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Experimental study on freezing point of saline soft clay after freeze-thaw cycling

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Abstract. The brine leakage is a tough problem in artificial freezing engineering. This paper takes the common soft clay in Wujiang District as the study object, and calcium chloride solutions with six salinity levels were considered. The 'classic' cooling curve method was employed to measure the freezing point of specimens after freeze-thaw. Results indicate that four characteristic stages can be observed including supercooling, abrupt transition, equilibrium and continual freezing, strongly dependent on the variation of unfrozen water content. Two characteristic points were found from the cooling curves, i.e., freezing point and initial crystallization temperature. A critical value for the former exists at which the increment lowers. The higher the saline content approximately linearly, lower the freezing point. In the initial five cycles, the freezing point increases and then stabilizes. Besides, the degree of supercooling was calculated and its correlations with water, salt and freeze-thaw cycles were noted. Finally, an empirical equation was proposed for the relationship of freezing point and three main factors, i.e., water content, saline content and freeze-thaw cycles. Comparison of calculated and measured data proves that it is reliable and may provide guidance for the design and numerical analysis in frozen soil engineering.

Keywords: freezing point; freeze and thaw; frozen soils; salinity; supercooling degree

1. Introduction

Urbanization and exploitation of natural resources have induced massive underground space development, e.g., crossing-river tunnels, subways and deep mining, accompanied with more inevitably difficult environments during construction such as high stress formation in deep stratum, water-rich soft ground and densely construction in urban areas (Wang *et al*. 2005, Ma *et al*. 2011, Marwan *et al*. 2016, Tengborg and Sturk 2016, Nelson 2016). The artificial freezing technique has already become one of the most reliable methods that facilitate the construction under such circumstances, especially when the soft clay is involved due to its characteristic engineering behaviors (Karstunen *et al*. 2006, Yazdani and Mohsen 2012, Fattah *et al*. 2015, Park 2016). This method better adapts to the stratum and the frozen wall generated during freezing has good bearing and waterproofing quality, and thus both temporary linings and dewatering measures can be reduced (Chen 1996, Tang *et al*. 2014, Vitel *et al*. 2016). More importantly, no additional materials intruded soil layers compared with conventional methods, e.g., chemical grouting, due to which artificial freezing is regarded as an environment friendly method (Yang *et al*. 2006). However, the brine leakage due to the crack of freezing pipe is a typical problem in artificial freezing construction and it threatens the stability of frozen wall in that the effective thickness and strength of frozen soils will be deteriorated due to salt inclusion. Among the physical indexes of soils, the first to bear the brunt is the freezing point that is generally used to classify whether soils are in frozen or unfrozen states (Sinitsyn and Loset 2010). Moreover, soil freezing point also acts as a baseline for evaluating the engineering properties of frozen wall (Bing and Ma 2011, Lai *et al*. 2017). However, up to now most of the engineering design and numerical analysis takes the freezing point as a constant (Teltayev and Aitbayev 2015, Wang *et al*. 2016), which have not well conisdered its correlations with test conditions. Thus, it is urgently needed to investigate the freezing point of saline soft clay.

With respect to the test methods for measuring soil freezing point, Parameswaran and Markay (1983) introduced the freezing potential to porous medium and its abrupt transition is taken to determine the freezing point of both saturated quartz sands and clays. However, the abrupt transition of freezing potential not only exist at temperatures close to the freezing point, and thus deviations inevitably occurred when measuring freezing point. Guan *et al*. (2014a) has drawn lessons from previous experimental work and suggest a novel method of determining soil freezing point at high stress conditions based on the relationship of resistance and temperature. This method well employed the merit that both temperature and resistance can be independently measured and mutually verified, and thus higher accuracy can be reached. The thermistor has been greatly improved in recent decades in three aspects such as miniaturization of volume, measuring

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accuracy and better stability. This enables us to directly measure the development of inner temperatures of soils during freezing and further determine the freezing point according to the abrupt transition phenomena. Thus, it is universally applied in frozen soil engineering due to both the simple structure of apparatus and convenient measuring procedure. Moreover, some new methods have been introduced to studies with increasing interdisciplinary researches, e.g., differential scanning calorimetry (DSC) (Kozlowski 2009). Kozlowski (2016) then proposed a simple method to obtain the soil freezing point depression, the unfrozen water content and pore size distribution curves based on the DSC.

