

Experimental Study on Pore Water Pressure and Microstructures of Silty Clay Under Freeze-Thaw Cycles



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Abstract The variations on physical properties and microstructures of soils are essential to explore the freeze-thaw mechanisms. We do some experiments on freeze-thaw test of Qinghai-Tibet silty clay and cryo-scanning electron microscope (cryo-SEM) observing after freeze-thaw test and monitoring the changes of temperature, pore water pressure, and displacement. The results showed that the change of pore water pressure was closely related to soil particles rearrange, particle size redistributing, and then develop a new arranging tendency of soil pores during freeze-thaw cycles. During the period of soil freezing, the pore water pressure decreased gradually, and the particles became more gathering from point to point ($P-P$) form along with freezing rate decreasing, the relevant microstructures had changed from the matrix structure to gel structure, and pores became more simplex. During soil melting, the pore water pressure rose to stability, and the soil mainly experienced a consolidation process. In our study, we also quantify cryo-SEM images by Imagepro Plus (IPP) software. The results reflect the change of pore water pressure had a certain correlation with structural parameters under freeze-thaw cycles. These findings substantially contribute to understand the freeze-thaw mechanism and assist in upscale the microscale physical characteristic of frozen soil.

Keywords Frozen soil · Freeze-thaw mechanism · Cryo-scanning electron microscope · Microstructure · Pore water pressure · Structural parameters

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1 Introduction

In cold regions, soils are commonly exposed to freeze-thaw cycle every year [1]. This phenomenon has a significant effect on the service performance of engineering facilities [2, 3]. As a specific form of temperature change, the freeze-thaw cycle is a strong weathering process that can remarkably change the physical properties and microstructure of soil [4]. During freezing, soil typically subjected to frost heave which involves the phase change of water, water migration, and ice segregation. Also, consolidation of soil typically develops in the thawing process [5]. Fine-grained soils influenced by freezing and thawing show changes in volume [6], in strength and compressibility, dry density, densification, unfrozen water content, pore water pressure, bearing capacity, and microstructure [7–11]. Virtually, when considering soil physical behaviors and microstructural transformations due to freezing and thawing, the balance of pore water pressure in the soil had a significantly affects to reveal these phenomena [12].

Investigation of the pore water pressure during soil freezing and thawing is essential when exploring the mechanisms of freeze-thaw cycle, which revealing the driving force of water migration, the mechanisms of frost heave, the validity of the Clausius-Clapeyron equation, and the consolidation phenomenon of soil [13]. In particular, the consolidation phenomenon in the freeze-thaw cycle is a result of an increase in effective stress due to a decrease in pore water pressure [14, 15]. And in an attempt to reveal this variation, researcher focuses on the consolidation pattern in the different stages of soil freezing based on the observed pore water pressure and its numerical simulations, proposes that consolidation in the unfrozen zone during soil freezing includes compression-induced consolidation which results from an increase in frost heaving stress, also vacuum induced consolidation which results from a decrease in pore water pressure [12]. Typically, the growth of ice lenses results in substantial decreases in pore pressure and ultimately dewatering and unsaturation in the unfrozen zone during closed system tests without an external water supply [16]. Moreover, pore water pressure during freezing was measured and could be correlated to the subsequently observed volume and water changes [17]. The changing of pore water pressure shows that the water moving is an important influencing factor on soils by its action on structure development, deformation, particle translocation, and consolidation under the freeze-thaw cycle.

Meanwhile, the frozen soil microstructure analysis has been more important [18, 19]. During the freeze-thaw consolidation, the soil became fissured and jointed [20], and ices in various sizes and shapes tend to segregate in soils resulting in the formation of a characteristic in micro- and macroscales [21]. After the melting of soil or thaw consolidation, the inter-fissures are still open, leading to gel or closed-cell macroscopic soil structure [22]. In the condition of alternating freezing and thawing, the frost heave and consolidation occur together with more or less developed particles and aggregate broken, leaded to compaction, displacement, rotation, and formation of the soil microstructure, further affect the shape and size of the aggregates and pore distribution [23–26], giving rise to different types of microstructure formation

Table 1 Physical properties of silty clay

Soil property	Liquid limit, L_L (%)	Plastic limit, L_P (%)	Plasticity index, P_I
Value	24.8	15.0	9.8

[10, 27, 28]. The resulting change in a microstructure, in turn, affects the water moving on soil during frozen frost and thaw consolidation.

