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Experimental Study on Temperature-time Characteristics of Loess under the Freeze-Thaw Cycles

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Abstract **– The loess in northern Shaanxi is situated in the seasonal frozen region, where the soil water phase transition is predominantly caused by freeze-thaw cycles. Engineering practices have evidenced that the transformation of water from soil causes considerable damage to structures, roadways, slopes, canals, and other infrastructure. The temperature-time characteristics of loess, particularly freezing point, serve as a crucial indicator for assessing the freeze-thaw state of soil. To investigate the influence of water content w and the number of freeze-thaw cycles N on temperature-time behaviours, the temperature-time curves of loess with diverse water content were depicted through freeze-thaw cycle tests. The alterations in freeze-thaw characteristics such as freezing point, freezing time, supercooling phenomenon, thawing time, and thawing point were analysed. The findings indicated that: 1) The phenomenon of supercooling was significantly affected by w and N. When $w = 9$ % and 13 %, the supercooling phenomenon gradually became significant as N increment. Conversely, when $w = 17$ %, the supercooling phenomenon became less significant as N increment. The decline curves of various w were essentially identical in the supercooling stage, and the cooling rate decreased as w increased during continual freezing stage. 2) The freezing point of loess gradually decreased as N increment. Freezing time did not exhibit significant variations in relation to N, however, a higher water content led to a longer freezing duration. All soil samples attained a stable freezing temperature within 12 hours. 3) The thawing point of soil samples remained constant at 0 °C under varying N, however, the stability levels of all curves at zero degrees varied. Except for $N = 3$, all other cases exhibited a gradual increment after 12 hours, which might be attributed to the instability of the thawing temperature during the experiment. 4) During the rapid ascent stage of the thawing curve, the ascending rate slightly increased with the increase of N. In the slow rise stage, the rising rate was relatively rapid during $N \leq 3$, the rate of increase experienced a sudden drop at $N = 10$, and then proceeded at a relatively slower. The change pattern of the thawing curve remained consistent across various w, with only a certain extent of influence on the rate of change. The results might provide theoretical support for the engineering design, construction, and maintenance of the seasonal frozen soil area in northern Shaanxi Province and other regions with comparable weather conditions.**

Keywords **– Freezing point; freeze-thaw cycles; loess; temperature-time curve; water content.**

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1. INTRODUCTION

Freeze-thaw erosion constitutes the third major type of soil erosion after water erosion and wind erosion [1], which seriously endangers agricultural production land, various buildings and water conservancy facilities [2]–[4]. Freeze-thaw erosion occurs in 58.2 % of China's permafrost area. Loess is distributed in the northwest region of China, particularly on the Loess Plateau in northern Shaanxi, and majority of it undergoes a seasonal frozen environment. The surface soil in this area undergoes freeze-thaw cycles for an extended period, experiencing freezing during winter and thawing during spring and summer. These repeated freeze-thaw cycles have a significant impact on the original structure of the soil, leading to degradation of its physical and mechanical characteristics [5]–[8], and eventually resulting in the occurrence of engineering hazards such as building cracking, slope instability, road denudation cracking, and others [9], [10]. This poses challenges to construction projects and infrastructure development in this region. It is highly significant for engineers and developers to consider these unique geological conditions when planning and implementing projects in areas with loess deposit.

The physical and mechanical properties of materials assume a crucial role in materials science, providing essential guidance for the selection, design, and application of materials. Currently, a considerable number of scholars have carried out extensive research on the static and dynamic mechanical properties of various materials, encompassing extensive range of materials and multiple research methods [11]–[13]. The physical and mechanical properties of loess are impacted by factors such as water content, temperature, freeze-thaw cycle, etc. [14], [15]. It is a crucial step to evaluate the condition of loess in the seasonal frozen soil area and determine its physical and mechanical properties to guarantee the stability and durability of infrastructure in the frozen soil area. The temperature characteristics of loess, particularly freezing point, constitutes a prerequisite for any laboratory or field investigation of the freeze-thaw characteristics of soil. It facilitates the identification of the freeze-thaw state of soil samples in laboratory freeze-thaw equipment. Simultaneously, it is also a significant design parameter for buildings related to freeze-thaw, such as frozen soil walls and the freezing depth of soil [16], [17]. The frozen depth of the soil under natural conditions constrains the depth of the foundation of industrial and civil buildings and also determines the thickness of the active layer in non-permafrost areas. A series of physical and mechanical processes occur in the active layer, which exert a highly significant influence on the strength and stability of the building foundation.

