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Article

Effects of coolant temperature on the characteristics of soil cooling curve

Dawei Wang[1], Yida Zhang[2]
[1] Boulder High School, Boulder, Colorado
[2] University of Colorado Boulder, Boulder, Colorado

SUMMARY

The cooling curves of soil are graphs that represent the variation of soil temperature with time when it is cooled. Knowledge of soil cooling curves and their characteristic parameters play an important role in most thermal analysis concerning soil freezing and/or thawing. Understanding the effect of environmental temperature on these parameters may help scientists better understand how frost heaves happen and how to predict the occurrence of frost heaves more accurately. For soils with identical type, water content, bulk density, and initial temperature, we believe that the freezing process and associated cooling characteristic parameters would be independent of the variation of external environmental temperature. To test this hypothesis, we carried out a series of freezing process experiments on soils with identical physical properties under different coolant temperature conditions. Here, the coolant temperature represents the magnitude of the environmental temperature. Our results indicate that the coolant temperature affects soil cooling curve profiles by changing the critical parameters of nucleation temperature, super-cooling, and phase-transfer duration of water in soil pores. According to the magnitude of the coolant temperature, soil cooling curves can be categorized into three different types: a "regular" freezing process without or with a little super-cooling, a sudden and small rise in temperature after the super-cooling "dip", and a permanent super-cooling of non-freezing process. We have shown that the freezing temperature of soils is not influenced by the variation of coolant temperature.

INTRODUCTION

Soil is a three-phase system consisting of solid particles, water, and air. The solid particles are small grains of different minerals and fragments of organic matter, which forms the skeleton of soils (1). The soil voids contain water and air in various proportions. To freeze soil is actually to freeze the water in soil pores. The previous research about water indicates that hot water often freezes faster than cold water (2). This means that the initial temperature will have an influence on water freezing speed. A cooling curve of soil is a temperature-time relationship graph that represents the phase transition of water in soil pores, typically from liquid to solid (1). Accurate cooling curve measurement data can be used to determine the critical parameters such as super-cooling degree, nucleation temperature, freezing temperature, and phase-equilibrium duration (3-5). All of these parameters are closely related to the existing form of pore water, the driving force for water migration in frozen soils, and the characteristics of cryogenic structure during the soil freezing process (6). The knowledge of soil cooling curves and their characteristic parameters play an important role in all thermal analysis concerning soil freezing and/or thawing.

In general, a typical soil cooling curve shows the sensible heat thermal storage stage, the latent heat release stage, the phase-change stage of free water and soil cooling stage without latent heat effects (**Figure 1**). When wet soil is cooled in an enclosed container, water in soil pores does not freeze at its freezing temperature (T_f) under atmospheric pressure condition. Instead, it is normally cooled below freezing temperature before ice nucleation takes place. At this time, the water in soil pores is called super-cooled water. Super-cooled water herein refers to a state of metastable liquid even though the water temperature is below its freezing temperature. The metastable state will end when ice nucleation occurs at nucleation temperature (T_n). At T_n, embryo nuclei form and grow to the critical sizes, and crystallization begins (7, 8). As a result of the release of the latent heat, temperature of

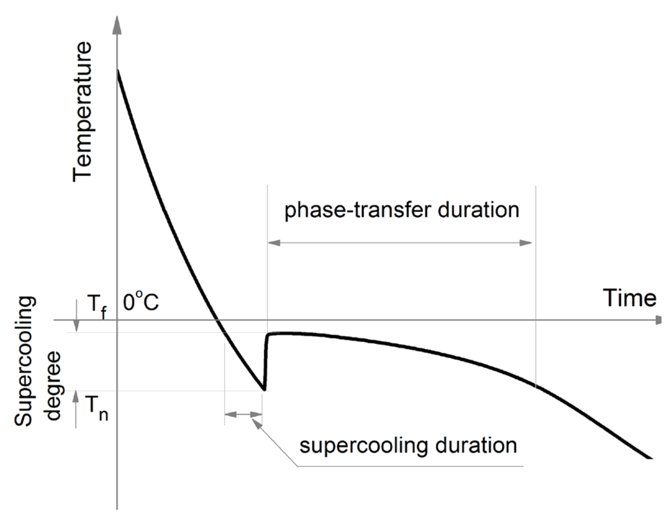

Figure 1. Typical soil cooling curve and its characteristic parameters. T_f represents the freezing temperature, where ice and water coexist inside the soil pores, and T_n represents the nucleation temperature, where embryo nuclei form and grow to the critical sizes, and pore water crystallization begins.