Although the significance of pore water pressure and microstructure change during freeze-thaw cycle has been widely acknowledged, few information has been reported on the relationship between pore water pressure and microstructure under the freeze-thaw cycle. Therefore, we examined the pore water pressure during freeze-thaw cycles of the silty clay in the Qinghai-Tibet of China, after that, investigated the microstructure of each layer of soil. By clarifying the variation in the pore water pressure and microstructure under freeze-thaw cycles, we expect to found a certain relationship to provide a reference to understanding freezing and thawing mechanism.

2 Experimental Study

2.1 Materials and Specimen Preparation

In this study, silty clay obtained from Qinghai-Tibet Plateau is subjected to do freeze-thaw tests. The main physical properties of this type of silty soil are given in Table 1. It was classified as CL (Low liquid limit Clay) per Unified Soil Classification System. The grain size distributions of the silty clay are presented in Fig. 1. The uniformity coefficient and the coefficient of curvature of the silty clay is 1.24.

The soil samples were prepared and tested at the State Key Laboratory of Frozen Soil Engineering CAS. Before making soil specimens, the soils were air-dried, sieved with an aperture of 2 mm. And then, the soils were slurry mixed with certain water content (21.4%) and left 24 h in a sealed plastic bag to ensure adequate water redistribution. The samples were compressed into a cylindrical Perspex cell with an inner diameter of 101 mm and a height of 140 mm, witch initial density was 1.69 g/cm^3 .

2.2 One-Dimensional Freeze-Thaw Test

The one-dimensional freeze-thaw test system consists of an insulated environmental chamber, a specimen cell, two cryostats with the temperature accuracy of $\pm 0.1 \text{ }^\circ\text{C}$, and various sensors necessary for measuring soil temperature, pore water pressure, displacement, and water intake (Fig. 2). A cylindrical Perspex cell with lateral thermal insulation was placed in the test machine box. Two columns of holes were drilled in the wall of the cell. One column contained night holes for installing the temperature