The development of test method makes it possible to investigate the soil freezing point and its correlations with test conditions. Grechischev *et al*. (2001) carried out laboratory tests on oil-polluted soils and found the effect of water content on the freezing point of oil-polluted soils, manifesting as that at lower water contents the freezing point tends to decrease at higher concentrations of petroleum pollutant while as the water content grows, the variation of the freezing point has little correlation with the concentration. Suzuki (2004) utilized the freezing point depression method in determining the matric potential of soil water for a wide range of soil types. Kozlowski (2004) verified the applicability of the differential scanning calorimetry thermogram in measuring the soil freezing point and the unfrozen water content, and assumed that the soil freezing point is a power function of both water content and plastic limit. Bing *et al*. (2011) observed similar changes in freezing point for four kinds of soils, i.e., silty clay and sands along Qinghai-Tibet railway, loess and silty sands in Lanzhou, and four kinds of soluble salt were also considered in revealing the influence of common ions on soil freezing point. Besides, an exponential equation was put forward to describe the relationship between soil freezing point and water content, and a linear equation for soil freezing point at various saline contents. Wan *et al*. (2015) obtained the freezing point of sodium sulfate saline soil by tests and discussed the effect of both the thermodynamic free energy and pore radius on soil freezing point. Guan *et al*. (2014a) measured the freezing point of soils by self-developed apparatus for high stress conditions and noticed that for deep clay within pressures of 0 - 10 MPa, the freezing point decreases approximately linearly at higher pressures and proposed an empirical linear equation with two variables, i.e., water content and applied pressure. Wu *et al*. (2015) analyzed the effect of both solute and water on soil freezing characteristics based on experiments in laboratory. Previous work indicates that relatively mature test methods have been formed so far and experiments have been carried out on various types of soils and correlations of soil freezing point with water content, saline content and applied pressures were also investigated. However, as one of the significant factors affecting soil behaviors, the effect of freeze and thaw cycling on soil freezing point have been rarely seen in literatures.

Considering the complex migration of heat and water as well as the structural evolution of soil during freeze and thaw cycling, this paper takes the frequently encountered soft clay in subway excavation in Wujiang District as the study object and produces saline soft clay specimens by adding one of the common coolants in artificial freezing engineering, i.e., calcium chloride solution. Then the freezing point of saline soft clay after cyclic freeze and thaw was measured by the ‘classic’ cooling curve method, and its correlations with water content, saline content and cycles of freeze and thaw were discussed. Finally, an empirical equation was proposed by fitting test data taking into account the above three factors.

Table 1 Physical parameters of soft clay

Parameters	Value
Natural water content, w/%	49.3
Dry density, ρ_d/ g/cm^3	1.70
Void ratio, e	1.40
The Atterberg limits	
Plastic limit, w_P/%	21.6
Liquid limit, w_L/%	41.8
Compaction test	
Optimum water content, w_{opt}/%	18.9
Maximum dry density, ρ_{dmax}/ g/cm^3	1.75
Particle grading characteristics	
0.075-2.0 mm	7.5%
0.005-0.075 mm	71.5%
<0.005 mm	21.0%

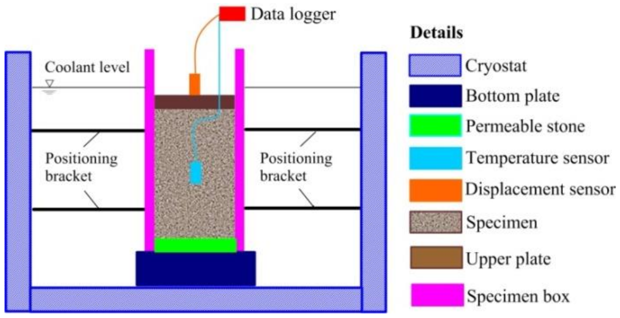


Fig. 1 Schematic diagram for the specimen box

2. Test plan

The soft clay samples were taken from a foundation pit in Wujiang District, Jiangsu Province, at a depth of 5.0-6.0 m. The physical parameters are listed in Table 1. The calcium chloride solution with five levels of saline contents were produced by mixing the 95% anhydrous calcium chloride and deionized water and was then placed in a waterproofing environment for 24 h. The prepared slurry sealed in plastic bags were put into a chamber with heat and moisture preserved for 48 h. The specimens for testing were produced in the stainless-steel box by layered compression of the slurry according to the target water content and dry density. The diameter and height for the specimens are 3.0 and 5.0 cm, respectively. The six levels of saline content for specimens were controlled to be 0.0%, 0.2%, 0.5%, 1.0%, 2.0% and 5.0% while six levels of water content were considered here, i.e., 10%, 20%, 30%, 40%, 50% and 60%. The dry density was controlled to be 1.70 g/cm^3 to eliminate the discrepancy caused by the variability of dry densities among soil specimens. The prepared specimens were kept in a waterproofing environment for 24 h to ensure the uniformity of both soil water and salt. Before testing, the differences among soil specimens were measured and it proves that the dry density ranges from 1.69-1.71 g/cm^3 and the difference of water content was less than 1.0%.