Many scholars have extensively studied the freezing point of soil. Liu and Chuvilin *et al.* [18], [19] evaluated the freezing point of soil by means of the potential jump method, also examined the changes in freeze-thaw temperature, water content, and pressure, it was determined that the thawing curve of soil presented a continuous increase without reaching a stationary phase. Xing [20] determined the freezing point by analysing the jump characteristics of soil temperature during freezing, the results indicated a positive correlation between higher water content and an increasing trend in the freezing point, which in accordance with a cubic polynomial relationship. Ren, Wu, Bing and Ming *et al.* [10], [21]–[23] investigated the effects of water content, salt content, salts and cooling rate on freezing point. Zhou *et al.* [24] determined that the phenomenon of supercooling is non-existent in any freezing method by measuring the temperature-time curves of soil samples under various freezing conditions and analysing the conditions for the occurrence of supercooling. Wang *et al.* [25] found that as the freeze-thaw cycles increased, the freezing point of salt loess decreased and then gradually stabilized. Wang *et al.* [26] observed a non-linear correlation between the freezing point of loess and water content, with negligible association with dry density. They also found significant variations within the first five freeze-thaw cycles, but minimal effects beyond this threshold. Majority of research on soil thawing processes mainly concentrates on thaw collapse and hydrothermal coupling migration [27]–[29], while the temperature characteristics associated with thawing have received limited attention. Although the phenomenon of supercooling was mentioned in the above studies, the influencing factors were not analysed. In the experiment regarding the thawing characteristics of permafrost, Fu [30] categorized the temporal variations in soil temperature into three stages: phase transition stage, non-phase transition unsteady stage, and stable stage, and analysed the duration of phase transition. There is limited research on the effects of water content and freeze-thaw cycles on soil thawing.

Therefore, based on the indoor freeze-thaw cycle experiments conducted on loess in northern Shaanxi Province, temperature-time curves of loess were obtained under varying water content w and freeze-thaw cycles N. The influence of w and N on the freeze-thaw curve was investigated, and the variations in freezing characteristics such as the freezing point, freezing time, and supercooling phenomenon were analysed, as well as thawing characteristics including the thawing point and thawing time. The results might provide theoretical support for the engineering design, construction, and maintenance of the seasonal frozen soil area in northern Shaanxi Province and other regions with comparable weather conditions.

2. MATERIALS AND METHODS

2.1. Materials

The loess extracted from a slope of Yan'an New District was used as the experimental material. After removing the topsoil due to the presence of vegetation cover, systematic sampling at regular intervals of 3–4 meters with a depth ranging from approximately 40 to 45 centimetres. The basic physical parameters of loess samples are given in Table 1.

The city of Yan'an, situated on the typical Loess Plateau, exhibits a warm temperate semi-humid climate with a susceptibility to drought. Summers are characterized by hot and rainy weather, while winters are dry and cold. The topsoil undergoes freeze-thaw cycles due to climatic influences, with a depth of approximately 0.80 m. According to climate statistics from 2011 to 2023, the maximum temperature in June, July and August with an average temperature of about +20 °C, while the minimum temperature in November, December, January and February with an average temperature of about −10 °C. Therefore, the recorded freezing temperature in this experiment is −10 °C, while the observed thawing temperature is +20 °C.

TABLE 1. PHYSICAL INDEX OF LOESS SPECIMEN

Physical index		**Value**
Water content	w (%)	13.6
Dry density	ρ_d (g/cm^3)	1.45
Atterberg limits		
Liquid limit	w_L (%)	26.5
Plastic limit	w_P (%)	14.3
Plasticity index	I_P	12.2

2.2. Sample Preparation

The soil was initially pulverized and air-dried, then screened with a standard diameter of 2 mm. The quantities of water were determined in accordance with the experimental protocol. The sifted loess was subsequently moistened with water until the desired level of water content was achieved. Then, the resulting slurry was sealed in plastic bags and incubated at room temperature for a minimum of 24 hours, ensuring uniform moisture dispersion throughout the soil. Subsequently, the prepared slurry was static compressed to make a cylindrical specimen with a height of 20.0 mm and a diameter of 61.8 mm. Finally, the prepared specimens were wrapped in plastic wrap to minimize potential water loss during preparation, and then underwent freeze-thaw cycles.

