Effect of Water Content on the Strength of Bio-Cemented Sand in Various Drying Process

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Abstract. Microbially induced calcium carbonate precipitation (MICP) is a novel ground improvement technique which has promising applications in geotechnical engineering. The research of this technique commonly focuses on calcium carbonate precipitation and geotechnical properties of bio-cemented soil in fully saturated or dry states. However, the natural soil may contain different amount of water, which will have influences on the geotechnical properties of soil. The objective of the present study is to investigate the mechanical properties of bio-cemented sand with different water contents and drying temperatures. During MICP treatment, all the bio-cemented sand samples were prepared at the same cementation level. Meanwhile, two drying temperatures (i.e., 30 °C and 60 °C) were adopted to obtain different water contents. Calcium carbonate content and water distribution were tested, and unconfined compression test was conducted to measure the unconfined compressive strength. The experimental results show that, as the water content increases, the strength decreases firstly and then increases at the same drying process. This phenomenon may be caused by the interaction of pore water and calcium carbonate crystals during the drying period. However, under the same water content, drying in 30 °C has more remarkable influences on the strength in comparison with the drying in 60 °C. The much higher strength obtained by the 30 °C drying in dried state implies that different temperatures may change the crystal forms of calcium carbonate.

Keywords: Bio-cemented sand · Water content · Temperature · Unconfined compression test · Calcium carbonate crystal

1 Introduction

Nowadays, due to the high-speed development in infrastructure, the land that is suitable for construction is limited. Traditional ground improvement techniques, such as chemical grouting, are usually adopted to improve the soil properties to satisfy the engineering demands. However, these ground improvement techniques would cause environmental problems. Hence, the use of human-made chemical materials should be reduced or banned (DeJong et al. 2010).

Currently, microbially induced calcium carbonate precipitation (MICP) has been introduced into the geotechnical engineering. It is a natural biomineralization process that the bacteria hydrolyse the urea into ammonia and carbonate ions to induce calcium

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carbonate crystals at the presence of calcium ions. The reactions involved in this process are as follows:

$$CO(NH_2)_2 + 2H_2O \rightarrow 2NH_4^+ + CO_3^{2-} \quad (1)$$

$$Ca^{2+} + CO_3^{2-} \rightarrow CaCO_3 \downarrow \quad (2)$$

The calcium carbonate crystals precipitate in the void to fill the pore space and/or bond the soil particles and then the soil properties can be improved (Ivanov and Chu 2008). It can be found from the reactions that four key parameters may influence the precipitation of calcium carbonate crystals, including calcium concentration, carbonate concentration, pH and the availability of nucleation sites (Whiffin et al. 2007; Hammes and Verstraete 2002). Many researchers have conducted investigations to study the effects of MICP treatment, such as the type and activity of bacteria (Okwadha and Li 2010; Hata et al. 2011), component of chemical solutions (Al Qabany and Soga 2013; Zhang et al. 2015), injection methods of bacterial suspension or chemical solutions (Rong et al. 2012; Rahim et al. 2015), and chemical reaction conditions (pH, temperature and catalyzer) (Stocks-Fischer et al. 1999; Bachemeier and Ringuelet 2002; Cheng and Shahin 2016).

However, the factors that affect mechanical properties of bio-cemented soil are more than these. The circumstance of soils after solidified treatment (i.e. MICP treatment) plays an important role in soil properties, such as water content and temperature of drying process.

There are many reports about the effects of water content and temperature on properties of different type of soils. With the increasing of water content, cohesion of soil would first increase to the peak and then decrease, while internal friction angle would decrease to nearly zero (Chen et al. 2007). For cemented soils, there is an optimum water content that can get the maximum strength of soil (Ribeiro et al. 2016). This may be caused by the optimum amount of bonds which can provide better junctions for soil particles. What's more, with the variation of water content, pores between soil particles would be affected that lead to the strength changes (Zuo et al. 2016). Although many researches have demonstrated strong connection between water content and soil properties, the studies about the influence of water content on the properties of bio-cemented soil are very few. This can cause the misestimate of bio-cemented soil properties. Besides, in many experiments of bio-cemented soil, temperature of drying process in the oven after MICP treatment are usually 30 °C (Li 2015) or 60 °C (Truong et al. 2012). But the comparison between these two temperatures of drying process are limited. The changing of water content and temperature of soils are common in the field which could be caused by weather changes, flow changes and so on. Therefore, it is very essential to investigate the effect of water content and drying temperature on mechanical properties of bio-cemented soil in drying process.