One of the common methods, i.e., the 'classic' cooling curve method proposed by Grechishchev *et al*. (2001), was used to obtain the freezing point of saline soft clay based on the equilibrium freezing phase that can be easily noted from the freezing curves for soils. Before testing, the specimen box was improved by adding a movable upper plate as a substitute so that the deformation of soil specimens during freeze and thaw is allowed to be monitored, which is facilitated by a high-precision displacement sensor that has been set on the upper plate. The inner temperature was live monitored by temperature sensors that is constituted by a thermocouple. The monitored data can be live recorded by the data logger. The schematic diagram for the specimen box is presented in Fig. 1. Before testing, the temperature of the coolant inside was lowered to -20℃ to create a uniform low-temperature environment and then the specimen was put into the cryostat for 4 h to ensure that the specimen was completely frozen. Then, the temperature was adjusted to be +20℃ and then it is maintained for 4 h so that soil specimen can be totally thaw. The accuracies for both temperature and displacement transducers are ±0.01℃ and ±0.01 mm, respectively. Besides, the number of freeze and thaw cycles are 0, 2, 5, 10 and 20.

3. Test results

3.1 The cooling curve and characteristic parameters

Fig. 2 illustrates the relationship between temperature and volumetric unfrozen water content of soils during freezing. Four characteristic stages can be observed from the curves (Wan *et al*. 2015), i.e., i) supercooling stage, at which the temperature of soil specimens was quickly lowered to the initial crystallization temperature, T_{sn}, at a low-temperature environment. However, the specimen has not been frozen and no ice crystals were formed; ii) the second stage with an abrupt temperature transition after the initial crystallization temperature is reached, demonstrating that the free water in soils begins to freeze and the unfrozen water content quickly decreases and ice crystals massively emerged with great amounts of latent heat released due to ice-water phase change, and thus, soil temperature rapidly increases; iii) equilibrium freezing stage, which proves that a thermodynamic equilibrium state has already been reached in specimens and this stable value in the cooling curve is generally taken as the freezing point of soils, T_f. At this stage, the temperatures in specimens tends to stabilize and the remaining free water and capillary water begins to freeze, with a relatively low decrease in the unfrozen water content; iv) continual freezing stage, at which a large proportion of soil water was frozen including free water, capillary water and film water, and the variation of unfrozen water tends to be stable and its content is mainly affected by both adsorptive action of colloid particles and supercooled water in micropores.

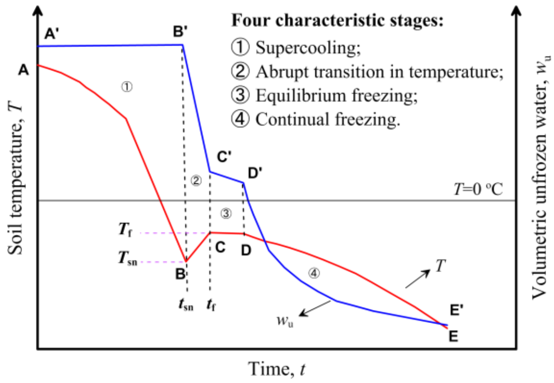


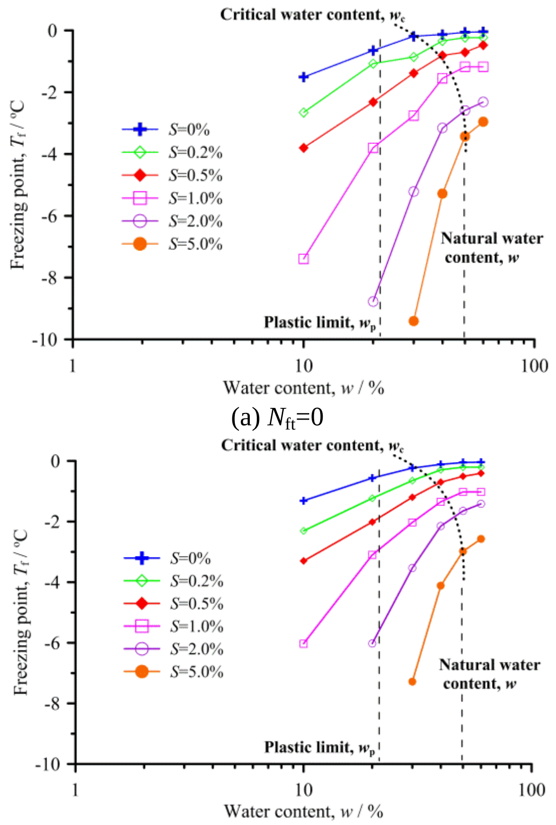
Fig. 2 Correlation of temperature and volumetric unfrozen water during freezing