This study investigated loess specimens with three levels of water content, specifically at $w = 9.0$ %, 13.0 %, 17.0 %, seven levels of freeze-thaw cycle, such as $N = 0$, 1, 3, 10, 20, 30, 40. Based on the meteorological records, the ambient temperature decreases to −10 °C during freezing and increases to +20 °C during thawing. A single freeze-thaw cycle is defined as consecutive period of freezing and thawing lasting for 12 hours each.

2.3. Experimental Scheme

The freeze-thaw experiments were performed within a regulated low-temperature chamber with an effective volume of 160 L. The chamber utilized the compressor cooling technique to regulate temperatures, allowing for a wide range from –60 to +100 °C. During the testing process, the prepared specimen is meticulously wrapped in plastic wrap and hermetically sealed within a bag to effectively impede water evaporation. Subsequently, they were grouped and positioned within a temperature-controlled chamber to ensure accurate environmental conditions.

The temperatures of soil specimens were determined by embedded temperature sensors and a data acquisition system. The temperature sensor was strategically placed within the central region of the soil sample to undergo predetermined freeze-thaw cycles, while the data acquisition system continuously monitored and recorded the temperatures of the soil specimens. The cooling phase at −10 °C for 12 hours to ensure complete freezing of the specimen, Subsequently, raising the temperature to +20 °C initiates for another 12 hours in the thawing phase, thus completing one freeze-thaw cycle, continue the procedure until the specified number of cycles has been achieved.

3. RESULTS AND DISCUSSION

3.1. Analysis of a Freeze-Thaw Cycle Curve

The temperature-time curve of loess in a freeze-thaw cycle at different water content is presented in Fig. 1.

Fig. 1 illustrated that the freeze-thaw curves of different water content exhibited comparable trends. During the freezing phase, the temperature of loess initially decreased, followed by an increase before gradually decreasing until the slope of the curve reached zero. Eventually, the temperature stabilized at its freezing temperature. During the thawing phase, the temperature of loess initially underwent a rapid increase followed by a steady state upon reaching zero degree. Subsequently, it exhibited a relatively slow rise until the slope of the curve became zero, ultimately attaining the thawing temperature.

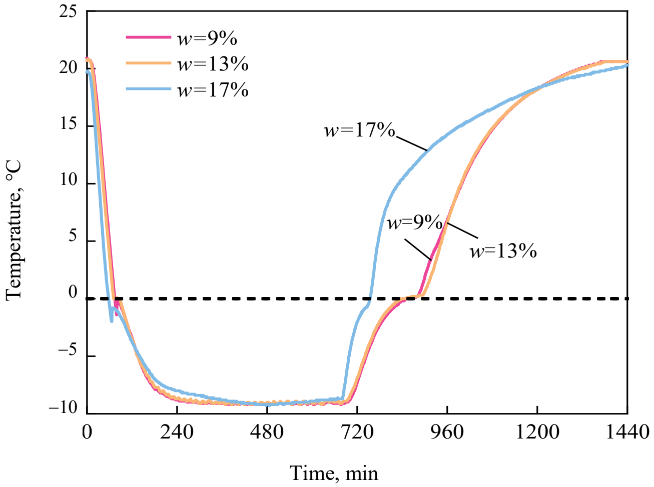
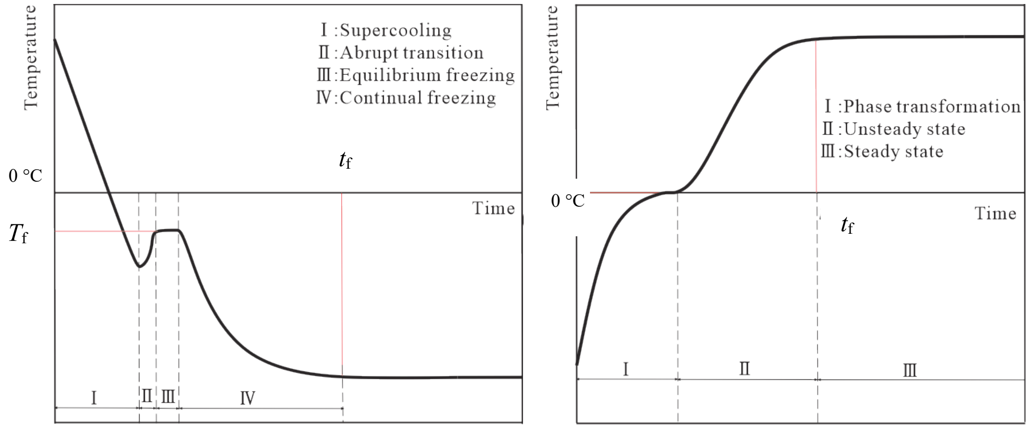


Fig. 1. Temperature-time curve of loess in a freeze-thaw cycle.