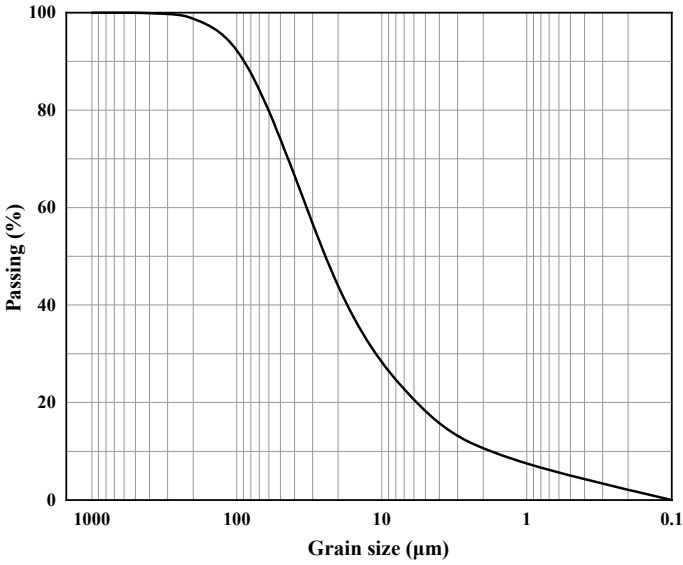


Fig. 1 Grain size distribution of silty clay

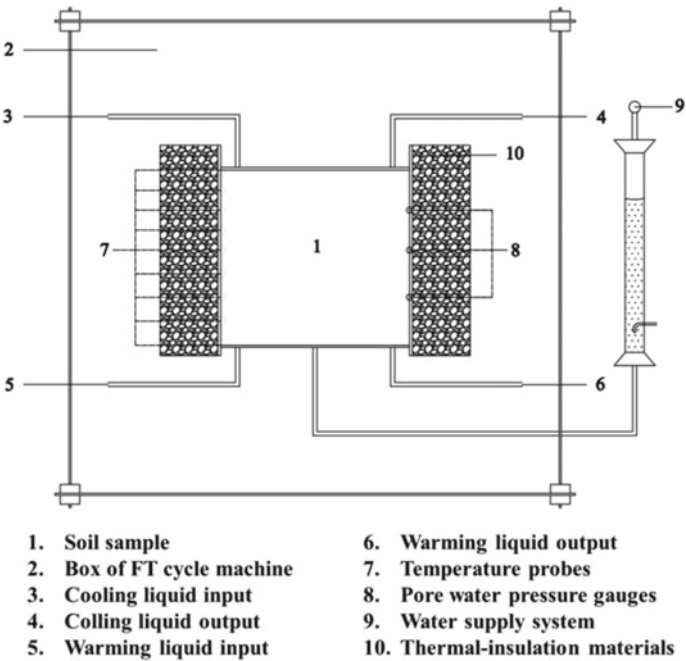


Fig. 2 Sketch of the one-dimensional freeze-thaw test system

probes at intervals of 15 mm, the high-sensitivity temperature probes with a precision of 0.05 °C and a valid temperature range of ±30 °C, and was manufactured by the State Key Laboratory of Frozen Soil Engineering [29]. Meanwhile, the other side’s holes of the specimen cell for inserting the pore water pressure transducer into the soil at different depths (see Fig. 3), which has a measuring range of ±100 kPa with a reference pressure point and an accuracy of ±0.1% full scale [12]. Moreover, the freeze-thaw tests were performed in the open soil-water system, using a Marriot bottle that monitored changes in water volume in real time.

To obtain conventional laboratory testing data, the specimens with test conditions were set for the freeze-thaw cycle. First, the prepared sample was placed into the cylindrical Perspex cell of the freeze-thaw cycling test machine. Next, the sensor of the temperatures, pore water pressure gauges, and water supply gauge were installed. And, all installed sensors were set to take measurements at 30 s intervals. Finally, the freeze-thaw experiment was conducted by controlling the top and bottom plate temperatures, as shown in Table 2.

Fig. 3 Holes arrangement of the sample

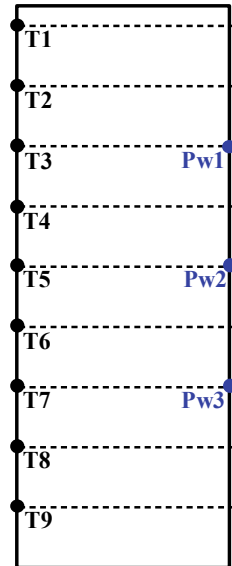


Table 2 A single freeze-thaw cycle test conditions

Soil type	Temperature at the cold end, °C	Temperature at the warm end, °C	Duration of freezing, h	Thawing temperature, °C	Duration of thawing, h	Water supply
Silty clay	-5	1	13	Room temperature	13	Yes

2.3 *Cryo-SEM Technique and the Microstructure Observation*

Cryo-SEM technique makes it possible to examine wet samples and to preserve their natural characteristics for further testing, which is an obvious advantage compared to conventional SEM [30]. Such direct imaging, in which, can transform the water into ice whose crystalline domain dimensions do not exceed those of the finest details that can be observed, and thus do not modify the morphology of the samples at the observation scale.