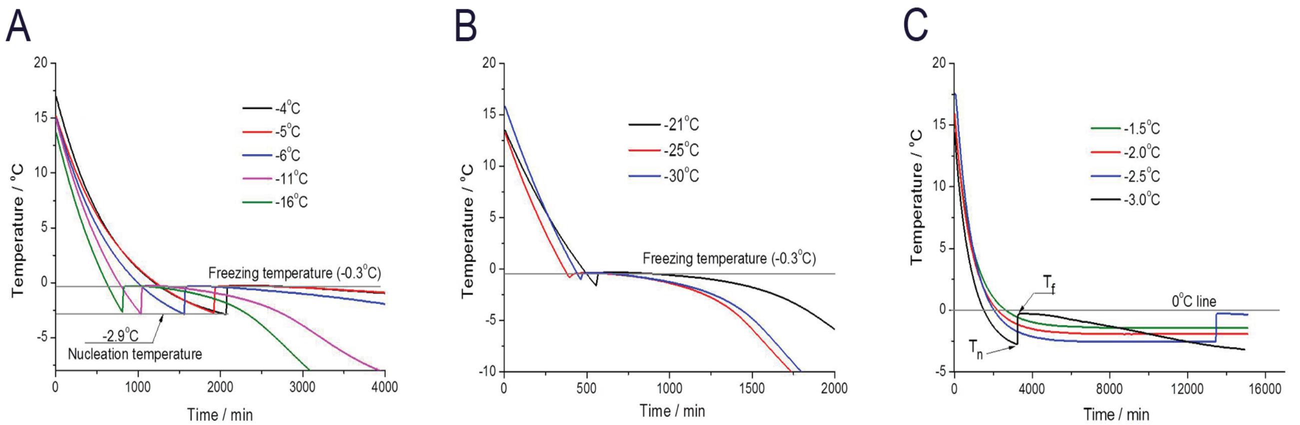

Figure 2: Cooling curves of freezing soil under different coolant temperature. (A) Soil temperature varied over time under the coolant temperature of -4, -5, -6, -11, and -16°C, respectively. (B) Soil temperature varied over time under the coolant temperature of -21, -25, and -30°C, respectively. (C) Soil temperature varied over time under the coolant temperature of -1.5, -2.0, -2.5, and -3.0°C, respectively.

the system rises to the value of T_f, the equilibrium freezing temperature (or freezing temperature), where all free water in soil pores will transfer into pore ice. Further extraction of heat leads to the decrease in temperature and successive freezing of the remaining unfrozen bound water.

In order to determine the threshold value of soil becoming frozen, most studies on properties of soil cooling curve are related to the freezing temperature of soils. For example, by applying the differential scanning calorimetry (DSC) technique, Kozlowski confirmed the strong dependency of the equilibrium freezing temperature (T_f) on the total water content and proved that freezing temperature could be expressed as a power function of the water content and the plastic limit (9). The State Key Lab of Frozen Soil Engineering in China observed that the freezing temperature decreased with increasing salt content and increased with increasing water content independent of the type of soil (10). Konrad and Morgenstern's study showed that the increase of external pressure caused a freezing temperature depression (11). As far as other parameters of soil freezing process are concerned, there is still little information available. If soil samples have an identical soil type, water content, bulk density and initial temperature, we hypothesize that the variation of external environmental temperature has no effect on the soil freezing process and cooling characteristic parameters. To test this hypothesis, we tested the soil cooling process under different coolant temperatures and measured how soil temperature changed over time. In our study, we used the variation of the coolant temperature as the variation of external environmental temperature. Our results may shed light on the understanding of soil cooling processes and may provide information on how the characteristic parameters of soil cooling curves change with the variation of external environmental temperature, which may help to better understand the formation mechanism of cryogenic structure and to predict the frost developing in the cold region's engineering.