Fig. 1 also indicated that the sample with *w* = 17 % exhibited a relatively rapid rate of change during both freezing and thawing phase, in comparison to those with *w* = 9 % and 13 %, and the samples with three different levels of *w* essentially achieved the same state by the final stage of both freezing and thawing. In the freezing phase, the higher the water content, the greater the amount of free water and the reduced adsorption of water on soil particle surfaces, resulting in accelerated rates of freezing. In the thawing phase, the ascending section of the curve represented the process of heat absorption by ice crystals, the higher the water content, the greater the formation of ice crystals, resulting in enhanced heat absorption and accelerated melting rate. The soil samples all attained freezing temperature and melting temperature at the final stage, with little influence from *w*, however, there were variations in the amount of unfrozen water present in the samples. Furthermore, there existed disparities in the water content at the stabilization stage, which might be attributed to the varying positions during freezing and thawing.

Typical freezing and thawing curves of loess are given in Fig. 2.



(a) (b)

Fig. 2. Typical freezing and thawing curves of loess: (a) Freezing characteristics of loess; (b) Thawing characteristics of loess.

The freezing curves all exhibited obvious characteristics [23], [26] as shown in Fig. 2(a), including four typical stages: supercooling, abrupt transition, equilibrium freezing, and continual freezing. In the stage of equilibrium freezing, the relatively stable temperature is referred to as its freezing point T_f. While the temperature decreases and the slope gradually decreases until it attains zero in the continual freezing stage, the corresponding time at which the slope is zero is defined as the freezing time t_f. The process of thawing can be classified into three distinct stages based on temperature variation, namely phase transformation, unsteady state, and steady state [30]. The temperature increases rapidly during the phase transformation and stabilizes at zero degrees, which is defined as the thawing point T_t. Subsequently, the temperature continues to increase, albeit at a gradually decelerating rate, until the soil reaches an equilibrium state, the corresponding time at which the slope of zero is defined as the thawing time t_t.

3.2. Analysis of the Impact on Freezing Behaviour

Fig. 3 illustrated the freezing curve of loess across different freeze-thaw cycles and water content levels. As indicated, the temperature of loess with various water content presented consistent trends over time during the cooling process under different freeze-thaw cycles. During the supercooling stage, the significant temperature difference between the internal of the soil sample and the external temperature led to a notably higher cooling rate, causing a rapid decrease in its temperature within a short period. In the abrupt transition stage, the temperature rapidly rose to the freezing point, however, there was a variation in the extent of this increase, which represents the degree of supercooling. During the equilibrium freezing stage, the soil temperature remained relatively stable for a certain period, however, the duration of this stability varied significantly depending on the water contents and freeze-thaw cycles. The temperature kept decreasing in the continuous freezing stage, with a relatively slow rate of descent that varies under different conditions, until it reached a stable temperature.

The phenomenon of supercooling can affect the determination of soil phase and the estimation of physical parameters [31]. As shown in Fig. 3, the water contents and freeze-thaw cycles had a significant influence on the occurrence of the supercooling phenomenon. When $w = 9$ %, the soil exhibited a negligible supercooling phenomenon during $N \leq 3$, and a conspicuous one after $N = 10$. The comparison revealed that the soil sample with $w = 13$ % showed negligible supercooling phenomena in the initial three trials, and its behaviour resembled that of $w = 9$ % after $N = 20$. Conversely, for $w = 17$ %, the significance of supercooling gradually diminished with an increment in freeze-thaw cycles. Consequently, when calculating various physical parameter regarding the supercooling phenomenon in frozen soil, a comprehensive consideration of water contents and freeze-thaw cycles was necessary. The phase state and physical properties of the soil should be accurately judged, and the parameters should be reasonably selected.

It was also apparent from Fig. 3 that the decline curves of various water content were essentially the same in the supercooling stage. During the equilibrium freezing stage, the soil sample with 17 % water content showed a prolonged duration due to the phase transition of free water within its pores, which leads to energy release and temperature elevation. External coldness counteracted this effect by lowering the temperature, ultimately determined the duration of freezing time [22]. During the continuous freezing stage, the cooling rate decreased as the water contents increased.