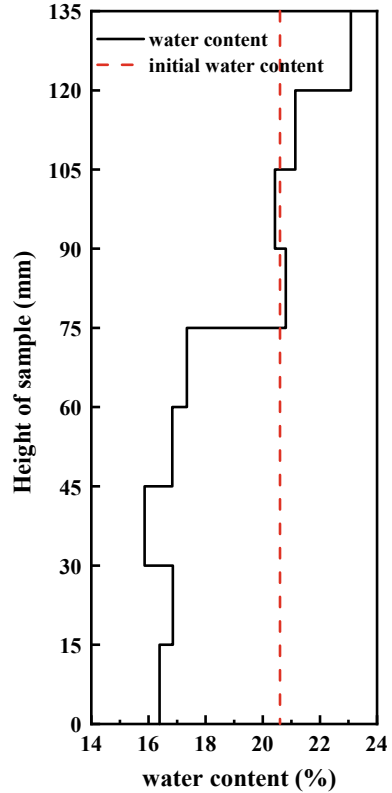
The samples of microstructure observation from laboratory tested specimen subjected to freeze-thaw cycle, which was layered every 15 mm. Given that the specific samples of cryo-SEM observation were stuck to a 5 mm hollow copper column. The sample was then hand-plunged into a nitrogen slush using sample holder. The plunge-frozen sample was then retrieved with a pre-cooled vacuum-cryo-transfer shuttle and transferred to a freeze-fracture system, where it was maintained at $-140\text{ }^{\circ}\text{C}$ and a pressure of 1.7×10^{-5} Pa. A layer of surface ice was removed by allowing the sample temperature to rise to $-90\text{ }^{\circ}\text{C}$ and holding the temperature at $-90\text{ }^{\circ}\text{C}$ for 5 min before returning the sample to $-140\text{ }^{\circ}\text{C}$. Finally, the sample was coated with gold under cryogenic conditions, and transferred sample to a cryo-SEM using sample holder. Then, the sample was imaged at $-140\text{ }^{\circ}\text{C}$ using an accelerating voltage of 10 kV and secondary electron detector.

3 Typical Results and Analysis

3.1 *Redistribution of Water*

There are some evidences that moisture redistribution continues within the frozen soil in the open soil-water system. Figure 4 presents the water content profiles of silty clay after the freeze-thaw cycle. It is apparent that the moisture significantly redistributed during freeze-thaw cycle. For example, water content at the bottom of the soil sample was 16.4% and at the top was 23.1%. Especially, it can be noticed that the gap at the height of 75 mm became bigger and with much higher moisture content than initial water content. It can be recognized that movement of water during freezing of this region soils causes the development of channels with provided water to the frozen regions and the system of channels benefits to remove water from the soil during the thawing process. In general, when a sudden negative temperature was applied to the top surface of the soil sample, unsteady heat flow is initiated. The freezing front progresses into the soil as a function of the imbalance of the heat and pore water freezes in situ. At the same time, a suction gradient develops in the frozen zone in response to any temperature gradient and water migrates from the unfrozen soil into the frozen zone. And the freezing-induced suctions further consolidate the unfrozen soil since its water content decreased from 21.4 to 16.4%. With the increase of sample

Fig. 4 Water content profiles after freeze-thaw cycle



temperature, the ice of soil was melting, and the pore volume decreased as the self-weight of overlying soil, thus also caused water migration and the consolidation of the sample.

3.2 The Change of Pore Water Pressure

Figure 5 illustrates the changes in the relationships among the pore water pressure, temperature, and time during the freeze-thaw cycle. Similar variation trends of pore water pressure with different soil sectors may be found in the silty clay at the same moment. When considering the freezing process, pore water pressure was generally divided into three stages: during the first stage, the pore water pressure increased as the temperature decreases. During the second stage, the pore water pressure decreased at first and then increased. During the third stage, the temperature quickly decreased from 5.65 to $-2.26\text{ }^{\circ}\text{C}$, the pore water pressure became to the negative value. It was also noted from Fig. 5 that the pore water pressure tended to 0 kPa from negative value as the temperature increased during thawing process. Based on the results, we