RESULTS

We chose the temperature of coolant to match the environmental temperature where the frost-susceptible silty clay soil was frozen. According to the variation of atmospheric temperature in permafrost areas (-1°C to -30°C), the coolant temperature was set in this range with an interval of 4°C or 5°C. However, considering the approximate range of soil freezing temperature and complexity of soil freezing under higher negative temperature between -1 and -4°C, within this temperature range, the coolant temperature was set with an increment of 0.5°C.

Cooling curves of freezing soil under different coolant temperatures

First, we set the coolant temperature to -4, -6, -11, and -16°C, respectively, and measured the temperature-time curves of the tested soil samples (**Figure 2A**). Generally, the trends of the curves from our data were similar to data that has been previously found (**Figure 1**). All soil cooling curves have undergone the processes of super-cooling, latent heat release, and free water freezing. Their nucleation temperature and freezing temperature were -0.3°C and -2.9°C respectively, which did not seem to be influenced by the variation of coolant temperature.

Then, we set the coolant temperature to a lower temperature such as -21, -25, and -30°C, respectively. We found that these cooling curve profiles (**Figure 2B**) differed from the typical soil cooling curve (**Figure 1**). Some cooling curves did not undergo super-cooling process, while some had a short super-cooling stage before the soil temperatures reached the corresponding coolant temperature (**Figure 2B**). This phenomenon may be due to the effect of coolant temperature on the soil nucleation temperature. We also noticed that although the freezing temperature for all tested soil samples was -0.3°C, their nucleation temperature was different from each other (**Figure 2B**). For the soil immersed in the coolant with temperature of -30°C, the nucleation

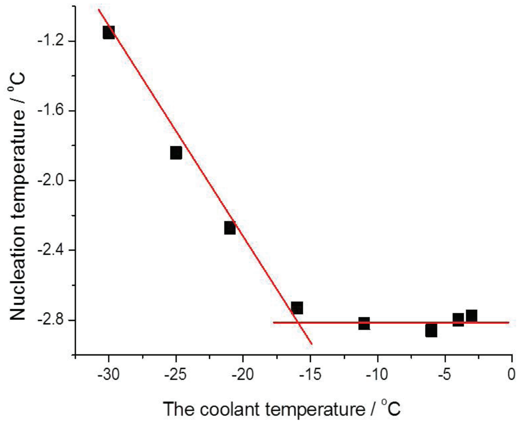

Figure 3: Nucleation temperature of soil at different coolant temperatures. Nucleation temperature (black squares) of soil across coolant temperatures. The red line shows the trend of nucleation temperature when the coolant temperature changes, which is obtained by fitting measured nucleation temperature.

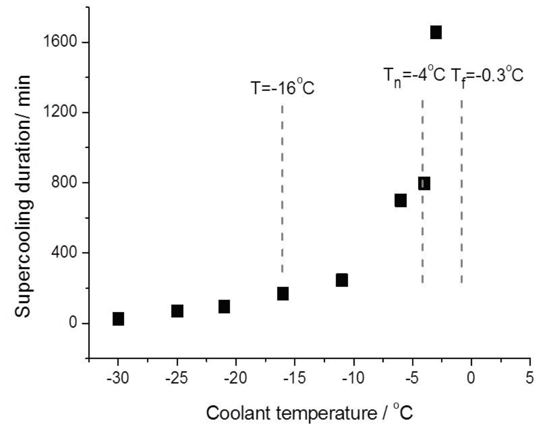

Figure 4: Super-cooling duration vs. coolant temperature. Small black squares represent the super-cooling duration of soil in the coolant with a certain temperature. The freezing temperature (T_f) is -0.3°C, the nucleation temperature (T_n) is -4°C, and the temperature of -16°C, below which the super-cooling duration decreases gradually.

temperature was close to its freezing temperature.