Nevertheless, salinity of bio-cemented soil after MICP treatment is an important factor which should be noticed first before the study of influences on drying process. Truong et al. (2010) found that a very low content of salt in soil can change the small strain stiffness greatly. In addition, this result was consistent with the study of Li (2015) that an overestimation strength of soil would be caused by the residual of calcium chloride. The salinity of samples is crucial for the estimation of factors on soil properties and some measures should be taken to eliminate this impact.

The objective of this study is to investigate the effect of water content on the strength of bio-cemented sand in two kinds of drying process. A series of bio-cemented sand specimens were prepared and then drying processes (30 °C and 60 °C) were conducted after the specimens were washed by tap water. After fully saturated and dried for different time, the unconfined compression tests were conducted to analyse the strength of bio-cemented sand. The results indicate that both water content and temperature correlate to the strength of bio-cemented sand. The overestimation of strength may occur without taking into account water content and drying temperature.

2 Materials and Methods

2.1 Bacterial Suspension and Urease Activity

Sporosarcina pasteurii [American type culture collection (ATCC 11859)] was used in this study. According to the NH_4-YE medium (ATCC 1376), 0.13 mol l^{-1} Tris buffer (pH = 9.0), 10 g l^{-1} $(NH_4)_2SO_4$, and 20 g l^{-1} yeast extract were included, and individual ingredients were autoclaved separately before mixing together. After inoculated into the medium, the bacterium was incubated at 30 °C with a shake rate of 150 r/min for 24 h. As the concentration of bacterial suspension was proportional to the optical density (OD_{600}), the visible light spectrophotometer was used to determine the OD_{600} of bacterial suspension, which was 1.66 in this study.

Urease activity could present the activity of bacteria that was vital for MICP process. To obtain the urease activity of the bacteria, conductivity method was used, which was similar to the research of Whiffin (2004). The rate of conductivity increase was 0.121 mS/cm/min, urease activity was 1.344 mM/min, and specific urease activity was 0.81 mM/min.

2.2 Sand and Specimen Preparation

PVC tube (140 mm in length and 37 mm in diameter) was used in this study to prepare the specimens (37 mm in diameter and 80 mm in length). The specimen preparation was similar to that of Cui et al. (2015). The tube was positioned vertically with a rubber plug in the bottom and packed with ISO standard sand for 80 mm in height, the grain size of ISO was in Fig. 1. The dry mass of each sand specimen was 160 g. A porous stone with thickness of 5 mm was put on the top and bottom of the sand sample to facilitate the solution diffusion. The dry density and initial porous ratio of these samples were 1.86 g/cm^3 and 0.30–0.32. Prior to the injection of bacterial suspension, each sand specimen was saturated for 24 h to exhaust air.

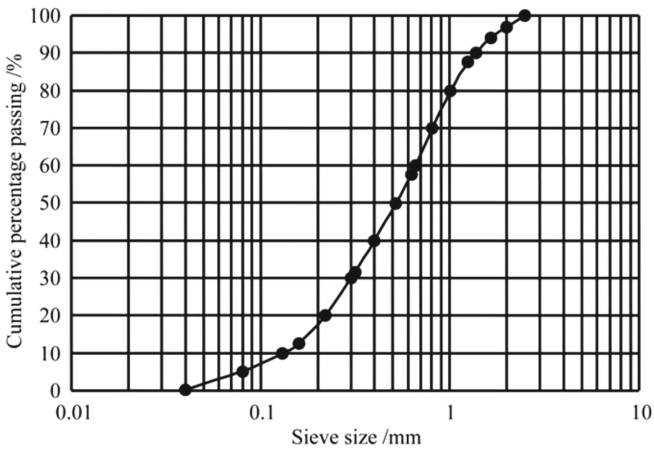

Fig. 1. Grain size sieve analysis curve of ISO sand

2.3 MICP Treatment

In the MICP treatment, bacterial suspension and cementation reagents were injected into specimens to induce calcium carbonate precipitation. The concentration of the cementation reagents (calcium chloride-urea solution) was 0.5 mol/L. The MICP treatment was conducted in the following two steps:

(1) Injection of bacterial suspension: One pore volume of bacterial suspension was injected into each specimen through a peristaltic pump with an input rate of 5 ml/min. Then, the injection was stopped for 6 h for the adequate adsorption of bacteria on sand particles.
(2) Injection of cementation reagents: One pore volume of cementation solution was injected into each specimen through the peristaltic pump with an input rate of 10 ml/min for every 24 h. All the sand specimens were treated for seven times in total.

