Effects of Freeze–Thaw Cycles on the Mechanical Properties and Microstructure of a Dispersed Soil

Shurui Zhang 1, Xin Xu 1,*, Xiaoqiang Dong 2, Haomin Lei 1 and Xun Sun 1

1 College of Construction Engineering, Jilin University, Changchun 130026, China; zhangsr2420@mails.jlu.edu.cn (S.Z.); leihm22@mails.jlu.edu.cn (H.L.); sunxun21@mails.jlu.edu.cn (X.S.)
2 College of Civil Engineering, Taiyuan University of Technology, Taiyuan 030024, China; dongxiaqiang@tyut.edu.cn
* Correspondence: xuxinjl@jlu.edu.cn

Abstract: Dispersed soil is highly sensitive to water and can easily disperse in low-salt water, leading to weakened structures and engineering issues. To investigate the freeze–thaw effects on the mechanical properties and microstructure of dispersed soils in western Jilin Province, we simulated 0, 1, 3, 5, 7, 9, 12, and 15 freeze–thaw cycles. Qualitative and quantitative analyses were performed using numerous methods, including soluble salt determination, density and water content determination, particle size analysis, dispersion identification tests, and scanning electron microscopy (SEM), to investigate the mechanism of deterioration in soil mechanical properties from various perspectives. The research findings indicate that the unconfined compressive strength (UCS) decreased from 156.843 kPa in the unfrozen state to 76.961 kPa and then stabilized. The freeze–thaw action resulted in particle fragmentation, increased soil porosity, and elevated crack content, thereby contributing to soil structure deterioration and strength reduction. Furthermore, the cohesion value (c-value) gradually decreased from 22.196 kPa in the unfrozen state to 7.997 kPa and then stabilized. The angle of internal friction (ϕ-value) started at 7.514°, peaked at 9.514°, and gradually declined. This comprehensive study provides valuable insights into the variations in soil mechanical properties under freeze–thaw cycles from multiple perspectives.

Keywords: dispersed soil; freeze–thaw cycle; dispersibility; microstructure; mechanical properties

1. Introduction

Dispersed soil, consisting of fine-grained soil with a low salt content, exhibits rapid dispersion in water [1,2]. Its low erosion resistance poses significant challenges in practical engineering. For instance, the slopes and foundations of excavated channels are susceptible to noticeable collapse under the influence of rainfall. The resulting soil loss threatens nearby engineering facilities and leads to substantial economic losses. In the seasonal freezing zone of western Jilin Province, dispersed soil is distributed widely [3]. Shallow soil or soil exposed to the surrounding environment in this region inevitably undergoes freeze–thaw cycles. Throughout the freeze–thaw cycles, water, ice, and water vapor in dispersed soil undergo transformations and continuous exchanges of matter and energy with the extrinsic environment, leading to changes in the soil structure [4]. Moreover, compared to other soil types, dispersed soil’s soluble salts are more prone to crystallization and dissolution during freeze–thaw cycles, resulting in more complex structural changes and engineering geological properties [5]. Consequently, dispersed soil’s physical and mechanical properties are altered [6–8], significantly reducing its physical properties and strength parameters [9]. These alterations directly influence the stability of foundations and superstructures constructed on scattered soil [10]. A comprehensive investigation of the mechanical properties and dispersion properties of dispersed soil in western Jilin Province is of vital practical significance precisely due to these issues.
Researchers worldwide have extensively studied dispersed soil in seasonally frozen regions, focusing primarily on water–salt migration, freeze–thaw deformation, and physical–mechanical properties. Nagy et al. [11] conducted experiments in Hungary to comprehensively investigate the physical and chemical properties, as well as the composition, of dispersed soil. Zhang et al. examined the engineering geological attributes of dispersed soil in western Jilin Province, China. They explored the relationship between salinity, microstructure, and soil dispersion [2]. Abbaslou et al. [12] discovered the mechanical strength parameters of dispersed soil depend mainly on cementation and inter-particle connections. Hion [13] investigated the impact of salt content, temperature, and unfrozen water content on the mechanical properties of dispersed soil under unconfined conditions, and the results revealed that soil particles, frozen water, and salt solution within the soil determine the strength of dispersed soil. Nixon [14], Joshi [15], and other scholars explored the creep and strength characteristics of dispersed soil under freezing conditions using experimental methods. In the study by Roman [16], experiments were carried out to examine the material composition, deformation behavior, and strength of dispersed soil in seasonally frozen regions and establish the correlation between the strength of dispersed soil and temperature. Sinitsyn [17] demonstrated a direct link between the shear strength of dispersed soil and the rate of applied strain. However, significant variations in soil properties arise from diverse geographical locations. As a result, investigations into the mechanical strength and microstructure of dispersed soil within the seasonal freezing region of western Jilin Province have encountered certain limitations.

Changes in pore content and particle size distribution significantly impact the macroscopic soil characteristics and mechanical behaviors [18]. Utilizing laser particle size analyzers and scanning electron microscopes (SEMs) has become essential for observing particle size distribution and structural changes in soil, offering a valuable understanding of the impact of freeze–thaw cycles on the soil microstructure [19–21]. Hence, utilizing a laser particle size analyzer and an SEM for investigating the evolution of soil pores, particles, and structures can contribute to comprehending alterations in the macroscopic physical and mechanical traits of soil subjected to freeze–thaw conditions. This study primarily investigated the relationship between the mechanical performance and microstructure of dispersed soil under freeze–thaw cycles. The UCS and direct shear strength indicators (c-value and $\varphi$-value) of dispersed soil under varying quantities of freeze–thaw cycles were determined through direct shear tests and UCS tests. Additionally, qualitative and quantitative analyses of the soil samples before and following the freeze–thaw cycles were conducted using particle size analysis tests and SEM tests. This study comprehensively explored the mechanisms underlying the microstructural changes in dispersed soil and their impact on its mechanical strength. The results provide a theoretical guide for improving the soil mechanical properties of channel slopes within the research region and promoting the seamless advancement of engineering projects.