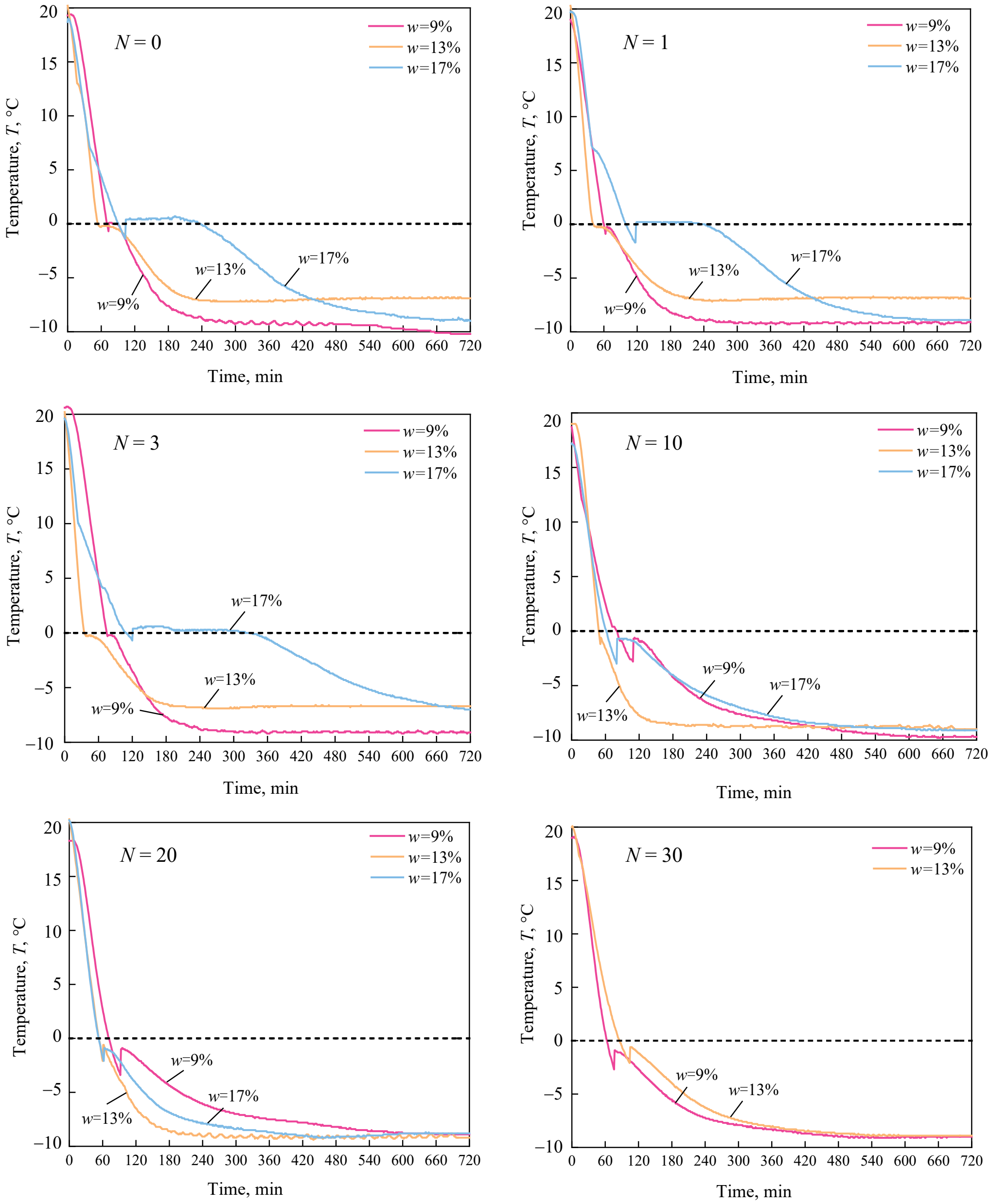


Fig. 3. Freezing curves with water contents *w* and freeze-thaw cycles (figure continues on the next page).