2.4 Drying Process and Unconfined Compression Test

After the completion of MICP treatment, all specimens were treated by following steps:

(1) Specimens were flushed by at least 10 pore volume water to cut off the reaction of remained reagents and eliminate the effect of salt on strength in sand samples (He and Chu 2016).
(2) Specimens were saturated for 24 h using vacuum saturation method. Then, the weight of the bio-cemented specimens would be measured quickly after the rubber plug was removed and water on the tube wall was wiped. The weight of samples was termed as m_1.
(3) Specimens were divided into three series as shown in Table 1. The samples of Series I would be conducted the unconfined compression test (UCT) immediately after saturation and removal of PVC tube. The samples of series II and III were dried in

the oven for different temperatures and time before UCT. The weight of specimens after different drying time was m_2, while the weight of PVC tube removed before UCT was m_{pvc}. The water loss of each specimen was decided by m_1-m_2. And there were five groups in each series depending on the average water loss of each group.

Table 1. Drying process of three series

Series	Drying process	Average water loss (g)				
		A	B	C	D	E
I	Saturation	–	–	–	–	–
II	60 °C	7.7	10.6	17.8	29.2	32.5
III	30 °C	7.7	10.6	17.8	29.2	32.5

Note: Specimens in group A-C were cut into three parts after UCT

(4) After the unconfined compression test, samples of group A-C were cut into three parts to measure the water and calcium carbonate distribution of each group. Then samples were dried in the oven of 105 °C until there was no weight change.

2.5 Measurement of Calcium Carbonate Content

The content of calcium carbonate was measured by acid treatment (Montoya and DeJong 2015). The dry weight before and after acid treatment by 2 M HCl was m_3 and m_4. The calcium carbonate mass (m_c) was determined as follows:

$$m_c = m_3 - m_4 \tag{3}$$

In this study, the water content (w_w) and saturation degree (S_r) of each sample were used to measure the water volume of samples by the following equations:

$$w_w = \frac{m_2 - m_4 - m_{pvc}}{m_4} \times 100\% \tag{4}$$

$$S_r = \frac{V_{water}}{V_{void}} \times 100\% = \frac{m_2 - m_4 - m_{pvc}}{m_1 - m_4 - m_{pvc}} \times 100\% \tag{5}$$

The V_{water} was the volume of water in samples after different drying process, and V_{void} was the volume of water of samples in saturation state.

3 Results

3.1 Mass of Calcium Carbonate of Bio-Cemented Sand

The precipitation of calcium carbonate is the key point of properties improvement of bio-cemented sand. In many researches, mass of calcium carbonate is a measurement for cementation level which can strongly affect the strength of samples (Whiffin and van

Paassen 2007; Cheng et al. 2013; Soon et al. 2013). In this study, the average calcium carbonate mass of each group is shown in Fig. 2. It can be seen that all the bio-cemented sand samples have similar calcium carbonate mass after the same MICP treatment, indicating that the amount of precipitated $CaCO_3$ in different groups is relatively uniform. This confirms that the influence of calcium carbonate content on the strength of bio-cemented sand can be ignored.

In the same level of calcium carbonate mass, differences of distribution in samples may have effects on the strength of bio-cemented sand (Harkes et al. 2010). In MICP process, calcium ions are attached to the bacteria due to the negative charge of cell wall. Hence, calcium carbonate will precipitate around the bacteria cell that is absorbed on sand grains (Muynck et al. 2010). In other words, the sites of calcium carbonate are fixed after MICP treatment and the distribution of calcium carbonate has no effects on strength of samples in this test.