2. Materials and Methods

2.1. Dispersed Soil

The soil specimens examined in this study were sourced from Qian’an, which is located in the western region of Jilin Province, northeast China, as shown in Figure 1. The essential geotechnical characteristics of the soil are condensed in Tables 1 and 2 based on the guidelines outlined in GB/T50123-2019 (Standard for Geotechnical Testing Methods) [22]. The information presented in Tables 1 and 2 demonstrates that the soil sampled from Qian’an predominantly consists of fine-grained particles, including sand, silt, and clay. As per the Unified Soil Classification System [23], this soil falls under the category of lean clay (CL). According to previous research results, it is known that soil in the Qian’an area has a dispersed nature [24–26]. To demonstrate this, SAR can be used to discriminate soil dispersion, according to Rengasamy et al. [27]. The sodium adsorption ratio (SAR) of the soil is 53.68. Therefore, according to Rengasamy et al., soil can disperse spontaneously when SAR >13.
The value obtained in this study is approximately 4 times higher than 13, indicating that soil in the Qian’an area exhibits significant dispersity.

### Table 1. Particle size composition properties of dispersed soil in the study area.

<table>
<thead>
<tr>
<th>Property</th>
<th>Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sand group (2–0.075 mm)</td>
<td>15.90%</td>
</tr>
<tr>
<td>Silt group (0.075–0.005 mm)</td>
<td>63.33%</td>
</tr>
<tr>
<td>Clay group (&lt;0.005 mm)</td>
<td>20.77%</td>
</tr>
<tr>
<td>Soil type information</td>
<td>lean clay (CL)</td>
</tr>
</tbody>
</table>

### Table 2. Physical properties and soluble salt content of dispersed soil in the study area.

<table>
<thead>
<tr>
<th>Property</th>
<th>Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Liquid limit</td>
<td>38.79%</td>
</tr>
<tr>
<td>Plastic limit</td>
<td>18.59%</td>
</tr>
<tr>
<td>Plasticity index</td>
<td>20.20</td>
</tr>
<tr>
<td>Optimum water content</td>
<td>16.76%</td>
</tr>
<tr>
<td>Maximum dry density</td>
<td>1.730 g/cm³</td>
</tr>
<tr>
<td>pH</td>
<td>9.27</td>
</tr>
<tr>
<td>Dispersion (from double hydrometer test)</td>
<td>88.18%</td>
</tr>
<tr>
<td>SAR</td>
<td>53.68</td>
</tr>
<tr>
<td>Total soluble salt content</td>
<td>0.146%</td>
</tr>
<tr>
<td>Na⁺</td>
<td>24.89 mmol/kg</td>
</tr>
<tr>
<td>HCO₃⁻</td>
<td>18.71 mmol/kg</td>
</tr>
<tr>
<td>CO₃²⁻</td>
<td>2.60 mmol/kg</td>
</tr>
<tr>
<td>Ca²⁺</td>
<td>0.28 mmol/kg</td>
</tr>
<tr>
<td>Mg²⁺</td>
<td>0.15 mmol/kg</td>
</tr>
<tr>
<td>K⁺</td>
<td>0.12 mmol/kg</td>
</tr>
</tbody>
</table>

#### 2.2. Specimen Preparation

The methodology employed for sample preparation in this investigation is illustrated in Figure 2a. Dispersed soil samples were collected and subjected to air drying. The dried soil specimens were comminuted and sieved through a 2 mm mesh to eliminate oversized particles. In this study, the specimens were analyzed and determined using a compaction degree of 95%. The soil–water mixture was enclosed within a self-sealing bag and allowed to incubate at a temperature of 20 °C for a duration of 24 h, ensuring uniform water distribution within the soil specimens. Following this, the soil samples were transferred...
into a sample-making mold and compressed under static pressure to obtain remolded soil samples of a desired size. Two sizes of remolded dispersed soil samples were prepared, ring knife specimens (Φ 61.8 mm × 20 mm) and cylindrical specimens (Φ 39.1 mm × 80 mm), as subsequent mechanical tests were required for further study. The remolded soil samples that were prepared underwent sealing with plastic wrap and were subsequently positioned within self-sealing bags to mitigate the potential for moisture evaporation. Changes in specimen volumes were limited using external devices. The remolded soil samples sealed with cling film were frozen in a freeze–thaw cycle chamber for 12 h to ensure complete freezing, and then the samples were taken out and thawed at room temperature for 12 h. This process constituted an entire freeze–thaw cycle. The aforementioned procedure was iterated to procure soil samples that had experienced varying numbers of freeze–thaw cycles for further investigations. This study divided the freeze–thaw cycles into eight stages: an initial state and the 1st, 3rd, 5th, 7th, 9th, 12th, and 15th cycles.

Figure 2. Procedure of sample preparation and corresponding test devices.