Lastly, we tested the temperature variation of soil in the coolant with a temperature close to or above the nucleation temperature, namely, in the range of -1.5 to -3°C (**Figure 2C**). We found that compared to the cooling curves obtained at lower coolant temperatures (**Figure 2A and 2B**), this group of cooling curve profiles were much more complicated (**Figure 2C**). Some temperature-time curves show the soil temperature had a stage of rapid increase, while some did not. More narrowly, when the coolant temperature was higher than the soil nucleation temperature obtained in **Figure 2A**, the soil temperature did not have a stage of rapid increase like what happened in the coolant with temperature lower than its nucleation temperature. This indicates that the moisture in soil pores will not be frozen even if the actual temperature of soil is lower than its freezing temperature (**Figure 2C**). So we cannot determine the freezing temperature of soil from this cooling curve. The moisture in soil pores are in a super-cooled state, namely in a liquid state, forever as long as there is no other external interference.

Effect of coolant temperature on nucleation temperature and super-cooling duration

Generally, the water nucleation temperature is the temperature at which water starts to transfer from liquid to solid, namely, the ice nuclei begin to form at this temperature. On the cooling curves, it is the lowest temperature point in its super-cooling state (**Figure 1**). Super-cooling duration is the total time water is in the super-cooling state, which can be calculated by the difference between the time when the soil temperature first reaches its freezing temperature and the time when the nucleation occurs (**Figure 1**). However, for the soils cooled in the coolant temperature above -4°C, the state of the pore moisture will change with the variation of coolant temperature and is in an unstable state (**Figure 2C**). So, we could neither determine the nucleation temperature, nor the super-cooling duration. The following analysis on the effect of coolant temperature on super-cooling duration will only consider the experimental results obtained with coolant temperatures below -4°C (**Figure 3 and 4**).

For the soil immersed in the coolant with temperature range from -30°C to -11°C, the super-cooling duration and nucleation temperature depended on the magnitude of coolant temperature. With increasing coolant temperature, the super-cooling duration increased gradually (**Figure 4**), whereas the temperatures of nucleation decreased sharply (**Figure 3**). However, for the soil cooled in the coolant with temperatures ranging from -11°C to -4°C, the super-cooling duration of soil pores water increased rapidly along with the increase of the coolant temperature (**Figure 4**), but there was no obvious variation in the magnitude of nucleation temperature (**Figure 3**). This means that when the soil was cooled between -11°C to -4°C, the nucleation temperature of the pore water in soil was maintained at a constant value without influence from environmental temperature, and the super-cooling duration increased remarkably with the increase of the environmental temperature.

Effect of coolant temperature on soil freezing temperature and phase-transfer duration

The freezing temperature of soil is defined as the phase transition temperature of water in soil pores, where liquid pore water changes to the pore ice. The relationship between the coolant temperature and the freezing temperature demonstrates that the freezing temperatures of the soil samples immersed in coolant with different negative temperatures were essentially equal; the mean value of the freezing temperature was -0.289°C with a corresponding standard deviation of 0.0309°C (**Figure 5**). This indicates that for certain types of soil with identical bulk density and water content, the changes of environmental temperature have no

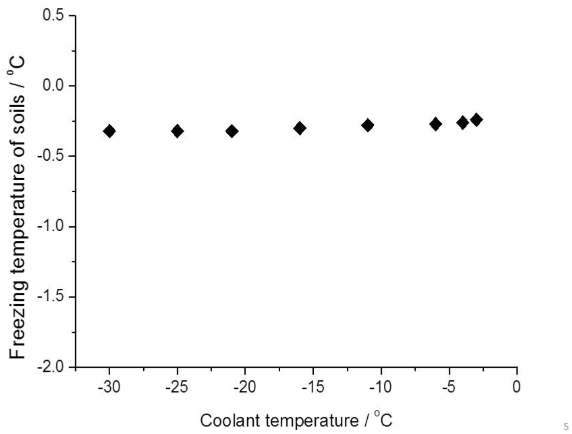

Figure 5: Soil freezing temperature vs. coolant temperature. Small diamond blocks represent the measured soil freezing temperature when soil is freezing in the coolant with different negative temperature. The mean value of freezing temperatures is -0.289°C with a standard deviation of -0.0309°C.

influence on the freezing temperature.