3.2 Effect of both water and salt on freezing point

Fig. 3 presents the effect of water content on the freezing point of saline soft clay at given saline contents. It is noted that the freezing point of saline soft clay grows at higher water contents before and after freeze-thaw cycling. Interestingly, the break points may exist as noticed from the curves, and lies around the plastic limit at lower saline contents while the critical water content for cases at higher saline contents are close to the natural state. This strongly depends upon the amount of water that is involved in the ice-water phase change. The increment of freezing point to water content significantly increases before this critical point is reached while it stabilizes afterwards. Besides, a larger increment can be easily noticed from the curve at higher saline contents, i.e., the slope of the curve, especially at those points lower than the natural water content. This indicates that the freezing point of soils are sensitive to the variation of soil water when larger salinities are concerned, and this is closely related to the enhancing adsorptive effect of soil particle surface on soil water. For saline soft clay at various saline contents, the critical water content lies between the plastic limit and natural water content. The Gibbs-Thomson equation was introduced here to interpret the effect of water content on the freezing point (Watanabe and Wake 2008)

$$T_m - T = \frac{T_m \sigma}{\rho_i L_f r} \tag{1}$$

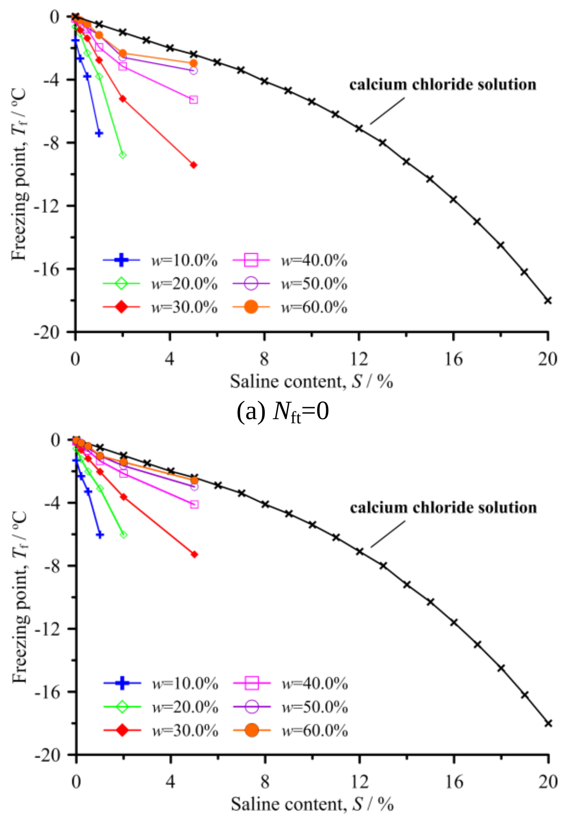
where, T_m is the freezing point of pure water at standard atmospheric pressure; T is the freezing point of water in capillaries; σ is the ice-water surface tension; ρ_i is the density of ice; L_f is the latent heat released during soil water freezing; r is the radius of capillaries. Experiments carried out by Lu *et al*. (2017) suggest that soil water can be simplified as capillary water and the radius of capillaries tends to decrease at lower water contents. Thus, the freezing point of soils can be obviously lowered due to capillary radius decrease, as can be interpreted by Eq. (1). Moreover, at higher water contents, the unfrozen water tends to grow and more latent heat will be released due to ice-water phase change at identical test conditions during freezing. Thus, the rate of cooling in soil specimen will be reduced and the amplitude of variation in freezing point is rather small.



(a) N_{ft}=0

(b) N_{ft}=2

Fig. 3 Effect of water content on freezing point of saline soft clay



(a) N_{ft}=0

(b) N_{ft}=2

Fig. 4 Effect of salinity on freezing point of saline soft clay

Fig. 4 gives the relationship between freezing point of saline soft clay and saline content before and after freeze-thaw cycling. Visually, the freezing point of soil specimens decreases approximately linearly at various water contents and it exhibits similar changes to the freezing point of calcium chloride solution at salinities that have been considered in these tests. As for the calcium chloride solution that has been used in testing, its freezing point also shows an approximately linear relationship with the salinity, as can be found from the freezing curves. Here, the calcium chloride solution can be assumed as a limit when the water content of specimens approaches the infinity. Experiments by Bing and Ma (2011) indicate that the inclusion of soluble salt in soil-water system enhances the adsorptive actions of soil particle surface and soil-water potential can also be reduced with cementation of soluble salt, and absorption, substitution and dispersion of ions, which further enhances the difficulty in ice-water phase change and thus lowers the freezing point of soils.