2.3. Testing Methods

In this study, we conducted multiple tests, including freeze–thaw, direct shear, and UCS tests. Additionally, we performed the pinhole test, crumb test, double hydrometer test, particle size analysis test, and SEM test. The experimental apparatus, methodologies, and techniques employed in this investigation are elucidated in the subsequent sections.

2.3.1. Freeze–Thaw Cycle Test

The freeze–thaw cycle testing was conducted using an integrated simulation platform designed for subjecting rock and soil to freeze–thaw conditions within an ultracold environment; the test device is illustrated in Figure 2b. According to the temperature data of recent years in the Qian’an area and the experience of previous studies [28], the freezing and thawing temperatures of this test were established to be −20 °C and 20 °C, respectively.
2.3.2. Identification of Dispersibility of the Soil Samples

As a particular type of soil, the dispersibility of dispersed soils cannot be determined by using a single traditional index or test; it is generally necessary to use a variety of identification tests to perform a comprehensive determination. In this research, the soil sample dispersion characteristics were assessed through the pinhole, crumb, and double hydrometer techniques. To ensure the robustness of the obtained results, concurrent assessments were conducted for each of the aforementioned tests. The procedures for each test are elucidated as follows.

1. Pinhole test

The pinhole test is a reliable method for clay dispersion identification. In the pinhole test, the soil specimens were compressed into cylindrical samples measuring $\Phi$ 37 mm $\times$ 38.1 mm. Before the test, a small cone was pushed into the top of the piece, and a 1 mm pinhole was formed by penetrating each sample with a needle (Figure 2g). The pinhole test was then performed under the pressure of a 50 mm water head, and distilled water was used. During the test, the liquid flow through the pinhole was measured for a certain period, and the color and turbidity of the liquid flow were observed. The pinhole diameter of the piece after erosion was measured using a graduated scale to determine the change in the pinhole size of the samples prior to and following the test. Finally, the soil samples were classified into different dispersibility classes according to the pinhole effluent’s final pore diameter, final flow rate, and turbidity [29].

2. Crumb test

The crumb test, which involves observing the degree of crumb formation in soil samples immersed in stagnant water, was conducted to assess the samples’ dispersibility. In the crumb test, a 15 mm sided cube was submerged in a beaker filled with 250 mL of distilled water. A timer was initiated to monitor the alterations in the soil samples when placed in water. Throughout the actual test, the beaker remained undisturbed. The outcomes of the crumb test were utilized to categorize the soil’s dispersibility classes [30].

3. Double hydrometer test

The double hydrometer test involved determining the clay content of the tested soil samples through a non-conventional test and a conventional test. The former involved subjecting the soil samples to vacuum extraction without adding any dispersing agent or boiling. The latter consisted of boiling the soil samples and adding a dispersing agent. The samples for this experiment were extracted from undisturbed unconfined compressive strength specimens after freeze–thaw cycles, and the sampling process strictly adhered to ASTM specifications. Dispersibility is the percentage of the mass of soil passing through a 5-micron sieve after the completion of the test under non-conventional conditions divided by the mass of soil passing through a 5-micron sieve after the completion of the test under conventional conditions. Soil samples exhibiting dispersibility values below 30%, ranging from 30% to 50%, and exceeding 50% were categorized as non-dispersive, transitional, and dispersed soils, correspondingly.

2.3.3. Unconfined Compressive Strength Test

The test procedure and data processing for the UCS test in this study were performed according to the guidelines outlined in GB/T50123-2019 [22]. The experiments were carried out utilizing a YYW-2 strain-controlled pressure instrument (Figure 2c). At least three soil samples with a size of $\Phi$ 39.1 mm $\times$ 80 mm were prepared for each freeze–thaw cycle stage to guarantee the precision of the experiment. The unconfined compressive strength of each soil sample was determined from the shape of the stress–strain curve. To visually assess the impact of freeze–thaw cycles on the strength and stiffness of the soil samples, we used the strength decline percentage ($SDP$) and modulus decline percentage ($MDP$) to evaluate the soil samples [31]. The variations in the strength decline percentage ($SDP$) and modulus
decline percentage (MDP) under different numbers of freeze–thaw cycles were determined according to Equations (1) and (2), respectively:

\[
SDP = \frac{q_{u0} - q_{un}}{q_{u0}} \times 100\%
\]  
\[
MDP = \frac{E_0 - E_n}{E_0} \times 100\%
\]

SDP and MDP represent the percentage reduction in strength and elastic modulus, respectively, after undergoing \( n \) freeze–thaw cycles. The parameters \( q_{un} \) and \( q_{u0} \) represent the unconfined compressive strength values of the soil after undergoing \( n \) freeze–thaw cycles and before any exposure to freeze–thaw cycles, respectively. Similarly, \( E_n \) and \( E_0 \) indicate the modulus values after \( n \) freeze–thaw cycles and prior to any freeze–thaw cycle exposure.

2.3.4. Direct Shear Test

Due to the poor permeability of dispersed soils in the Qian’an area, they are susceptible to damage before sufficient consolidation occurs during loading. Therefore, the direct shear test was opted for to assess the shear strength of individual soil samples. The direct shear test was conducted using an electric shear apparatus, depicted in Figure 2d. (All specimens used in the experiments were at their optimum moisture content.) The soil samples were of dimensions \( \Phi 61.8 \text{ mm} \times 20 \text{ mm} \). The test procedure and data processing followed the guidelines outlined in the “Standard for Geotechnical Testing Methods (GB/T50123-2019)” [22]. The vertical stress levels were configured at 100, 200, 300, and 400 KPa, with a constant shear rate of 0.8 mm/min. The respective shear stress and displacement values were meticulously documented. These test outcomes were subsequently employed to construct shear stress–shear displacement curves for every soil sample across varying vertical stress conditions. The determination of shear strength parameters for each sample was predicated on the Mohr–Coulomb criterion.