At freezing temperature, ice and water coexist inside the soil pores until the release of latent heat ends. The cooling curves of soil show that the temperatures of soils remain at freezing temperature for a long time and then gradually decrease with time (**Figure 2A and 2B**). Generally, we define the period from the time that the soil temperature reaches its freezing temperature to the time that the free water is completely frozen as the phase-transfer duration. The relationship between the coolant temperature and the phase-transfer duration suggests that the duration of phase-transfer of water in soil pores increases with the increment of coolant temperature (**Figure 6**).

DISCUSSION

The purpose of this study is to determine the influence of the environmental temperature on the cooling process of soils with identical physical properties. To do this, we carried out a series of soil cooling process experiments where we obtained the variation of soil temperature with time. Our results show that the environmental temperature influences the profile of soil cooling curves by impacting critical parameters in soil cooling curves such as nucleation temperature, super-cooling duration, and freezing temperature. In order to better understand this effect, we analyzed the response of these parameters to variation in the environmental temperature.

From the analysis of the cooling curves of freezing soil under different coolant temperatures, we conclude that the profile of the soil cooling curve is affected by the magnitude of the environmental temperature even if the initial temperature and the physical properties of the soil are identical. This is different from our hypothesis. According to the magnitude of coolant temperature, the soil will experience three different types of cooling processes: a "regular" cooling process with or without a little super-cooling, a sudden rise in temperature after the super-cooling "dip", and a permanent super-cooling of non-freezing process.

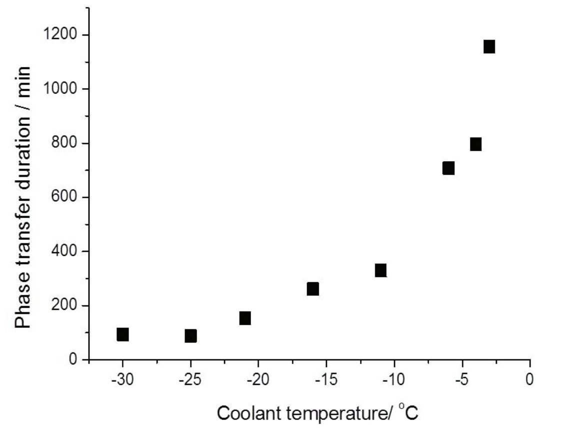

Figure 6: Phase transfer duration of soil across coolant temperatures. The duration of phase-transfer of water in soil pore increases from 100 minutes to 1200 minutes with the increment of coolant temperature from -30°C to -4°C.

Our results indicate that the magnitude of the coolant temperature has no effect on the soil freezing temperature, but it has a significant effect on the phase-transfer duration. With the increase of the coolant temperature, the phase-transfer duration increases gradually. This phenomenon is mainly dependent on the relative magnitude between the release rate of latent heat of soil and the absorption rate of latent heat in the surrounding environment (5, 12). If the latent heat release rate equals the absorption rate, the soil temperature will equal the freezing temperature. If the latent heat release rate is less than the absorption rate, the soil temperature will gradually decrease with time until the end of latent heat release in soil. The end of the latent heat release also means that almost all free water in the soil pores will be frozen into pore ice.

For soil samples with identical physical characteristics and initial temperature, when freezing under different environmental temperatures, their cryogenic structure might be different from each other. This difference will lead to a significant difference in soil frost heave (1). According to our research results about the effect of the coolant temperature on pore water nucleation temperature and super-cooling

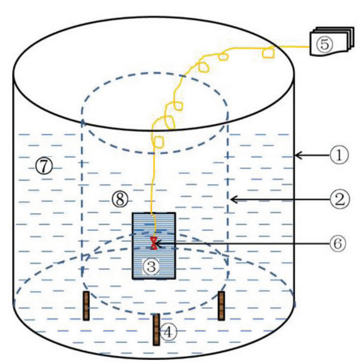

Figure 7: Schematic diagram of the experimental apparatus. The experimental set up included a (1) stainless-steel chamber, (2) isolated cooling tank, (3) aluminum sample cell and tested soil sample, (4) support, (5) data logger, (6) thermistor, (7) circulating coolant, and (8) stationary coolant.

duration, we hypothesize that the cryogenic structure and frost heavy of soils might relate to the pore water nucleation temperature and super-cooling duration. Therefore, in order to better predict the development of soil frost heave, we should investigate the cause of soil frost heave from the perspective of pore water nucleation temperature in future research studies.