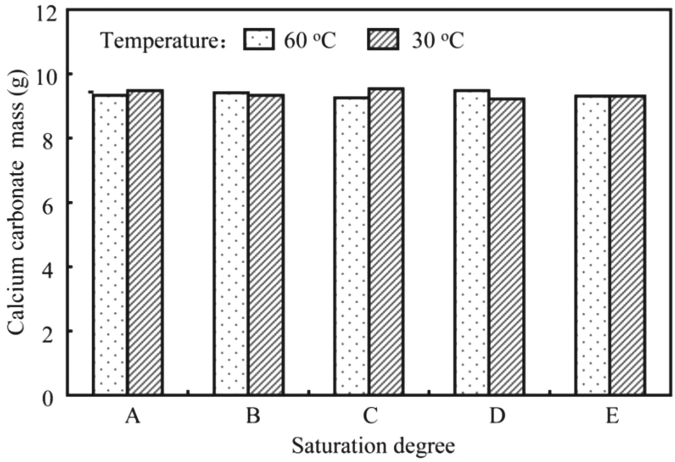

Fig. 2. The average calcium carbonate mass of each group

3.2 Effect of Water Content on Strength of Bio-Cemented Sand

The relationship between water content and unconfined compressive strength (UCS, termed as strength hereon) is presented in Figs. 3 and 4. The saturation degree of 100% and 0% represents the fully saturated and fully dried states, respectively. It can be found that strength has a strong correlation to the water content of bio-cemented sand samples. As the saturation degree decreases, the strength decreases slightly first and then increases quickly up to the peak strength of the fully dried samples. The variation trends of water content versus strength are both in concave shape in two figures. The strength of fully dried bio-cemented sand samples is higher than that of fully saturated ones, which is similar to the findings of Cheng (2013) and Li (2015). However, the enhancement of strength in fully dried state is not as high as the reported strength of Li (2015) due to the salt washing of samples before saturation.

During the drying process, the rate of water loss in different parts is various with the approach to the central of samples. In this case, water content of different parts in samples (termed as water distribution hereon) is measured to study the effect of it on the strength. The result of water distribution in different water loss is shown in Fig. 5. It can be seen that the total water content of samples decreases with the increase of water loss. Nevertheless, there is no distinct relationship between water distribution and water loss along samples. It suggests that water distribution is similar under the same drying temperature.

