.9	28.11
	Dr60-P2	4(800)	48	1.504	60.0	51.77
	Dr60-P3	8(1600)	96	1.504	59.9	130.61
	Dr60-P4	12(2400)	144	1.504	60.0	212.11
Dr85	Dr85-N	—	—	1.589	84.9	—
	Dr85-P1	2(400)	24	1.589	85.0	27.12
	Dr85-P2	4(800)	48	1.589	85.0	57.38
	Dr85-P3	8(1600)	96	1.589	84.9	117.13
	Dr85-P4	12(2400)	144	1.589	85.0	198.55

2.3 *Triaxial test method*

Triaxial tests (CD condition) were conducted using the specimens produced by the method explained in 2.2. To reduce disturbance caused by the demolding/shaping of specimens, the specimens were frozen in the mold after the free water was removed. The specimens that featured high $CaCO_3$ precipitation were also frozen to equalize test conditions. The frozen specimens were removed from the molds and shaped to 10 cm in height and 5 cm in diameter. The shaped specimens were measured to check the diameter and height and then placed in a triaxial cell and defrosted under a negative pressure of 30 kPa. The defrosting time was set at about 1.5 h, which was the approximate time needed for stabilization of the axis displacement caused by contraction in the process of defrosting. We measured the diameter and the height of the defrosted specimens, covered them with a cell cover, and saturated them with degassed distilled water via the double-negative pressure method. The back pressure was increased to 200 kPa, and effective consolidation stresses of σ_c' = 100 kPa were applied for isotropic consolidation. After consolidation, we confirmed that the *B* values in all the specimens were 0.95 or higher. Then, axial compression was performed at a strain rate of 0.5%/min. The axial force was measured by the load cell inside the cell, axial strain was measured by the displacement gauge outside the cell, cell pressure and back pressure were measured by the water pressure gauge, and the volume change was measured by the low-capacity differential pressure gauge.

After the triaxial tests were completed, the specimens were dried in a furnace and weighed, and the $CaCO_3$ precipitation in each specimen was then obtained by measuring the decrease in mass resulting from $CaCO_3$ decomposition by hydrochloric acid.

3 TEST RESULTS

3.1 *Relationship between the amount of nutrient mixture injected and the $CaCO_3$ precipitation*

Figure 2 shows the relationship between the amount of nutrient mixture injected and the $CaCO_3$ precipitation. The quantity of $CaCO_3$ precipitation is given per unit volume of the test specimen.

The tendency for $CaCO_3$ precipitation to increase as the injections of the nutrient mixture increased can be confirmed for each relative density. When the total injection of the nutrient mixture is less than 800 ml, the differences among specimens with different densities is unclear. When more than 800 ml of the nutrient mixture is injected, the differences among the specimens with different densities are observed. It is confirmed that lower the relative density, the more $CaCO_3$ precipitates. This is because the low relative density increases the amount of microbes and nutrients absorbed by the test specimen.

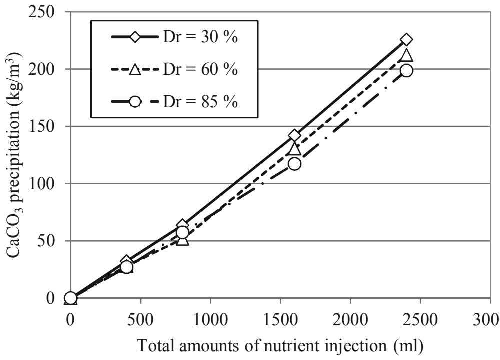

Figure 2. Total amounts of nutrient mixture injected and $CaCO_3$ precipitation

3.2 *Relationships among axial strain, principal stress difference, and volumetric strain*

Figure 3 shows the principal stress difference–axial strain curves and volumetric strain–axial strain curves. Toyoura sands of each relative density saturated only with distilled water (unsolidified) and those injected with 800 ml of the nutrient mixture and $CaCO_3$ precipitation of 51.8–63.7 kg/m^3 are shown in Fig. 3. Photo 2 shows the solidified test specimen after shear.

It can be confirmed that the solidification caused by the $CaCO_3$ precipitation leads to an increase in the maximum principal stress. In specimens with about the same $CaCO_3$ precipitation, the higher relative density of the soil, the increase in the maximum principal stress difference is the greater. Strain softening behavior is observed when the principal stress difference reaches the maximum in the solidified test specimen. A residual state occurs when axial stress reaches 5% or more; then, the principal stress difference is constant at every relative density, and its value shows no difference at each relative density. It is thought that the principal stress difference becomes equality in the residual domain because the test specimen is sheared along the sliding surface. Photo 2 also shows a shearing plane along the sliding surface. The increase in the volumetric strain on the expansion side is confirmed clearly in the solidified test specimens when the axial strain is small at each relative density in comparison with the unsolidified test specimens, and the ratio of increase becomes small around an axial strain over 5%.