3.3 Effect of freeze-thaw cycling on freezing point

Test data for soft clay at a given saline content of 0.5% is taken as an example to illustrate the relationship between freezing point and water content of saline soft clay that experienced various cycles of freeze-thaw, as shown in Fig. 5. It demonstrates that the freezing curves for saline soft clay specimens all exhibits similar changes as the water content varies, i.e., freezing point grows rapidly at lower water contents, with a larger increment while it is relatively slow at higher water contents. Besides, the freezing point of saline soft clay increases as freeze-thaw cycling proceeds. Three combinations of water-salt system were selected to further reveal the effect of freeze-thaw cycles on the freezing point. Easily we have found the freezing point of soil specimens tends to grow after more cycles of freeze and thaw, especially the initial five cycles and afterwards little change can be observed. This may be related to the remolding of soil pore structure that has been disturbed by freeze-thaw cycling (Yao *et al*. 2009, Qi *et al*. 2006).

Fig. 6 gives the deformation of saline soft clay at three water-salt combinations and it is noted that in the initial ten cycles of freeze and thaw, the deformation fluctuated over time and ten cycles can be easily found, over the deformation induced by freeze-thaw cycling growing with time as a whole. Also, the deformation of specimens caused by the initial five cycles of freeze and thaw accounts for a major part of the total deformation and afterwards the deformation due to both frost heave and thaw consolidation tends to stabilize and smaller increment of residual deformation can be noticed, indicating that the soil structure stabilizes. The specimens that have been used in testing have a density close to the maximum, and thus after freeze and thaw cycling, this kind of densely compacted soils generally tends to loosen, which has been verified by experiments on muddy clay and two characteristic indexes identifying soil structure evolution, i.e., pore size and density of pores, both increases after freeze-thaw (Tang *et al*. 2012).

To highlight the influence of freeze-thaw cycling on soil structure, the residual deformation after each cycle was

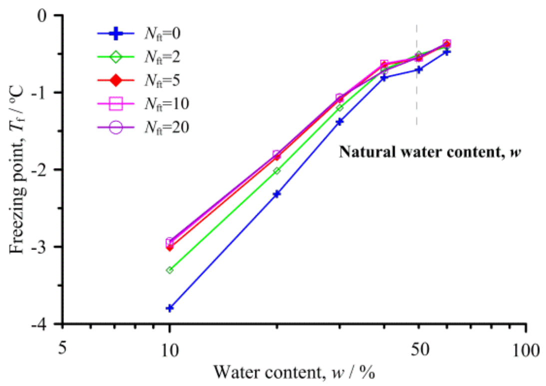


Fig. 5 Relationship between freezing point and water content at various freeze-thaw

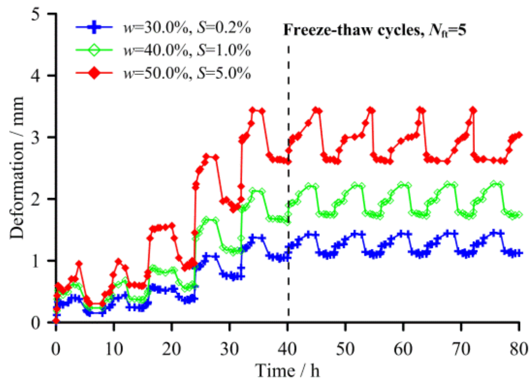


Fig. 6 Freeze-thaw deformation of saline soft clay at three water-salt combinations