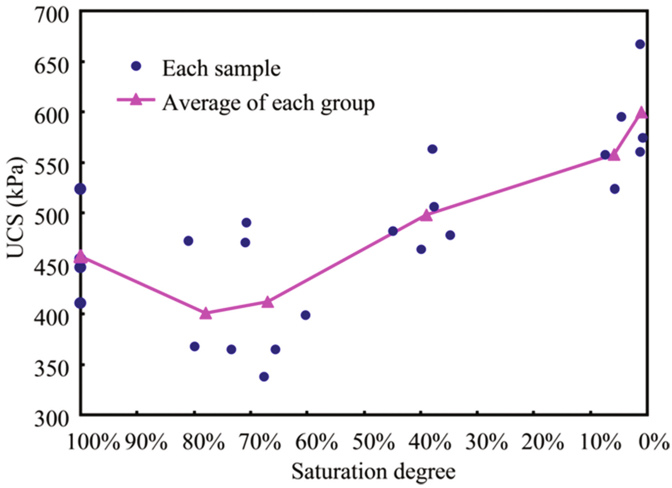

Fig. 3. Effect of water content on strength of bio-cemented sand in 60 °C drying

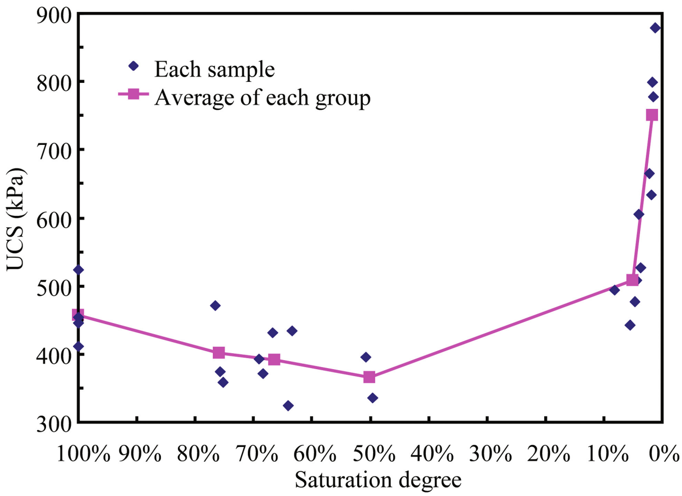

Fig. 4. Effect of water content on strength of bio-cemented sand in 30 °C drying

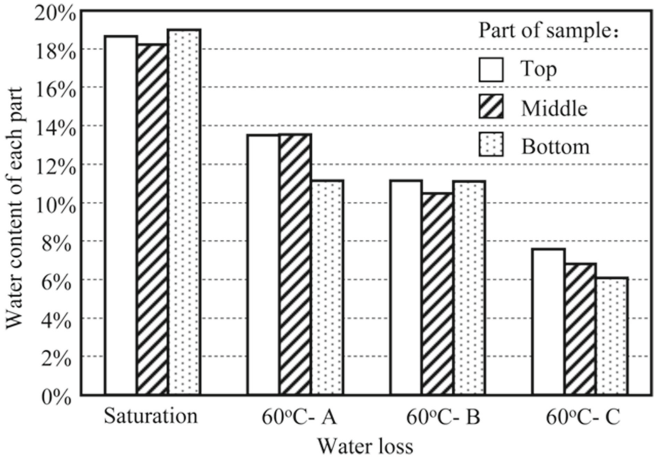

Fig. 5. Effect of water loss on water distribution of samples in 60 °C drying

3.3 Effect of Temperature on Strength of Bio-Cemented Soil

The influence of drying temperature on the strength of bio-cemented sand samples is shown in Fig. 6. Overall, the variation of strength is highly dependent on the temperature, presenting that higher strength can be obtained at higher temperature when the saturation degree is not too high. In the present study, when the saturation degree is larger than 80%, the strength is barely influenced by the drying temperature. However, if the bio-cemented sand is fully dried (i.e., saturation degree is zero), a relatively higher strength

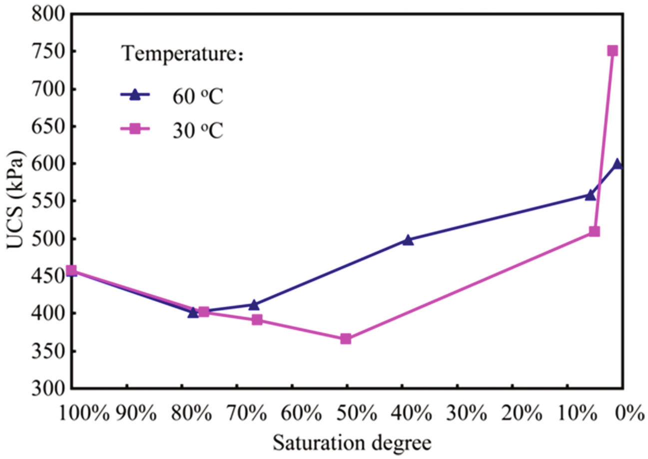

Fig. 6. Effect of drying temperature on strength of bio-cemented sand

is achieved. The considerable differences may be related to the variation of crystal structure caused by the different drying temperatures.

Similarly, the stiffness of bio-cemented sand samples is denoted by the average secant modulus, E_{50}. The value of E_{50} is equal to the ratio of 50% strength and its corresponding axial strain. The variation of E_{50} is shown in Fig. 7. The variation trend of stiffness is similar to that of strength, presenting a concave shape. That is to say, the stiffness of bio-cemented sand samples is also influenced by the drying temperature.

As a conceivable factor that affecting strength of samples, the water distribution of samples under different drying process is showed in Fig. 8. Although the average water content of top-middle-bottom parts is similar under the same water loss, the variation range is different, which represents the water distribution of samples. In the same water loss, the variation range of 30 °C drying is higher than that of 60 °C. In addition, the difference of variation range increases with the rise of water loss, indicating the non-uniform water distribution in 30 °C drying. In other words, the distribution of water content in 30 °C drying samples is inferior, which can cause the reduction of strength. It is consistent with the result of strength comparison under different drying temperatures in Fig. 6.

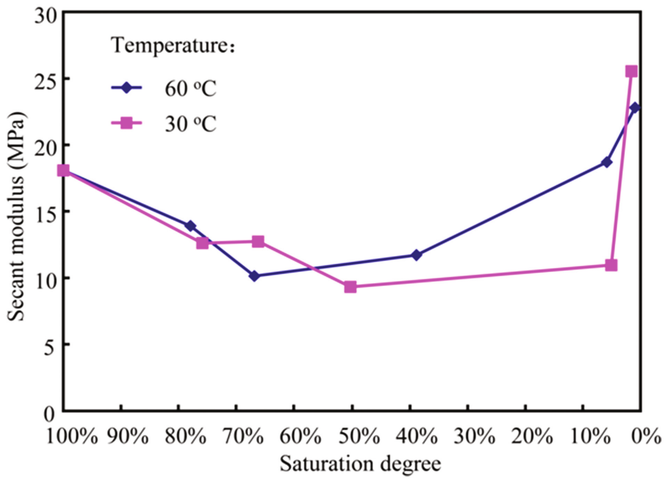

Fig. 7. Effect of drying temperature and water content on stiffness of bio-cemented sand