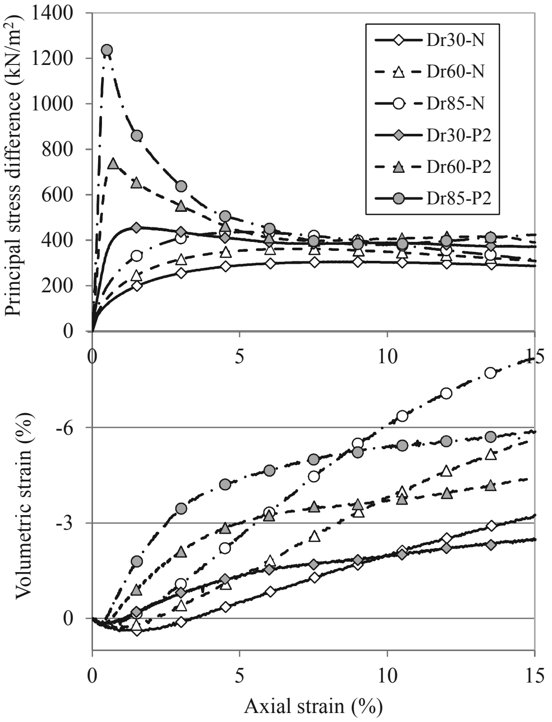

Figure 3. Relationships among axial strain, principal stress difference, and volumetric strain (σ_c' = 100 kPa)

Photo 2. Condition of the test specimen after shear

3.3 *Relationship between $CaCO_3$ precipitation and maximum principal stress difference*

Figure 4 shows the relationship between $CaCO_3$ precipitation and the maximum principal stress difference. There is no change in the maximum principal stress difference when $CaCO_3$ precipitation is less than 30 kg/m^3 at each relative density. At precipitation levels greater than 30 kg/m^3, the maximum principal stress difference increases monotonically depending on $CaCO_3$ precipitation. The increase in strength is remarkable in test specimens have higher relative density but little $CaCO_3$ precipitation. In the case of Dr = 85%, in comparison with Dr = 30% and Dr = 60%, the maximum principal stress difference increases even as $CaCO_3$ precipitation stays at the same level.

3.4 *Relationship between $CaCO_3$ precipitation and secant modulus*

Figure 5 shows the relationship between $CaCO_3$ precipitation and the secant modulus. The secant modulus was calculated from the principal stress difference at the axial strain of 0.4%. At each relative density, there is little change when $CaCO_3$ precipitation is less than 30 kg/m^3; however, when $CaCO_3$ precipitation is more than 30 kg/m^3, the secant modulus increases linearly according to the increase in $CaCO_3$ precipitation.

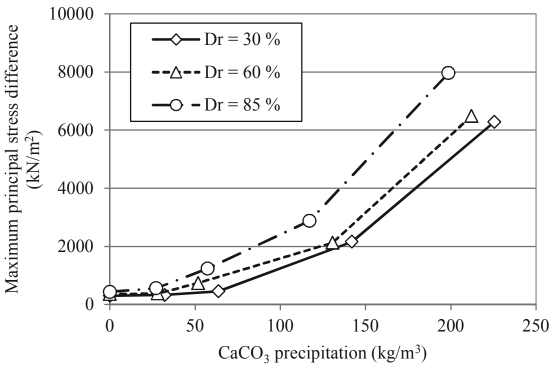

Figure 4. Relationship between $CaCO_3$ precipitation and maximum principal stress difference

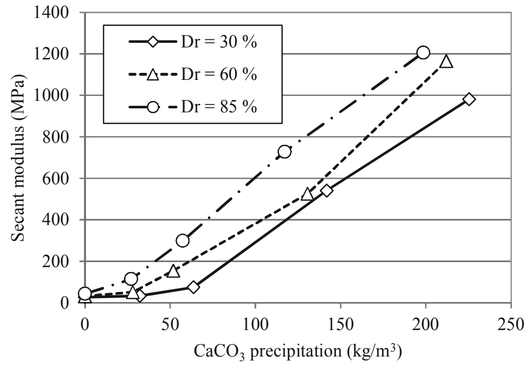

Figure 5. Relationship between $CaCO_3$ precipitation and secant modulus (axial strain 0.4%)