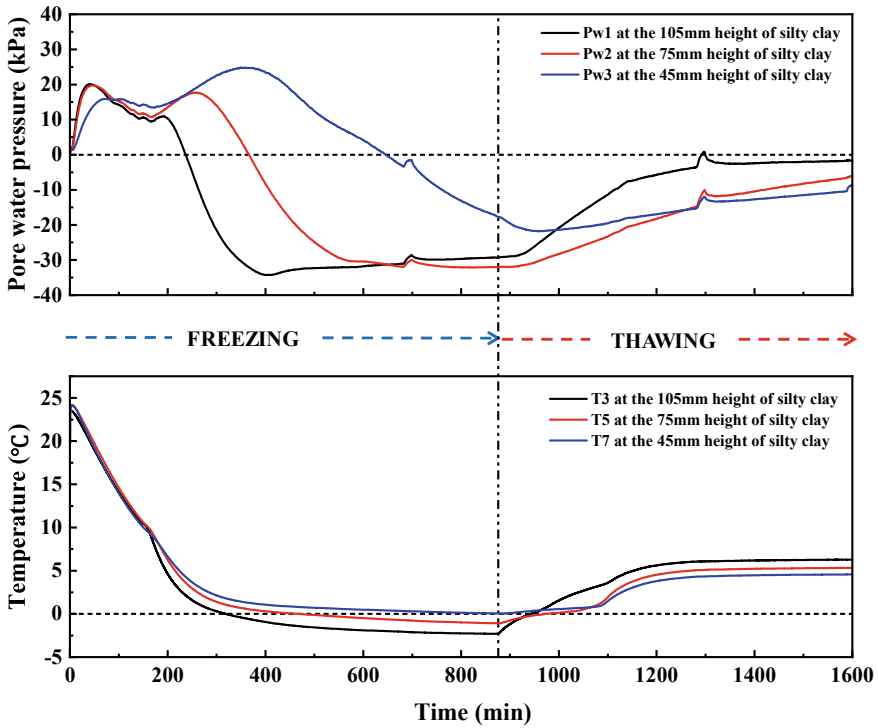


Fig. 5 Variation of the pore water pressure of silty clay during freeze-thaw cycle

concluded that the changes in pore water pressure were attributed to the influence of the phase change of water. At the beginning of experiments of silty clay, the lower temperature made the capillary water first turned into ice, causing a change in the capillary hydrostatic pressure. And then, the thickness of the unfrozen water film and adsorption potential in the soil sample changes because of adsorbed water is transformed into ice. During the process of ice melting, the radius of curvature of the ice–water interface increased, which changed the capillary and adsorption potential causing the pore water pressure increases.

The Pw1, Pw2, and Pw3 pore water pressure probes were located at different soil heights during the freeze-thaw experiment of the silty clay samples, as shown in Fig. 5. Although the pore water pressure at different heights presented similar tendency, it was clearly found that the dissipation of the Pw1 pore water pressure occurred earlier and faster than that of Pw2 and Pw3 pore water pressure. For example, the Pw1 pore water pressure decreased to -34.2 kPa from 19.7 kPa during the freezing process and then increased to -2.7 kPa along with temperature increased. While the Pw3 pore water pressure changed from 24.7 kPa to -21 kPa, and finally increased to -9.1 kPa. So, we concluded that the more rapid dissipation of the pore water pressure occurred near the freezing front, and the dissipation of the pore water

pressure proceeded from the top to bottom as the water migrated from the unfrozen to frozen zone.

Therefore, the differences in the pore water pressures may result from a combination of two factors. First, the change of pore water pressure during the freeze-thaw cycle due to the transformation of the ice–water phase. Second, the dissipation of the pore water pressure resulted from the migration of water in the soil sample.

3.3 Analysis of Microstructure

Freeze-thaw cycles also have been shown significant influence on soil microstructural transformations. Commonly, the change in soil structure due to freezing and thawing is basically caused by the phase transformation of pore water and water distribution. Therefore, structural form following freeze-thaw was dependent on both temperatures of the top sample and the water migrating at the time of the freeze-thaw cycle. Indeed, when considering soil deformation, soil samples that have been subjected to unidirectional freezing are generally divided into three zones: the frozen zone, the frozen fringe, and the unfrozen zone [12]. And according to the tested temperature and pore water pressure, such zones correspond to the 105, 75, and 45 mm heights of the specimens.

At the top of the specimen subjected to fast freezing conditions, the restricted water flow and resultant in situ growth of ice crystals are usually considered to be structurally destructive, can strongly affect the type of ice forms and structure in the soil. Besides that, the water content at the time of freezing is also an important control in determining the effects of pore ice formation, shown in Fig. 6, which was the cryo-SEM image at the 105 mm height of silty clay after freeze-thaw cycles.