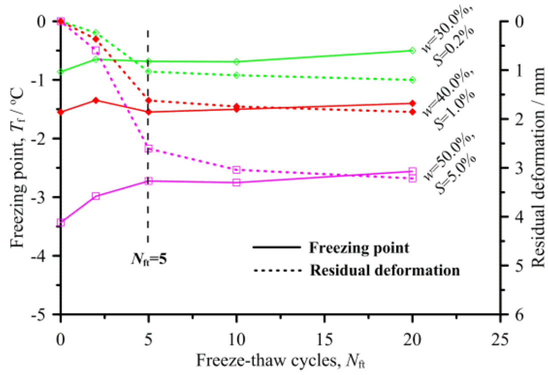


Fig. 7 Correlation of freezing point and residual deformation during freeze-thaw cycling

collected from test data and was plotted in a graph, together with the development of freezing point, as presented in Fig. 7. It implies that the two characteristic indexes are closely correlated in that for the two curves with similar shapes, the break points for both the freezing point and residual deformation appeared at five cycles of freeze and thaw. Within the preset cycles, the porous areas expanded due to loosened soil structure and lower compression emerged on soil water, as suggest by Sterpi (2015), and it also proves that the changed porosity of soils is strongly dependent on the degree of compaction. We can also find clues from the work by Guan *et al*. (2014a) and Guan (2014b) that the higher stress significantly lowers the freezing point of soils.

3.4 Degree of supercooling

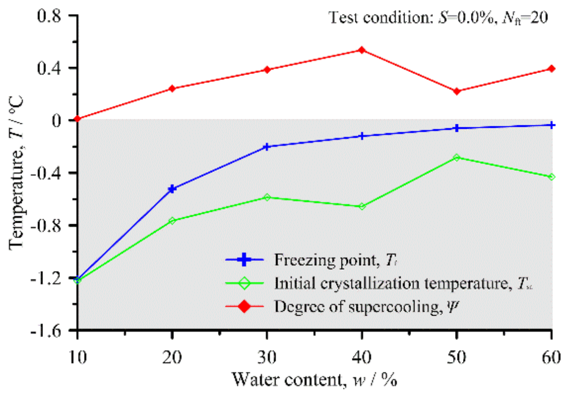
The degree of supercooling characterizes the process of lowering the temperature of a liquid below its freezing point without it becoming a solid. Here we introduced the concept of degree of supercooling, Ψ, to illustrate the amplitude of temperature transition at the second stage during freezing

$$\psi = T_f - T_{sn} \tag{2}$$

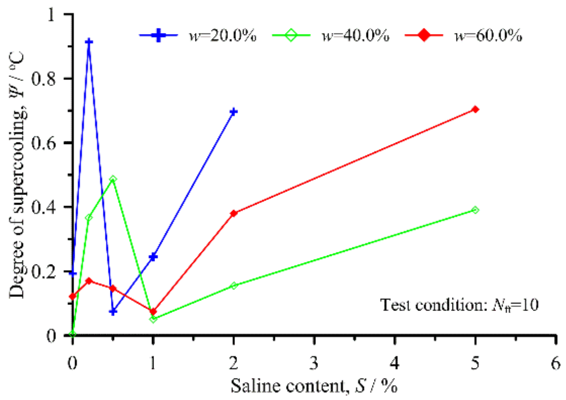
Both the freezing point and initial crystallization temperature were obtained from test results and the degree of supercooling was calculated based on Eq. (2). Fig. 8(a) gives the degree of supercooling for the saline soft clay at various water contents. It shows that both the freezing point and initial crystallization temperature increase in general at higher water contents, with the degree of supercooling grows initially and followed by the reduction. Besides, the relationship between supercooling degree and saline content at given water contents, as shown in Fig. 8(b), indicates that the salinity obviously influenced the degree of supercooling and a critical saline content may exist, i.e., the degree of supercooling grows continually with salinity higher than the limit while it fluctuated at lower saline contents. Test data with four combinations of water-salt system was selected to illustrate the dependence of the degree of supercooling on the cycles of freeze and thaw, as presented in Fig. 8(c). The degree of supercooling shows complex variations with freeze and thaw cycling, and it in general decreases rapidly in the initial five cycles and stabilized afterwards, approaching the zero centigrade. It indicates that the freezing point is close to the initial crystallization temperature. This can be explained by the Gibbs-Thomson equation for the ice-water phase change. As for the capillary water, the parameter, *T*, as presented in Eq. (1), represents the initial crystallization temperature, T_{sn}, which strongly depends on the diameter of capillaries when the supercooling of soil water occurs. The diameter of capillary is one of the important indexes representing the soil porosity characteristics, and experiments have proven that the porosity of soils can be obviously changed by freeze and thaw (Qi *et al*. 2006). For densely compacted specimens, the soil structure tends to loosen and correspondingly the capillary diameter tends to grow, which also supports that the initial crystallization temperature increases with the void ratio, and the degree of supercooling approaches to zero centigrade. This is consistent with the experiments that were carried out in this paper.