3.5 *Relationship between $CaCO_3$ precipitation and axial strain at maximum principal stress difference*

Figure 6 shows the relationship between $CaCO_3$ precipitation and axial strain at the maximum principal stress difference. For each relative density, when $CaCO_3$ precipitation is less than 60 kg/m^3, the axial strain at the maximum principal stress difference increases linearly according to the increase in precipitation. When $CaCO_3$ precipitation is more than 60 kg/m^3, the axial strain at the maximum principal stress difference remains in the vicinity of 0.5% and shows no difference at each relative density.

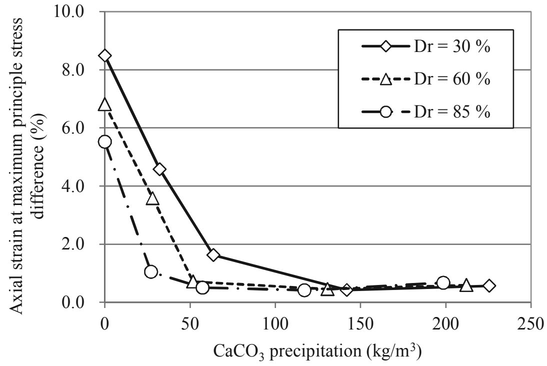

Figure 6. Relationship between $CaCO_3$ precipitation and axial strain at maximum principal stress difference

3.6 *Relationship between $CaCO_3$ precipitation and residual stress*

Figure 7 shows the relationship between $CaCO_3$ precipitation and residual stress. The residual stress indicates the minimum principal stress difference, less than axial strain 15%, after the maximum principal stress difference.

There is little change in the residual stress at each relative density up to about 30 kg/m^3 of $CaCO_3$ precipitation in comparison with a case of no precipitation. It is confirmed that the residual stress increases as the $CaCO_3$ precipitation increases at precipitation levels of more than 30 kg/m^3. The specimens for each relative density show no difference at $CaCO_3$ precipitation levels less than 60 kg/m^3, and it is unclear whether a difference is observed at precipitation levels greater than 60kg/m^3.

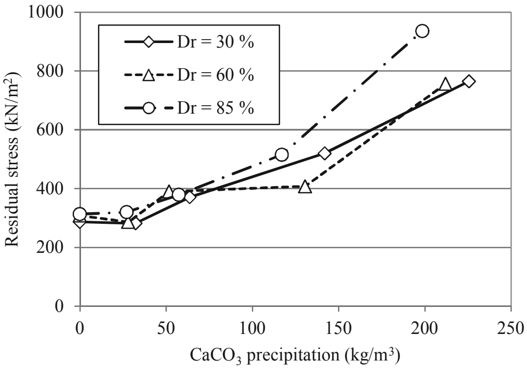

Figure 7. Relationship between $CaCO_3$ precipitation and residual stress

4 CONCLUSION

We investigated the influence of the relative density of soil on $CaCO_3$ precipitation by microbial metabolism and on the soil's mechanical properties. The results of our experiment are as follows:

- The $CaCO_3$ precipitation tends to increase as the relative density of the soil decreases.
- The maximum principal stress difference increased monotonically with the $CaCO_3$ precipitation at each relative density. The secant modulus increased linearly.
- The increase in the maximum principal stress difference was remarkable in soils with high relative density.
- The axial strain at the maximum principal stress difference decreased depending on $CaCO_3$ precipitation in specimens of all relative densities and became constant regardless of $CaCO_3$ precipitation when it approached 0.5%.
- The residual stress increased monotonically depending on $CaCO_3$ precipitation, but the differences among the relative densities are unclear.
- A meaningful difference is not seen in the mechanical properties of the soil among specimens with no precipitation and those with $CaCO_3$ precipitation up to 30 kg/m^3.

These results indicate that when applying this injection solidification technique in the field, the density of the existing ground will affect the strength increase. Therefore, like a conventional compaction method used in construction, this method would require a combination examination beforehand in order to confirm extreme expression characteristics.

Because the axial strain at the maximum principal stress difference becomes constant regardless of density when $CaCO_3$ precipitation becomes constant, we suggest that the approximate strength of the soil can be estimated using the secant modulus.

We will investigate the influence of soil density on a permeability change attributable to solidification by microbe metabolism in the future.

5 REFERENCES

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