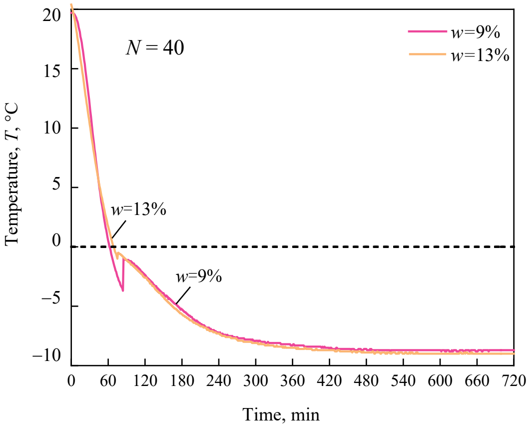


Fig. 3. Freezing curves with water contents *w* and freeze-thaw cycles.

The regularity curve between freezing point T_f and freeze-thaw cycles N is plotted in Fig. 4. It was observed from the results that there existed a remarkable influence of N on freezing point T_f, with a steady decline in the freezing point as N increment. However, it was worthy to note that the soil sample with $w = 9$ % manifested an increase in freezing point during $N = 3$. The freezing point of soil samples with low water content exhibited significant variations when $N \leq 3$, whereas the freezing point of those with high water content approached 0 °C, echoing that of distilled water. The freezing point of both high and low water content soil samples underwent abrupt fluctuations during $N = 10$, followed by a gradual alteration and demonstrated a tendency towards relative stability. Previous research had evidenced that the influence of N on freezing temperature was primarily ascribed to alterations in soil structure [25]. The structural disruption of the soil was typically most conspicuous during the initial cycles and gradually subsided over time. Consequently, the freeze-thaw cycles given rise to alterations in the physical and mechanical characteristics of the soil, as evidenced by experiments conducted on different types of soil [32].

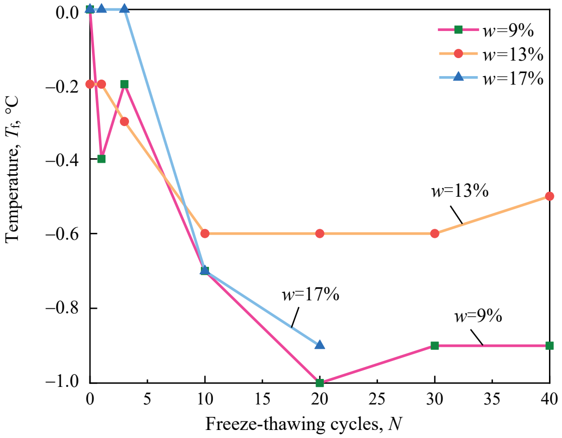


Fig. 4. The regularity curve between freezing point T_f and freeze-thaw cycles N.

The variations in freezing time (t_f) and stable temperature, as influenced by the number of freeze-thaw cycles (N) and water contents (w), are illustrated in Fig. 5.

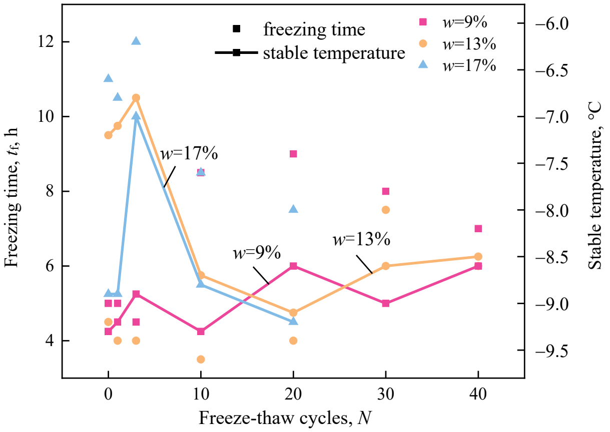


Fig. 5. The variation of freezing time t_f and stable temperature in relation to N and w.

It could be observed from the scatter diagram in Fig. 5 that the freezing time of the sample with 17 % water content, namely the blue scatter point, was comparatively lengthy. This phenomenon could be attributed to the fact that the higher the water content was, the larger the amount of free water would be, and the longer the time required to reach the freezing temperature. No significant regularity was absent in the variation of freezing time with freeze-thaw cycles N. The continuous occurrence of freeze-thaw cycles exerted an impact on the internal structure of soil and further affected the freezing process of soil samples. All soil samples attained a relatively stable temperature within 12 hours. Therefore, when conducting experiments, the freezing time should be determined in accordance with factors such as soil quality and water content.