In this region, because of the strong temperature gradients, upward moisture flow is minimized as the rapid advancement of the freezing front does not have sufficient time for appreciable pore water moving, leading to frozen in situ. As such, the approximately 9% volumetric expansion resulting from ice formation has been credited with rupturing existing aggregates and increasing the proportion of fine

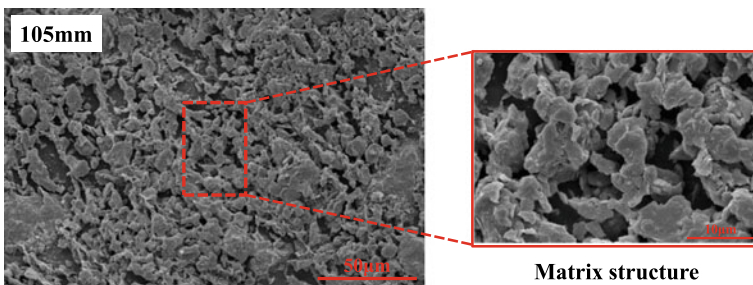


Fig. 6 Cryo-SEM image at the 105 mm height of silty clay

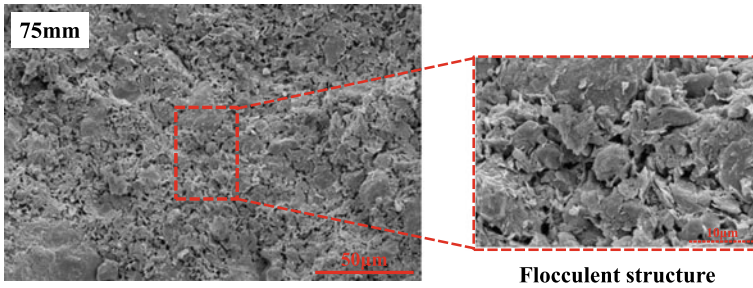


Fig. 7 Cryo-SEM image at the 75 mm height of silty clay

material [17]. Meanwhile, the ice crystal growth would tend to shatter aggregates into smaller particles and increase the size of inter-aggregate pores, resulting in the formation of a loose structure on the soil. With the further dropping of temperature, ice would eventually invade the successively smaller intra-aggregate micro-pores. Immediately the following thaw, large decreases in pore ice alter the pores on soil through an alteration of the particle connection, and leading pores between particles have good connectivity. Cryo-SEM shows that the volumetric expansion due to the phase change of water increased the pore volume on the soil.

Under these conditions, analyses of the frozen zone have noted a typical matrix structure. In which, the freeze-thaw cycle commonly results in point–point ($P-P$) form mostly consisting of smaller aggregates and particles. And, less continuous intra-aggregate micro-pores are separated by inter-aggregated macro-pores developed more complex pore system.

As the moving of the freezing front to higher temperature bottom, a weaker temperature gradient usually occurs at deeper levels within the sample. It is thus highly likely that consolidation occurs in front of the $0\text{ }^{\circ}\text{C}$ isotherm and in the unfrozen or partly frozen regions between ice accumulations in freezing clay [31]. So, the mineral skeleton adjacent near this area underwent appreciable compression, shown in Fig. 7, which was close to freezing front, represents the structural characteristics of the frozen fringe regions. The whole system shows the flocculent structure of this area which were more complicated, individual particles and smaller aggregates arrange in a random orientation rather than matrix structure by face–face ($F-F$) and face–edge ($F-E$) contacts. These results indicate that the presence of a pre-existing soil structure provides intra-aggregate and inter-aggregate pathways for thermally induced water flow and zones of weakness in which ice can form. At the boundary of freezing on soil of this region, coupled heat and moisture flow generate a tension to draw pore water toward the point of ice nucleation, resulting in a drastic phase change of water, as the inter-aggregate pore plays an important role in the water migration. As the films of bound water around the particles become thinner, the particles draw closer and their interaction increasing, their structural bonds become stronger, resulting in bigger aggregate happens. Meanwhile, if unfrozen pore water can continue to slowly migrate toward frozen zone through this area, it may contribute to weak the

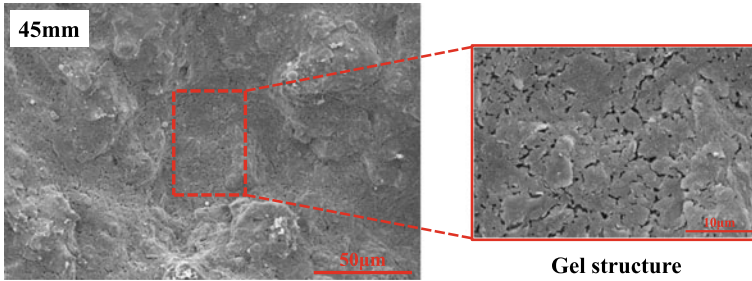


Fig. 8 Cryo-SEM image at the 45 mm height of silty clay

connection between particles. As Leo [32] noted that as a dramatic region of ice and water phase transition, many small crystals associated with pore ice destroyed the microstructure of the soil by destroying the micro-pores and increased aggregation through compression.