2.3.5. Particle Size Analysis Test

A BT-9300LD laser particle size analyzer (Figure 2e) was employed to analyze the particle size distribution of the dispersed soil samples following varying quantities of freeze–thaw cycles. Since dry testing involves air-drying and grinding of soil samples, it is inevitable that the particle size distribution of the soil will be greatly disrupted. Therefore, in this experimental study, a wet dispersion system was employed. During the particle size testing process, the freeze-thawed ring knife samples were first gently broken apart. A suitable amount of soil from the interior was then collected in a small beaker and thoroughly soaked in distilled water. The resulting soil suspension was completely poured into the recirculation tank, and the inner walls of the beaker were rinsed with distilled water, with the rinse liquid also poured into the recirculation tank to ensure minimal loss of soil particle composition. To reduce experimental errors, two positions were sampled for each ring knife sample, and the average of three consecutive measurements at each position was taken. The results were recorded only after ensuring that the particle size measurement outcomes exhibited good single-run repeatability and multi-run reproducibility. In cases where stability was not achieved, retesting was performed to attain consistent results. It should be noted that the laser particle size analyzer used in this experiment has an upper limit of 1 mm for measuring particle sizes. However, according to Liu et al. [32], no particles larger than 1 mm are present in a suspension formed by soaking soil in distilled water. Therefore, the utilization of solely the laser particle size analyzer sufficed to achieve a precise particle size distribution of the soil samples. In this particle size analysis test, no dispersant was introduced to maintain the agglomeration effect induced by the freeze–thaw cycles and to prevent interference with the particle structure.
2.3.6. Scanning Electron Microscopic Test

The scanning electron microscope (SEM) utilized in this study was the Phenom ProX Desktop model. It allows detailed imaging and analysis of samples, contributing to a comprehensive understanding of the research subject from a microscopic perspective (Figure 2f). Before capturing the SEM pictures, the samples were freeze-dried using liquid nitrogen and subjected to vacuum treatment to prevent structural changes. Following freeze-drying, the samples underwent gold sputtering utilizing a high-vacuum sputter coater. The analysis of SEM images and the differentiation of structural units within pores based on their gray levels were facilitated through the utilization of IPP 6.0 software in this study. The objective of this approach was to unveil the microscopic mechanisms accountable for the modifications in the mechanical characteristics of the soil samples. Comprehensive assessments, encompassing both qualitative and quantitative analyses, were conducted using the SEM micrographs. The qualitative examination directly juxtaposed the distinctions in soil structure and inter-particle spacing depicted in the SEM micrographs of the soil samples across various freeze–thaw stages. The quantitative analysis utilized one indicator, surface porosity (N). This indicator is the percentage of S0 (pore space) over S (total area of the SEM image). N represents the surface porosity of a typical SEM image. Although surface porosity may not precisely mirror the actual condition of a soil sample as volumetric porosity does, it can still function as a comparative indicator for assessing alterations in pore structure.

3. Result and Discussion

3.1. Soil Sample Dispersion Identification Test

3.1.1. Pinhole Test

The pinhole test involved subjecting the soil samples to a water pressure of 50 mm head to observe the erosion of small holes and the flow and turbidity of water. The degree of soil dispersion was determined according to the specifications [29]. The pore size obtained from the pinhole test, as shown in Figure 3, is considered a reliable indicator for determining the dispersibility of soil samples [33]. It provides valuable information about the soil’s ability to resist erosion under specific hydraulic conditions. The dispersion of each soil sample was evaluated based on the identification criteria, and the outcomes of the pinhole test are displayed in Table 3, indicating the level of dispersibility of each sample.

![Figure 3. Final pore diameter of dispersed soil after different numbers of freeze–thaw cycles (where “F–T n” indicates the quantity of freeze–thaw cycles).](image)

For a more comprehensive comprehension of the alterations in dispersibility, the final flow rate and pore diameter curves were constructed for all highly dispersible soil samples, depicting their evolution relative to the number of freeze–thaw cycles. As depicted in Figure 4a,b, both the final flow rate and pore diameter exhibit an ascending trajectory with an augmentation in the number of freeze–thaw cycles. This trend signifies a gradual amplification in dispersibility. The outcomes of the pinhole test unveil that with the
escalation of freeze–thaw cycles, the soil’s structure experiences progressive deterioration, leading to a weakening of inter-particle cohesive forces and subsequent reduction in the soil’s resistance against water erosion. Furthermore, under identical hydraulic conditions, the soil sample’s final pore size and effluent turbidity are gradually augmented.