It was evident from the point plot in Fig. 5 that the stable temperature varied in depend on the different water content. This phenomenon was attributed to the disparity in internal temperature resulting from the refrigeration mode employed in the low-temperature chamber., while the stable temperature of soil samples also exhibited discrepancy due to their different placement locations. The stable temperature also varied in accordance with different freeze-thaw cycles, owing to the inconsistency in stable temperature, it became challenging to determine the pattern of change. Consequently, further advancements should be carried out in subsequent experiments.

3.3. Analysis of the Impact on Thawing Behaviour

The temperature-time curves for thawing loess subjected to varying freeze-thaw cycles are plotted in Fig. 6.

Fig. 6 demonstrated a consistent tendency in the thawing curve under diverse freeze-thaw cycle, indicating a gradual rise in the temperature of the soil sample over time. During the initial stage, the temperature underwent a rapid rise and subsequently stabilized at zero degrees for a specific period, with variations in retention time being observed across different freeze-thaw cycle. The temperature increased gradually during the second stage, and freeze-thaw cycles exerted a certain influence on the rate of increase. During the final stage, the temperature remained relatively stable.

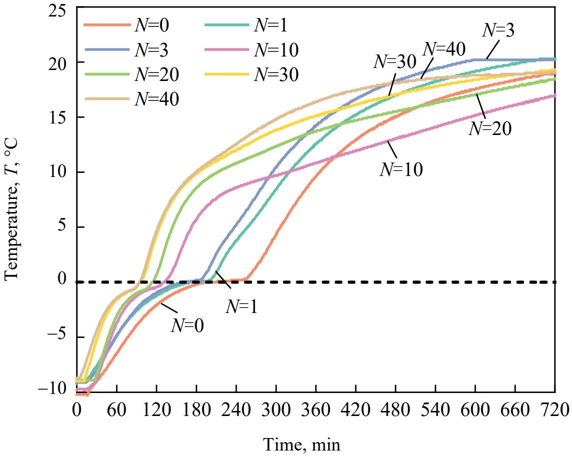


Fig. 6. The temperature-time curve for thawing loess with 9 % water content.

It could also be observed from Fig. 6, the stability levels of all curves at zero degrees differed, and the temperature at this point was referred to as the thawing point, denoted as $T_t = 0$ °C, resembling the process of pure water melting. During this phase, the soil sample absorbed heat while keeping a constant temperature. When $N = 3$, the temperature remained relatively stable after 10.5 hours, that is, $t_t = 10.5$ h. In all other cases, a gradual increase was noted, possibly attributed to the instability of the thawing temperature during the experiment. Consequently, future experiments should focus on improving this aspect. Furthermore, it is great significance to determine the appropriate thawing time for soil samples in accordance with specific working conditions.

Fig. 6 indicated that during the rapid ascent stage, there was a slight increase in ascent rate with an increase in N. The duration at zero degrees was longer when $N = 0$, and it gradually diminished to nearly zero with the increase of N. In the slow rise stage, the rise rate was relatively rapid during the initial three freeze-thaw cycles. However, when $N = 10$, there was a sudden decrease in the rise rate. Subsequently, when the freeze-thaw cycles exceeded ten, the rise rate became relatively slower. Furthermore, with an increase in N, the rise rate showed a gradual increment.

The thawing curves of loess with different water content and freeze-thaw cycle are presented in Fig. 7.

It could be observed from Fig. 7 that the change pattern of thawing curve remained consistent across various water content, with only a certain extent of influence on the rate of change. The longevity of soil samples with $w = 13$ % at 0 °C was significantly higher than that of soil samples with $w = 9$ % and 17 %, which exhibit extremely low durability. When $w = 9$ % and 17 %, the change trend of the curve was essentially identical, and the change trend of $w = 13$ % was significantly distinct from that of the curve. When $N = 10$ and 20, the soil samples with $w = 13$ % demonstrated a more conspicuous rate in the slow rising stage. When $N = 30$ and 40, the change trend of the curve was essentially identical, although a discernible temporal difference existed.

