Effect of freeze–thaw cycling on the soil-freezing characteristic curve of five Canadian soils

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Abstract
The frozen soil processes and their interaction with the environment in the vadose zone of cold regions is vital in both agricultural and engineering practice applications. In a frozen soil, unfrozen water and pore ice coexist. The relationship between the unfrozen water content and subzero temperature is widely known as the soil-freezing characteristic curve (SFCC). The SFCC is a valuable tool for predicting the hydromechanical properties and for modeling the coupled thermal–hydraulic–mechanical–chemical process in frozen soils. In spite of its importance, the effect of freeze–thaw (F–T) cycling on SFCC has not been well investigated or understood. In this technical note, the effect of F–T cycles on the SFCC of five soils from cold regions of Canada were investigated. The SFCC (including both freezing and thawing branches) of the five soils for different F–T cycles were measured using frequency domain reflectometry (FDR) technique. The experimental results suggest that the effect of F–T cycles on the SFCC of the five soils is not significant. Such a behavior may be attributed to the destruction of soil structure during the saturation process. However, all the five soils’ SFCC exhibited hysteresis behavior for all the F–T cycles. The results of the study are valuable and contribute towards better understanding of the fundamental behavior of SFCC of various cold region soils.

1 | INTRODUCTION

Complex interactions among soils, water, the environment, and human activities occur in the vadose zone of seasonally frozen regions, which extend to ~50% of the exposed land in the northern hemisphere (Hayashi, 2013). In the pore spaces of a frozen soil, both unfrozen water and ice exist. Bouyoucos (1917) was one of the earliest investigators who demonstrated the existence of liquid water in a frozen soil–water system.

The unfrozen water mainly exists as thin films adsorbed on the surfaces of soil particles that are in equilibrium with the pore ice at temperatures typically below 0 °C (Harlan, 1973). The subzero temperature is the principal factor controlling the amount of water remaining in an unfrozen state. The relationship between unfrozen water content and subzero temperature in a frozen soil is widely referred to as the soil-freezing characteristic curve (SFCC) (Azmatch, Sego, Arenson, & Biggar, 2012; Koopmans & Miller, 1966; Mu, Zhou, Ng, & Zhou, 2019; Spaans & Baker, 1996; Watanabe & Wake, 2009). Several soil properties of importance in cold region engineering practice, such as the hydraulic conductivity (Azmatch et al., 2012), segregation potential (Konrad, 2001), resilient
modulus (Ren & Vanapalli, 2017), and strength (Agergaard & Ingeman-Nielsen, 2012; Akagawa & Nishisato, 2009), can be estimated using the SFCC as a tool. In addition, the SFCC is required in modeling the transport mechanism of water, heat, and solutes in frozen soils (Yu, Zeng, Wen, & Su, 2018; Zhang et al., 2016).

Two different approaches are commonly used for determining the SFCC of soils. The first approach focuses on the direct measurement of SFCC, with the aid of convenient and/or advanced testing equipment, which can be used in laboratory, in situ, or both. The principles, advantages, and/or limitations of these measurement methods are succinctly summarized by Ren (2019). The second approach focuses on the estimation of SFCC using soil physical properties, the similarity between SFCC and soil-water characteristic curve (SWCC), and/or physical and theoretical mechanisms. More details of the second approach are discussed in Ren (2019).

Ren and Vanapalli (2019) investigated the SFCC of two fine-grained soils from Toronto, Canada. Different freezing methods (temperature paths) were imposed on these two soils for undertaking extensive experimental investigations. In addition, the SWCC behavior of the two soils was measured and compared with their SFCC. The quantitative dissimilarity between the measured SWCC and SFCC of the two soils suggests that rigorous and more extensive investigations are required for better understanding fundamental behavior of the SFCC and its similarity to the SWCC.

Kozlowski and Nartowska (2013) examined the effect of freeze–thaw (F–T) cycling on the SFCC of several highly plastic bentonites with different water contents. The bentonite specimens were subjected to repeated freezing to −90 °C and thawing at 20 °C with free expansion in a differential scanning calorimeter (DSC). The thawing branches of SFCC during the applied five F–T cycles were obtained and compared. They concluded that the F–T effect on the SFCC of the bentonite specimens was statistically insignificant; however, reasons associated with this characteristic were not explained. On the other hand, they found that the effects of subzero temperature and soil type were highly significant on the SFCC behavior. This observation is consistent with the well-known behavior of frozen soils. In a study by He and Dyck (2013), the SFCC of a silty loam and a loamy sand were measured under two F–T cycles. The focus of their test was to investigate the effect of different final freezing temperatures on the SFCC of the two soils. From the results of their study they also found that the freezing branches under the two F–T cycles follow the same path (i.e., approximately the same); however, the thawing branches are different.