### Table 3. Identification results of the pinhole test.

<table>
<thead>
<tr>
<th>Number of F–T Cycles</th>
<th>Water Head (mm)</th>
<th>Duration at a Given Water Head (min)</th>
<th>Final Flow Rate (mL/s)</th>
<th>Turbidity of Water</th>
<th>Final Pore Diameter (mm)</th>
<th>Results</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>50</td>
<td>5</td>
<td>2.00</td>
<td>Turbid</td>
<td>2.0</td>
<td>D1</td>
</tr>
<tr>
<td>1</td>
<td>50</td>
<td>5</td>
<td>1.83</td>
<td>Turbid</td>
<td>2.4</td>
<td>D1</td>
</tr>
<tr>
<td>3</td>
<td>50</td>
<td>5</td>
<td>1.67</td>
<td>Turbid</td>
<td>2.3</td>
<td>D1</td>
</tr>
<tr>
<td>5</td>
<td>50</td>
<td>5</td>
<td>2.00</td>
<td>Turbid</td>
<td>3.5</td>
<td>D1</td>
</tr>
<tr>
<td>7</td>
<td>50</td>
<td>5</td>
<td>2.03</td>
<td>Turbid</td>
<td>3.6</td>
<td>D1</td>
</tr>
<tr>
<td>9</td>
<td>50</td>
<td>5</td>
<td>2.33</td>
<td>Very turbid</td>
<td>4</td>
<td>D1</td>
</tr>
<tr>
<td>12</td>
<td>50</td>
<td>5</td>
<td>2.5</td>
<td>Very turbid</td>
<td>4.5</td>
<td>D1</td>
</tr>
<tr>
<td>15</td>
<td>50</td>
<td>5</td>
<td>3.17</td>
<td>Very turbid</td>
<td>4.8</td>
<td>D1</td>
</tr>
</tbody>
</table>

**Figure 4.** Effect of freeze–thaw cycles on the pinhole test: (a) variation curve of final flow rate and (b) variation curve of final pore diameter.

### 3.1.2. Crumb Test

Figure 5 displays crumb test images of each soil sample following freeze–thaw cycles. The disintegration condition of the crumb test showed that each soil sample started to react severely when it was put into distilled water, and the disintegration condition of the soil sample hardly changed after one hour. A large amount of cloudy, thick, viscous particle suspension could be observed around the soil block, and the mist spread more than 10 mm at the bottom of the cup. The soil sample was judged as strongly dispersed soil of grade 4 according to the criteria of the crumb test specification. And as the number of freeze–thaw cycles increases, the cloudy suspension spreads more and more (the most apparent observation is at 2 min), and the solution becomes increasingly turbid.

As the number of freeze–thaw cycles increases, the interaction forces between colloidal particles gradually weaken, leading to more severe damage to the soil structure. Therefore, colloidal particles that act as connectors between soil particles interact with water and dissolve, leading to an increased degree of fine particle precipitation in the soil sample. Simultaneously, the turbidity of the surrounding water gradually increases. As colloids continue to dissolve, the connections between particles gradually weaken, ultimately enhancing soil dispersibility.
3.1.3. Double Hydrometer Test

The double hydrometer test is an additional method employed to assess soil dispersibility. The double hydrometer test provides insights into the particle composition of soil by dispersing a soil sample into primary particles in water. The grading of soil particles has a significant influence on soil dispersibility. The experimental data are presented in Table 4. This dispersion exceeds 85% in this experiment (Figure 6), indicating strongly dispersed soil. The double hydrometer test results demonstrate that a significant portion of clay particles can be separated under vacuum conditions, indicating that the soil samples exhibit a relatively loose structure and poor particle aggregation after undergoing freeze-thaw cycles. As a result, the soil samples exhibit a state of high dispersion.
The outcomes of the pinhole, crumb, and double hydrometer tests consistently indicate a progressive increase in the dispersibility of the examined soil samples as the number of freeze-thaw cycles rises. During the freezing process, water-facilitated movement of sodium ions results in the augmentation of exchangeable sodium ions enveloping soil particles and an expansion of the diffusion layer. This phenomenon effectively elucidates the elevated dispersibility of the dispersed soil in the investigated region. Throughout the winter season, certain sodium salts crystallize within the frozen soil layer, establishing a concentration gradient between the frozen and unfrozen soil layers. Consequently, sodium salts migrate to the frozen layer alongside a surge in thin-film water. The accumulation of substantial salt content on the soil surface drives notable dispersion. Moreover, frost heave during freezing augments the inter-particle spacing, leading to a reduction in cohesive forces between soil particles and encouraging soil dispersion [2]. Additionally, freeze–thaw cycles alter the interconnection among soil particles, disrupting the existing colloidal connections and causing aggregates to disintegrate, further enhancing soil dispersibility.

### 3.2. UCS Test

The freeze–thaw cycles have a substantial impact on the mechanical behavior of soils, mainly attributed to the interconnected relationship between water, ice, and soil [34]. Figure 7 depicts the stress–strain behavior of the soil samples following freeze–thaw cycle testing. The stress–strain curves reveal notable differences when comparing the unfrozen soil sample, which exhibits significant peaks indicating a strain-softening behavior and brittle damage, with the curve obtained after a freeze–thaw cycle. In the latter case, the stress–strain plot exhibits a noticeable decline, characterized by a gradual reduction in the peak magnitude. Subsequent to attaining the peak, the axial strain further increases, accompanied by a gradual attenuation in the rate of axial stress reduction. This transition from strain softening, observed in the 0 freeze–thaw cycle, to strain hardening indicates plastic failure of the soil samples.

![Figure 6. Variations in soil dispersion with different numbers of freeze–thaw cycles.](image-url)
3.2. UCS Test

The freeze–thaw cycles have a substantial impact on the mechanical behavior of soils, mainly attributed to the interconnected relationship between water, ice, and soil structure. The structural damage on qu in the soil samples diminishes a higher number of freeze–thaw cycles, followed by a significant increase after 7–9 freeze–thaw cycles, and finally stabilizes after 12 freeze–thaw cycles. Therefore, it is crucial to study the irreversible damage occurring in soils within the first ten freeze–thaw cycles.