Figure 8 shows the gel structure at the 45 mm height of silty clay, which typically represents the unfrozen region after freeze-thaw cycles. The structure on unfrozen region commonly subjected to compaction because of overburden pressure and pressures induced by growing ice crystals, which have been characterized as hardening and curing. At the hardening and curing, the water migration is thought to be a major controlling factor through its influence on particle rearrangement. During rearrangement, this close proximity between soil particles is also thought to promote the colloids and face-face ($F-F$) form development. In the freezing process, pore water is drawn toward growing ice crystals, leading particles gathering and stacking on unfrozen zones. While water transfers from the unfrozen to the freezing region, the mineral skeleton of the unfrozen part becomes shrunken irrespective of the rate of water inflow to the soil sample. Desiccation by the removal of moisture from within the clay inter-layer during the growth of pore ice which stable aggregates may be formed during the freezing and thawing process. Moreover, on unfrozen regions lying rather far from the freezing boundary, the pore characteristic does not change much, and inter-granular pore most development.

3.4 Relationship Between Macroscopic Phenomena and Microstructure

It is commonly hypothesized that the freezing-induced volumetric expansion of pore water is responsible for aggregate breakdown [31] and it has also been confirmed that the freeze-thaw cycles had a significant effect on microstructure on soils. We further provided a quantitative analysis of microstructure to explore the relationship between pore water pressure and structural parameters, to understand the freeze-thaw cycle mechanism.

Cryo-SEM images of different heights of sample were presented in Figs. 6, 7 and 8. A view magnification of $\times 1000$ was selected, and the image was employed to quantitative analysis using Photoshop and Image-pro Plus(IPP) software. Images were all converted a 2-bit, a black-and-white image format for examination of particle distribution and pore characteristics in the soil sample (shown, e.g., as Fig. 9). Particles were turned white, and the pores were colored black. After that, the structural parameters were extracted, and the pore areas also be calculated.

Figure 10 shows the particle properties, including clay content and accumulated fractal dimension of particle distribution. Generally, the higher fractal dimension means the more dispersed of particle distribution, the lower gathering of particles, and the more complex of distribution pattern, which reveals the particle distribution in the plane. For the height of 75 mm on specimen which has higher fractal dimension and the highest clay content. For example, the fractal dimension of the particles was 1.24, and the clay content was 30.33% of this region. Obviously, the effect of freeze-thaw cycle on the behavior of the particle is very significant. Comparing the

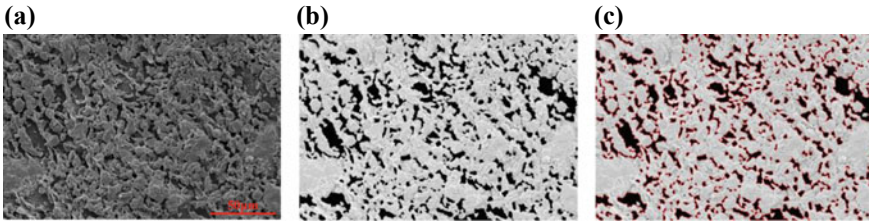


Fig. 9 A cryo-SEM image of the 105 mm height that was used for the analysis of example section **a** cryo-SEM image; **b** cryo-SEM image after thresholding by the Photoshop; **c** quantitative analysis using by IPP