3.5 Empirical equation for predicting freezing point of saline soft clay

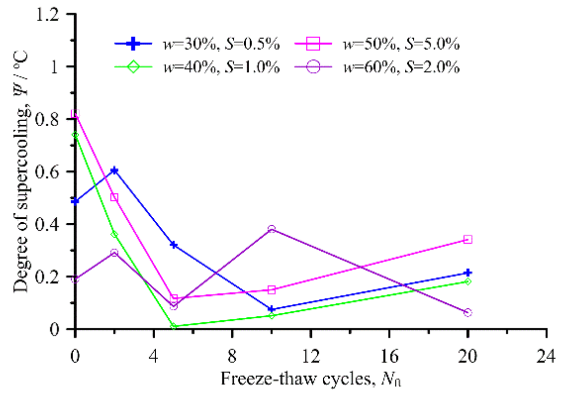
Test data for saline soft clay at various water contents implies that the unfrozen water approaches to a minimum value when the water content approaches zero, and in this case the freezing point will approach infinity, i.e., enhancing the difficulty in freezing while as the water content grows to the infinity, i.e., the content of soil particles minimizes, the freezing point of calcium chloride solution will be the limit for soft clay with calcium chloride. Considering this feature, the water content was replaced by a new parameter by introducing the plastic limit, w_p, i.e., $\bar{w} = w/w_p$.



(a) Effect of water content

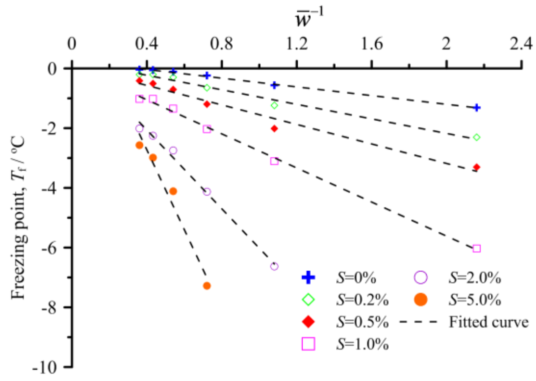


(b) Effect of saline content

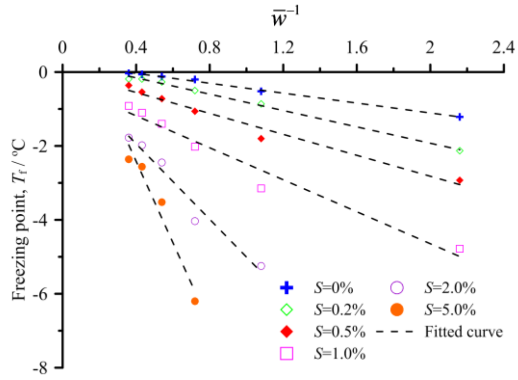


(c) Effect of freeze-thaw cycling

Fig. 8 Variation of the degree of supercooling



(a) N_{ft}=2



(b) N_{ft}=20

Fig. 9 Relationship between freezing point and water content

Fig. 9 presents the relationship between freezing point, T_f and $\bar{w}$. Clearly it shows that for the selected two cycles of freeze and thaw, the freezing point for saline soft clay varies approximately linearly with the parameter $\bar{w}^{-1}$, and thus the following equation can be used

$$T_f - T_0(S) = b\bar{w}^{-1} \tag{3}$$

where, $T_0(S)$ and b are intercept and slope of the curves. It is noted that the intercept of the freezing curves, $T_0(S)$, will represent the freezing point of saline soft clay at an infinite water content, i.e., calcium chloride solution, and can be determined by the freezing curve illustrated in Fig. 4.

The two fitted parameters, $T_0(S)$ and b, were plotted in a graph with saline content, S, as shown in Fig. 10. For the parameter b, the following equation can be obtained

$$b - b_0 = mS \tag{4}$$

where, b_0 and m are intercept and slope of the curves in Fig. 10. Then plot the two parameters in a graph with the cycles of freeze-thaw included as the lateral axis, as shown in Fig. 11. It clearly shows that the parameter b_0 exhibits little change within the preset cycles of freeze-thaw, and can be taken as a constant while the parameter m decreases with the cycles and an exponential equation can be fitted

$$m = \alpha e^{\beta \bar{N}} \tag{5}$$

with $\bar{N} = N + 1$.

As for the parameter $T_0(S)$, we can use a proportional function for simplicity

$$T_0(S) = \gamma S \tag{6}$$

Note that the effect of freeze-thaw cycling on the physical properties of calcium chloride solution is neglected here and its freezing point is regarded as a parameter simply dependent on the saline content. Thus, the following empirical relationship can be derived

$$T_f = \left[\alpha e^{\beta \bar{N}} S + b_0\right]\bar{w}^{-1} + \gamma S \tag{7}$$

where, the parameters above can be easily obtained from test results. For the saline soft clay used in testing, α=-2.000, β=-0.021, γ=-0.48, b_0=-0.592.