As depicted in Figure 8a, the unconfined compressive strength (qu) significantly decreases after the first freeze–thaw cycle, and following the initial freeze–thaw cycle, it can be divided into three distinct stages based on varying reduction percentages. The first stage encompasses one to five freeze–thaw processes, during which the reduction in qu remains relatively consistent, with an average decrease of approximately 40%. The second stage occurs after seven to nine freeze–thaw cycles, where the qu value after the seventh cycle decreases compared to that after the fifth cycle, indicating the initiation of the second stage. At this stage, the reduction in qu remains relatively constant, with an average decrease of around 50%. The second stage occurs after the seventh cycle decreases compared to that after the fifth cycle, indicating the initiation of the second stage. At this stage, the reduction in qu remains relatively constant, with an average decrease of around 50%. By the 12th cycle, the decreasing trend starts to slow down, although the reduction continues. It can be assumed that the impact of structural damage on qu in the soil samples diminishes a higher number of freeze–thaw cycles during the third stage. The relationship between SDP and the number of freeze–thaw cycles is illustrated in Figure 8b. The change in SDP can also be roughly divided into three stages. After the first freeze–thaw cycle, SDP remains stable after 1–5 freeze–thaw cycles, followed by a significant increase after 7–9 freeze–thaw cycles, and finally stabilizes after 12 freeze–thaw cycles. The variations in SDP indicate that the strength loss caused by the freeze–thaw action is more pronounced in the first two stages. In contrast, the degree of strength deterioration gradually diminishes with an increase in freeze–thaw cycles. SDP shows slow growth with an increasing number of freeze–thaw cycles and eventually stabilizes before reaching 15 freeze–thaw cycles. Therefore, it is crucial to study the irreversible damage occurring in soils within the first ten freeze–thaw cycles.

Figure 7. Stress–strain curves of dispersed soil subjected to varying freeze–thaw cycles.

Figure 8. Effect of freeze–thaw cycles on the UCS test: (a) the qu of soil and (b) the SDP of soil.
Based on the results obtained from the UCS test, the modulus of deformation (E50) is defined as the stress-to-strain ratio corresponding to 50% of the unconfined tensile strength. This parameter is commonly used in geotechnical studies to evaluate soil deformation behavior. A higher modulus of deformation indicates greater stiffness and improved resistance to deformation. Figure 9 illustrates the relationship between the modulus of deformation percentage (SDP) and the number of freeze–thaw cycles. The SDP demonstrates an increasing trend as the number of freeze–thaw cycles increases. After ten cycles, the SDP of the soil samples exhibits a relatively stable range between 52% and 55%, with minimal variation.

![Figure 9. Variations in SDP of dispersed soil after different numbers of freeze–thaw cycles.](image)

### 3.3. **Direct Shear Test**

The obtained results reveal that the configurations of the shear stress–shear displacement curves and the patterns of alterations in each soil sample under varying pressures exhibit similarities. This finding is depicted in Figure 10. (Furthermore, the relationship curves between vertical deformation and shear displacement can be found in Figure S1 of the Supplementary Material.) It is evident from the figure that, in all cases, an increase in the number of freeze–thaw cycles leads to a decrease in the shear stress of the soil samples. As compressive stress applied to soil samples decreases, shear strength reduction becomes more evident. Additionally, the shear stress–shear displacement curves of soil samples after freeze–thaw cycles show strain-hardening behavior without notable peaks.

Cohesion (c) and angle of internal friction (ϕ) are two critical parameters that determine the shear strength of soils and can be determined using the Mohr–Coulomb criterion. Figure 11 presents the changes in shear strength parameters for each soil sample under varying quantities of freeze–thaw cycles. The findings suggest that the c-value is more profoundly impacted by the number of freeze–thaw cycles compared to the ϕ-value. The c-value decreases by 65% throughout the process, while the ϕ-value only exhibits a change of approximately 30%. In general, the ϕ-value of the soil samples does not undergo significant changes with an increasing quantity of freeze–thaw cycles. However, there is a trend of an initial increase, followed by a decrease, and the ϕ-value peaks at seven freeze–thaw cycles.

The decrease in the c-value can be attributed to two factors. Firstly, the soil used in this experiment has a high concentration of Na⁺ ions, which can enhance the thickness of the hydration film surrounding soil particles. This increase in hydration film thickness leads to a more significant separation distance between soil particles, consequently leading to reduced soil cohesion [35]. Secondly, the escalation in the frequency of freeze–thaw cycles can disrupt the original structural properties of the soil. The soil samples, before undergoing the freeze–thaw cycles, exhibit a relatively intact structure. In the freeze–thaw process, the existence of expanded pores and fissures in the soil intensifies, resulting in a weakened interconnection among soil particles. Consequently, this leads to a notable decline in the c-value. The behavior of the ϕ-value follows a pattern of initial increase.
and subsequent decrease with an escalation in the frequency of freeze–thaw cycles. This phenomenon can be ascribed to the crystallization of substances within the pore solution. With a higher number of freeze–thaw cycles, soluble salt crystals gradually form and serve as a bridging agent among soil particles [36]. This linking effect partially mitigates structural damage and leads to an increase in the $\varphi$-value. However, the highest degree of crystalline substance precipitation is reached after seven freeze–thaw cycles. The widening of cracks beyond this point is no longer conducive to crystallizing soluble salts in the pore solution, resulting in a gradual decrease in the $\varphi$-value. The changes in the shear strength parameters, including the decline in the $c$-value and the initial increase followed by a reduction in the angle of internal friction $\varphi$-value, significantly affect the macroscopic mechanical strength of the soil samples, according to the Cullen strength criterion. Despite the initial increase and subsequent decrease in the $\varphi$-value, the magnitude of this change is much smaller compared to the reduction in the cohesion $c$-value. Therefore, the overall shift in strength primarily relies on the variation in the $c$-value, as a significant reduction in cohesion results in a consistent decline in the overall macroscopic mechanical strength of the soil specimens.