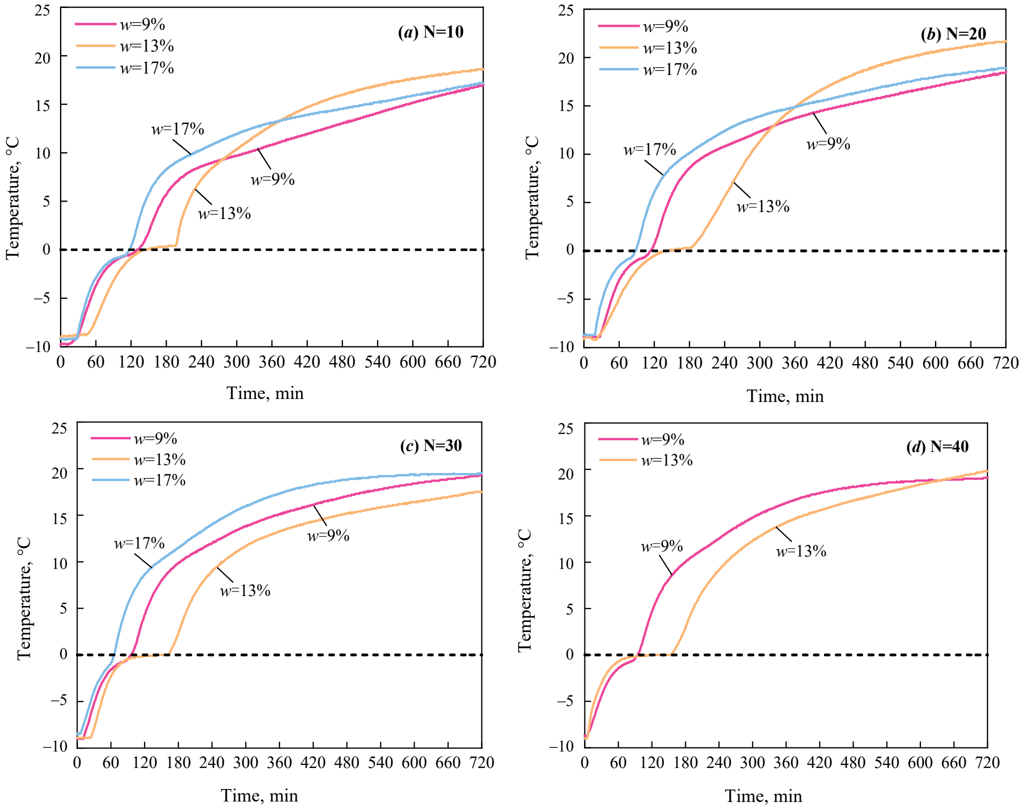


Fig. 7. The thawing curves of different water contents and freeze-thaw cycles.

4. CONCLUSIONS

In this research, freeze-thaw cycles experiment was conducted on loess in Yan'an, Shaanxi Province. The temperature-time curves of soil samples were measured under different water content levels and freeze-thaw cycles, and the analysis was performed to examine the influences of water content and freeze-thaw cycles on the temperature-time characteristics of loess. The following conclusions were drawn from the analysis:

1. The water contents and freeze-thaw cycles exerted a significant influence on the phenomenon of supercooling. When $w = 9$ % and 13 %, the supercooling phenomenon became gradually significant as the number of freeze-thaw cycles N increased, and the mutations occurred at $N = 10$ and 20, respectively. Conversely, for $w = 17$ %, the supercooling became progressively less significant as freeze-thaw cycles increased. The decline curves of various water contents were essentially the same in the supercooling stage, and the cooling rate decreased as the water content increased during the continual freezing stage.
2. The freezing point of loess decreased as the number of freeze-thaw cycles increased. The freezing point of soil samples with low water content exhibited significant variations during the initial three cycles, whereas the freezing point of those with high water content approached 0 °C. The freezing point of both high and low-water-content

soil samples underwent abrupt fluctuations during the tenth freeze-thaw cycle. Freezing time did not exhibit significant variations in relation to freeze-thaw cycles, however, a higher water content led to a longer freezing duration. All soil samples attained a stable freezing temperature within 12 hours.

3. The thawing curve under various freeze-thaw cycles exhibited a consistent trend, indicating a gradual increase rise in the temperature of the soil sample over time. The thawing point of soil samples remained constant at 0 ℃ under varying freeze-thaw cycles, however, the stability levels of all curves at zero degrees varied. Except for the third freeze-thaw cycle, all other cases exhibited a gradual increment after 12 hours, possibly attributed to the instability of the thawing temperature during the experiment.
4. During the rapid ascent stage of the thawing curve, the ascending rate slightly increased with the increase in the number of freeze-thaw cycle. In the slow rise stage, the rising rate was relatively rapid during the initial three freeze-thaw cycles, the rate of increase experienced a sudden drop at $N = 10$, and then proceeded at a relatively slower. The change pattern of the thawing curve remained consistent across various water content, with only a certain extent of influence on the rate of change.

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