METHODS

Soil Samples

The soil used in the freezing experiments was frost-susceptible silty clay taken from the Qinghai-Tibet Plateau. The soil physical parameter of liquid limit and plastic limit was 23.5% and 11.5%, respectively. The water content of the tested samples was 19% and the dry density was 1.78 g/cm^3. The grain size distribution is shown in **Table 1**.

Experimental Apparatus

The experimental apparatus was composed of a stainless-steel chamber, an isolated cooling tank, a thermistor, and a data logger. The stainless-steel chamber was a thermostatic chamber filled with circulating coolant (an alcohol-water solution). The temperature of coolant was controlled by a laboratory chiller with a precision of ±0.1°C. In order to alleviate the interference of vibration caused by coolant cycle on soil water nucleation, a small isolated cooling tank filled with the same kind of coolant was placed into the stainless-steel chamber. The tank was made of aluminum with a wall thickness of 0.5 mm to ensure the coolant temperature inside tank reached the outside tank temperature quickly by heat exchange. At the same time, the temperature of the coolant in tank was monitored in real-time by a thermistor immersed in the liquid coolant. The soil sample cells were placed in the center of the tank (**Figure 7**).

Experimental Approach

As a preliminary preparation for the experiment, a series of saturated soil samples were prepared first. According to the pre-determined results about the tested soils, its saturated water content and dry density were 19% and 1.78g/cm^3, respectively. We then calculated the amount of dry soil and distilled water required in our experiments and prepared soil samples in saturated state based on those values. A total amount of 1200 g dry soil was put into a vessel with 240 mL distilled water (a little more water was added because of evaporation loss) to achieve the required uniform water content of 19%. This moist soil was stored in an airtight container for about 24 hours to allow moisture uniformity. Then, we took out 55 g of moist soil and compacted it in three layers into an aluminum sample cell to form a saturated soil specimen (30 mm in diameter by 35 mm in depth). A thermistor with a precision of ±0.05°C was inserted into the tested soils before the aluminum sample cell was sealed hermetically. The thermistor was connected to a data logger to record the soil temperature variation during the process of soil cooling.

Table 1. Grain size distribution (%) of studied silty clay

	sand				clay
grain size (mm)	>0.5	0.25-0.5	0.1-0.25	0.074-0.1	<0.074
percent-age (%)	1.37	4.49	12.51	7.50	74.13

We prepared 22 samples using this method.

To avoid the effect of different initial temperature on soil cooling process, we made all testing soil samples had an identical initial temperature. Therefore, all sample cells filled with saturated soil were put into a thermostatic chamber for at least 24 hours to make soil sample's temperature equaled with the thermostatic chamber before the cooling process experiment began. The temperature of the thermostatic chamber was 15°C.

In order to measure the influence of environmental temperature on the soil cooling process, different environmental temperatures were applied to different soil samples to collect the soil temperature data during their cooling processes. In our investigation, the variation of coolant temperature represented the variation of environmental temperature for tested soil samples. Two identical soil samples were tested simultaneously for each coolant temperature to avoid contingency factors. Thus, when the coolant temperature in the stainless steel chamber arrived at a desired temperature, two soil samples were taken out from the thermostatic chamber and placed into the cooling tank promptly. The soil samples were cooled under a constant coolant temperature. Temperature variation over time was collected by the data logger simultaneously. Soil temperature data was collected every three seconds during the super-cooling to ice crystal growth stage and every ten minutes from the beginning and ending stage of soil freezing.

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