![Figure 10](image10.png)

**Figure 10.** Variations in shear stress–shear displacement curves of the soil samples after different numbers of freeze–thaw cycles.

![Figure 11](image11.png)

**Figure 11.** Effect of freeze–thaw cycles on shear strength indicators: (a) $c$-values and (b) $\varphi$-values.
3.4. Particle Size Analysis Test

The soil particle size distribution curves under varying freeze–thaw cycles were obtained by analyzing the test results and plotting the data, as illustrated in Figure 12. The arrows indicate the curves’ trend with increasing freeze–thaw cycles. The alteration in the particle size distribution of the soil samples encompasses a reduction in the proportion of large particles and an augmentation in the proportion of small particles.

![Particle Size Distribution Curves](image1)

Figure 12. The particle size distribution curves of the soil samples’ particles after different numbers of freeze–thaw cycles. (The arrows indicate the curves’ trend with increasing freeze–thaw cycles).

To better analyze the changes in the particle size composition of the soil samples, the particles in each sample were categorized into three groups: clay particles (<0.005 mm), silt particles (0.005–0.075 mm), and sand particles (0.075–2 mm). The change curves depicting the proportion of each particle group with the progression of freeze–thaw cycles were graphed, as depicted in Figure 13. The findings illustrate that the granular composition of the soil samples undergoes alterations as the number of freeze–thaw cycles increases. Specifically, the content of sand particles shows a significant decrease as the number of freeze–thaw cycles increases. Conversely, there is a gradual overall increase in the contents of clay and silt particles. Thus, the freeze–thaw process exerts a dual effect on soil particles.

![Particle Composition Changes](image2)

Figure 13. Variations in particle size composition of the soil samples after different numbers of freeze–thaw cycles.

On the one hand, the freeze–thaw process causes larger particles in the soil to fragment into smaller particles. On the other hand, it leads to the aggregation of smaller particles into larger ones. Consequently, an increase in the number of freeze–thaw cycles results in the breakdown of sand particles into finer clay and silt particles. Additionally, some
clay particles may transform into silt particles due to aggregation. During the test, the fragmentation of sand grains was more pronounced, resulting in a decline in sand content and a rise in the proportions of silt and clay particles. Notably, the increment in clay content was relatively higher than that of silt particles. Overall, the content of finer particles in the soil samples increased after undergoing freeze–thaw cycles compared to samples that did not experience such processes.

3.5. Influence of Freeze–Thaw Cycles on the Microstructure of the Soil Samples

Figure 14 displays the SEM images of the soil samples after undergoing various numbers of freeze–thaw cycles. The unfrozen–thawed soil particles (Figure 14a) have well-defined bound pores, primarily linked through point-to-point or point-to-face interactions. However, significant changes in particle morphology and pore characteristics are observed after the freeze–thaw cycles, as depicted in Figure 14b–h. After experiencing freeze–thaw cycles, newly developed fissures (highlighted by dashed green lines) emerge within the soil matrix, resulting in a more porous arrangement. Throughout the freeze–thaw phenomenon, microcracks initially form on the exterior of larger particles and progressively amalgamate to create more extensive fissures. These cracks continue to propagate with subsequent freeze–thaw cycles, ultimately resulting in the fragmentation of soil particles (highlighted in the red box). With an escalation in the frequency of freeze–thaw cycles, the gaps within the soil extend over greater distances, leading to a more porous and loose soil structure.

![Figure 14. Scanning electron microscopic photographs of the tested soil samples after different numbers of freeze–thaw cycles (F–T n represents the number of freeze–thaw cycles and (a–h) represent microstructural images after 0 to 15 cycles of freeze-thaw).](image-url)
Additionally, the fracture width tends to increase, which is accompanied by a higher incidence of particle breakage, particularly for larger particles. Consequently, the proportion of smaller particles in the soil rises over subsequent freeze–thaw cycles. Furthermore, salts crystallize from the pore solution, as illustrated by the yellow circle in Figure 14b,f.

The average diameter of soil particles can reflect their size. The fluctuation in the average particle size of the examined soil samples, with an escalation in the frequency of freeze–thaw cycles, was determined by analyzing the magnified SEM images (800 times magnification) using the IPP 6.0 software. As shown in Figure 15a, it is evident that the average particle size of the soil samples tends to decrease as the number of freeze–thaw cycles increases. The most significant changes occur within the range of 0–9 freeze–thaw cycles, decreasing from 3.9 µm to 3.21 µm and stabilizing after that. This trend aligns with the results obtained from particle size analysis tests. This phenomenon can be attributed to the rapid destruction of larger soil particles into smaller ones during the initial nine freeze–thaw cycles, resulting in a weaker inter-particle association and increased distance between soil particles. Surface porosity is an indicator that reflects the pore content of soil to some extent. A higher value indicates a more significant proportion of pores in the soil and a looser soil structure. The variation in surface porosity (N) of the tested soil samples with an increase in the number of freeze–thaw cycles was calculated using the IPP 6.0 software, and the results are presented in Figure 15b.