The freezing point at various test conditions were

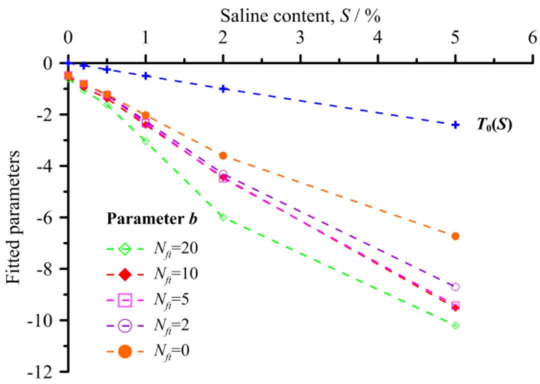


Fig. 10 Relationship between fitted parameters and saline content

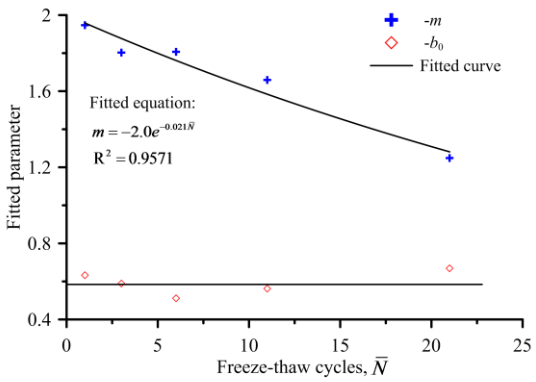


Fig. 11 Correlations of b_0 and m vs. freeze-thaw cycles $\bar{N}$

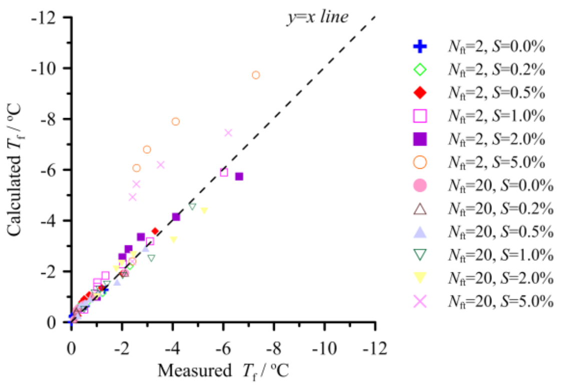


Fig. 12 Comparison of calculated and measured data

calculated based on the above equation. Comparison of calculated and measured data was illustrated in Fig. 12. It shows that most of the points lies around the y=x line indicating that the calculated freezing point agrees well with test data. It also proves that the empirical equation is reliable for predicting the freezing point of saline soft clay when both complex water-salt systems and freeze-thaw cycling are involved.

4. Conclusions

This paper carried out laboratory experiments on the freezing point of saline soft clay at five levels of salinities produced by the calcium chloride solution. The effect of water content, saline content and freeze-thaw cycles on the freezing point of soil specimens were discussed. The primary conclusions are as follows.

- Four characteristic stages can be observed from soil freezing curves of saline soft clay, i.e., supercooling, abrupt transition in temperature, equilibrium freezing and continual freezing. The four stages are closely related to the variation of unfrozen water content in soils as the thermal state changes.
- The freezing point of saline soft clay tends to grow at higher water contents, and a critical water content can be noted between the plastic limit and natural water content. Besides, the freezing point decreases almost linearly with the saline content, and also varies with freeze-thaw cycles, especially in the initial five cycles. This strongly depends upon the stabilized soil structure during freeze-thaw cycling. This suggests that the freezing point testing should pay more attention to the effect of pore characters.
- The degree of supercooling characterizes the process of lowering the temperature of a liquid below its freezing point without it becoming a solid. It was generally calculated based on the two characteristic points in soil freezing curves such as the freezing point and the initial crystallization temperature. It shows complex correlations with water content, saline content and freeze-thaw cycles.
- Finally, an empirical equation was proposed taking into account the coupled effect of water, salt and freeze-thaw cycling. Comparison of calculated and measured data proves a good fitting accuracy. This may provide guidance for engineering design and numerical analysis in frozen soil engineering, especially when dealing with the adverse condition occurred during artificial freezing, e.g., the brine leakage.

Acknowlegements

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