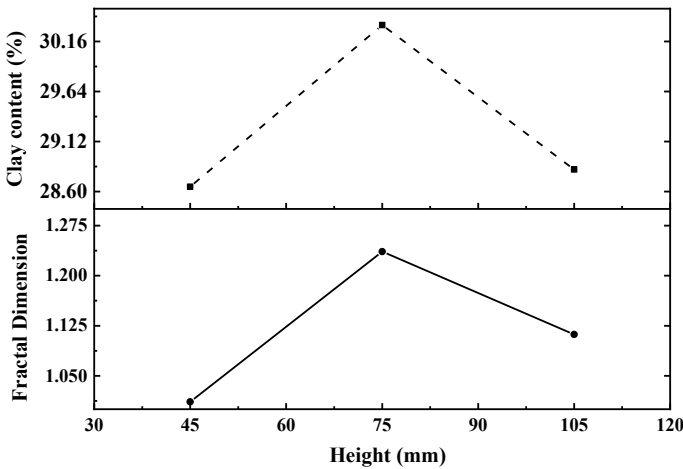


Fig. 10 Particle properties analysis at different heights of sample

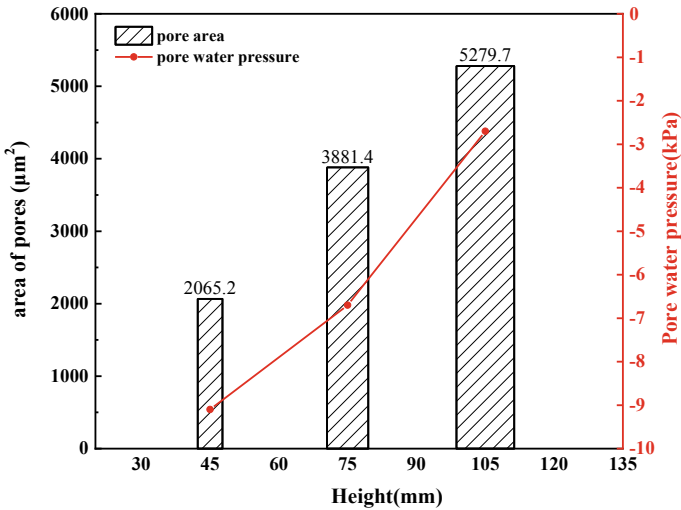


Fig. 11 Area of pores at different heights of sample

change of pore water pressure in this region during the freeze-thaw cycle, a positive relationship between structural parameters of particles and pore water pressure can be defined. More specifically, when there is a bigger gap variation of pore water pressure in this region during the freeze-thaw cycle, the structure is more chaotic and the structural parameters become larger. It is quite obvious that the clay content and fractal dimension present lower and the variation amplitude of pore water pressure is smaller on the else height of soil.

The change of pore has reflected an alteration of the arrangement of mineral particles within the soil. Figure 11 shows the pore area and pore water pressure values at the different height of silty clay after freeze-thaw cycles. The change of the pore area after the freeze-thaw cycles is discussed and a certain relationship between pore water pressure values is also determined. As can be seen from the histogram, the frozen zone of the sample corresponds to a much more pore distribution. Meanwhile, the area of pores decreases at the lower height of the specimen, in which has a positive correlation with changes in pore water pressure. Such as at the 105 mm height of silty clay, the pore water pressure value was -2.7 kPa. While at the 45 mm height of soil, the pore water pressure value was -9.1 kPa.

4 Conclusions

In this paper, the pore water pressure and microstructure under freeze-thaw cycle was tested. Through the information obtained from the laboratory data and cryo-SEM

images, the relationship between pore water pore pressure and structural parameters is preliminary analysis. The following points are drawn based on this study:

- (1) During freezing and thawing periods, the pore water pressure changes are complicated along with temperature first decreased and then increased. Meanwhile, the microstructure has a significant change at the different height of the specimen. Typically, the bigger gap of pore water pressure values variation, more complex structure, higher clay content and fractal dimension of the particle were more sensitive at the 75 mm height on soil to freeze-thaw cycle than other regions.
- (2) In the process of soil freezing and thawing period, there was a certain relationship among particle properties, pore area, and pore water pressure of soil. The magnitude of the change in pore water pressure is positively correlated with the structural parameters of the particles during the freeze-thaw cycle on the timescale. While, the pore water pressure value is positively correlated with the pore area after the freeze-thaw cycle on the spatial scale.

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