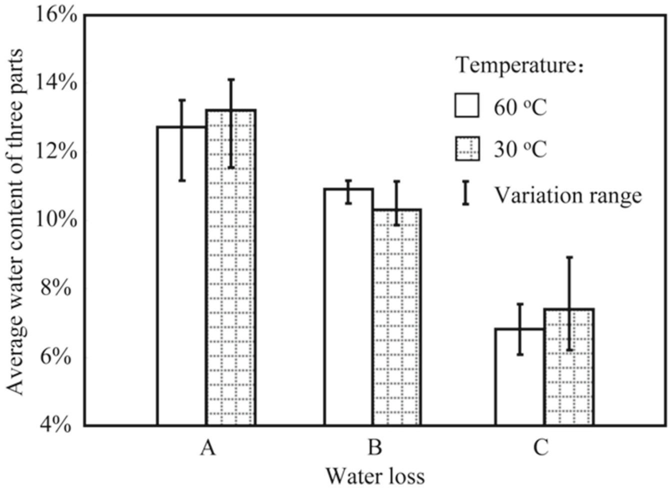

Fig. 8. Effect of water loss on average water content of top-middle-bottom parts in 60 °C and 30 °C drying

4 Discussion

From the theory of MICP process, calcium carbonate content, distribution, and crystal forms are three important factors of strength changing in the perspective of precipitated $CaCO_3$. According to the analysis above, it verifies that the content and distribution would not change in drying process after the same MICP treatment. Thus, it can infer that the crystal forms are responsible for the strength changing in drying process.

There are three crystal forms that have been detected in bio-cemented soil including calcite, aragonite and vaterite (Zhang et al. 2015). The differences between crystal forms can lead to the changing of cementation properties. It has been proved in aquatic environment that pure crystal of calcite, vaterite and aragonite is found with the temperature of 10 °C, 55 °C and 70 °C, respectively. In addition, mixture of vaterite and calcite is gotten in 25 °C (Huang et al. 2017). It suggests that the crystal forms of the precipitated calcium carbonate are various in different temperature of drying process which can cause different strength of samples as shown in Fig. 4. Moreover, the increase of supersaturation can accelerate the rate of nucleation and crystal growth (Han et al. 2006). In this study, with the water loss of samples, supersaturation can rise up to affect crystal forms of precipitated calcium carbonate. In that way, the strength of samples is various with different water content as shown in Figs. 2 and 3. This suggests that the influences of water content may be caused by the interaction of pore water and calcium carbonate crystals during the drying period. However, the crystal forms of calcium carbonate in different drying process should be tested to survey the polymorph of them, which is a valuable study in prospective research.

This study shows the relationship between strength and the temperature and water content in drying process which is meaningful for experimental work and engineering applications. In the lab, many studies commonly focus on the influences in material and MICP treatment and use fully saturated or dried samples for strength measurement but neglect the factors of water content and temperature in drying process. This can lead to the overestimation of strength. Compared to lab environment, the temperature and water content are different in the field, especially in seashore or riverside. The overestimation of strength could cause potential safety risk, while the strength changing may lead to damage of structure. The effect of these factors should be noticed to promote the application of MICP techniques.

5 Conclusion

In this study, the mechanical properties of bio-cemented sand with various water content are investigated under two drying temperatures. All samples are treated with the same MICP treatment and dealt with different drying process after salt washing. The following conclusions are drawn:

(1) In the same drying temperature, the strength of samples is strongly related to the water content. With the saturation decreases, the strength of samples is decreased slightly first and then increases quickly up to the peak strength in fully dried condition. And the interaction of pore water and calcium carbonate crystals in drying period is responsible for that.
(2) In the same water content, the strength of samples is various in different drying temperatures. The strength of samples in 30 °C drying is lower than that of 60 °C under the saturation degree of 5%–80% which may be caused by the inferior water distribution. However, when the saturation degree is lower than 5%, the strength of samples in 30 °C drying is higher than that of 60 °C, especially in the fully dried condition. And the stiffness of samples is influenced by temperature in the similar way of strength. These phenomena are mainly caused by the crystal forms of calcium carbonate which are transformational in different temperatures.

The results of this study indicate that the mechanical properties of bio-cemented sand after MICP treatment can be affected by the water content and drying temperature. This will cause the overestimation of strength in lab work or filed applications which should be noticed in the prospective studies.

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