![Figure 15. Effect of freeze–thaw cycles on microstructure: (a) the mean diameter of the soil samples and (b) the surface porosity of the soil samples.](image)

It can be observed that the surface porosity (N) of the soil samples exhibits an increasing trend with an escalation in the frequency of freeze–thaw cycles. During the 0–9 freeze–thaw cycles, the value increases from 14.47% to 30.5% and gradually stabilizes. While the surface porosity of the soil samples, determined through the SEM analysis, does not encompass internal pores within soil particles, it does offer valuable insights into the changes in pore content of the soil samples as the number of freeze–thaw cycles rises.

3.6. Mechanisms of Changes in the Microstructure of the Soil Samples

Based on the particle size analysis test and the SEM experiment (the data summary table is available in Supplementary Materials as Table S1), it is evident that the content of sand particles in the soil samples gradually decreases, while the contents of clay particles and silt increase with an increasing number of freeze–thaw cycles. Throughout a freeze–thaw cycle, soil particles undergo simultaneous fragmentation of coarse particles and aggregation of fine particles. The complete process of soil particle transformation is illustrated in Figure 16. As the number of freeze–thaw cycles increases, internal stress is generated within soil particles, resulting in the fragmentation of coarse particles. During freezing, the liquid-free water in the pores transforms into solid ice, forming cracks on the surface of soil particles. The expansion of ice crystals and their wedging into the cracks...
cause a volume expansion of approximately 9% [37], thus exerting pressure on soil particles and leading to the development and extension of microcracks, ultimately forming larger cracks. Consequently, the water film thickness within the gaps between soil particles increases, leading to further fragmentation and disintegration of coarse particles into smaller ones. There is consensus among researchers regarding these findings [20,38].

![Figure 16. Illustrative diagram illustrating the transformation of soil particles under freeze–thaw conditions.](image)

The aggregation of fine particles occurs due to a bound water film adsorbed around clay particles and the action of the double electron layer, which promotes the aggregation of fine particles. Additionally, the negatively charged surface of clay particles attracts polar water molecules, resulting in the formation of a bound water film and the development of attractive forces between soil particles. Freeze–thaw cycles disrupt the lattice of minor minerals, alter the charge characteristics of soil particles, and enhance molecular forces between fine soil particles, leading to their aggregation. Moreover, during the freezing process, dissolved substances migrate along unfrozen water, resulting in their absorption by the surface of clay mineral particles. This contributes to the binding of clay particles into powder particles and further promotes particle aggregation [38]. Furthermore, the dissociation and crystallization of polymers during ice’s volume expansion generate new particles [39]. With the aggregation of fine particles, the porosity within the soil increases gradually, as evidenced mainly by the increase in surface porosity observed in this study.

When subjected to shear deformation, the unfrozen soil samples need to break up intact soil particles, resulting in a higher shear strength. In contrast, when subjected to shear stress, the frozen–thawed soil samples primarily overcome the bonding forces between fine particle aggregates. However, due to the disruption of colloidal connections between fine particle aggregates caused by the freeze–thaw cycles, the inter-aggregate connections weaken, thus significantly reducing strength. Simultaneously, with the gradual escalation in the number of freeze–thaw cycles, the soil structure forms irreversible through cracks that progressively widen, posing a severe threat to the stability of the soil structure and further reducing its strength. Consequently, the mechanical and physical properties of the soil are affected, potentially causing instability and damage to engineering projects within the study area.

4. Conclusions

This study explored the correlation between the mechanical strength of soil and freeze–thaw cycles in a seasonal permafrost region. The geotechnical properties of the tested soil samples were comprehensively investigated, considering factors such as particle size composition, microscopic characteristics, and dispersion. The mechanism of soil deterioration under freeze–thaw conditions was determined by analyzing the experimental results. Drawing upon the acquired findings and subsequent discussions, the following conclusions can be inferred:

1. The dispersibility of the soil samples progressively increases with an increasing number of freeze–thaw cycles. Simultaneously, the corresponding increase in dispersion negatively affects the soil’s mechanical strength. Overall, these two variables exhibit a negative correlation.
With an increasing number of freeze–thaw cycles, the particle size distribution of the soil samples exhibits a general trend of fragmentation of large particles and gradual accumulation of smaller particles. The sand content decreases from 38.76% to 16.59%, and the clay content increases from 7.48% to 24.20%.

During the first nine freeze–thaw cycles, the reduction in strength is significant, with the unconfined compressive strength decreasing from 156.843 kPa to 76.961 kPa. After nine freeze–thaw cycles, the rate of decrease in strength slows down, and the unconfined compressive strength stabilizes at around 70 kPa. Furthermore, the $c$-value gradually decreases from 22.196 kPa in the unfrozen state to 7.997 kPa and then stabilizes. The $\varphi$-value starts at 7.514° in the unfrozen state, peaks at 9.514°, and gradually declines.

With increasing freeze–thaw cycles, new cracks continuously form, existing cracks expand, and the surface porosity increases from 14.47% in the unfrozen state to 32.10% after the 15th freeze–thaw cycle.

Supplementary Materials: The following supporting information can be downloaded at https://www.mdpi.com/article/10.3390/app13179849/s1. Figure S1: Relationship curves between vertical deformation and shear displacement in direct shear tests of tested soil samples after different numbers of freeze-thaw cycles (where F-T n represents the number of freeze-thaw cycles); Table S1: Summary of Final Results.

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