To date, the effect of F–T cycling on the SFCC is not well investigated or understood in the literature. For example, Kozlowski and Nartowska (2013) did not provide comparisons between the freezing branches of SFCC under different F–T cycles. Unfortunately, the DSC equipment that they used restricted their tests on tiny soil pastes with mass <20 mg. In addition, their study only focused on bentonite specimens that are highly plastic with a large specific surface area. Information related to widely available natural soils with low to moderate plasticity is lacking in the literature. The recent climate change also justifies the urgency of these investigation studies of various types of soils. According to the IPCC (2018), global warming effects will contribute to an increase in annual temperature of 1.5 °C above pre-industrial levels as early as 2030. This implies that more F–T cycling is likely in the vadose zone of cold regions, which covers a variety of soils with low to moderate plasticity such as clayey and silty soils, and glacial tills.

For this reason, focus of the present study is directed towards investigating the effect of F–T cycles on the SFCC behavior of five Canadian soils. This includes the two soils that were investigated by Ren and Vanapalli (2019) for understanding the similarity and dissimilarity between the SFCC and SWCC. The SFCCs of the five soils were experimentally determined on saturated specimens subjected to repeated freezing and thawing under closed-system conditions. The unfrozen water content of the soil specimens was measured by frequency domain reflectometry (FDR) technique without the effect of external stresses. The experimental investigation in the present study is valuable to better understand the fundamental behavior of the SFCC of fine-grained soils with low plasticity and is a novel supplement to the previous studies by Kozlowski and Nartowska (2013) and Ren and Vanapalli (2019).

### Core ideas
- The SFCCs of five fine-grained soils under freeze–thaw (F–T) cycling were measured and analyzed.
- The effect of F–T cycles on the SFCCs of the five soils is not significant.
- The five soils’ SFCCs exhibited hysteresis behavior for all the F–T cycles.

#### 2 SPECIMEN PREPARATION AND EXPERIMENTAL SETUP

Five Canadian soils (i.e., Toronto silty clay [Soil 1], Toronto lean clay [Soil 2], Kincardine lean clay [Soil 3], Ottawa Leda clay [Soil 4], and Indian Head till [Soil 5]) were investigated in the present study. The first four soils were collected from 0 to 3 m below the natural ground surface at different locations in southern Ontario. The fifth soil was a glacial till collected from Indian Head, SK, Canada. The natural soil samples were
TABLE 1  Physical and chemical properties of the five soils

| Property          | Toronto silty clay (Soil 1) | Toronto lean clay (Soil 2) | Kincardine lean clay (Soil 3) | Ottawa Leda clay (Soil 4) | Indian Head till (Soil 5) |
|-------------------|-----------------------------|---------------------------|-------------------------------|--------------------------|--------------------------|
| \( w_{L, \%} \)   | 20                          | 25                        | 31                            | 48                       | 36                       |
| \( w_{P, \%} \)   | 14                          | 13                        | 21                            | 22                       | 17                       |
| PI, \( \% \)     | 6                           | 12                        | 10                            | 26                       | 19                       |
| \( w_{opt, \%} \) | 13.5                        | 12.3                      | 20.3                          | 23.0                     | 13.9                     |
| \( \rho_{d_{max}}, g \, cm^{-3} \) | 1.915                      | 1.962                     | 1.631                         | 1.616                    | 1.839                    |
| \( G_s \)        | 2.68                        | 2.69                      | 2.71                          | 2.75                     | 2.72                     |
| Sand, \( \% \)   | 70                          | 45                        | 52                            | 43                       | 43                       |
| Clay, \( \% \)   | 16                          | 19                        | 25                            | 32                       | 30                       |
| USDA              | silt loam                   | loam                      | silt loam                     | clay loam                | clay loam                |
| Dominant minerals | quartz, dolomite, calcite, albite, microline | quartz, calcite albite, dolomite, blodite | quartz, muscovite, anorthite, ferro-pargasite, titanite, montmorillonite | quartz, dolomite, calcite, albite, muscovite |

\( w_L \), liquid limit, the water content of a soil at the boundary between the semiliquid and plastic states; \( w_P \), plastic limit, the water content of a soil at the boundary between the plastic and semisolid states; PI, plasticity index, the range of water content over which a soil behaves plastically (\( PI = w_L - w_P \)) (ASTM, 2018); \( w_{opt} \) and \( \rho_{d_{max}} \), the optimum water content and maximum dry density as determined by the Standard Proctor compaction test (ASTM, 2012); \( G_s \), specific gravity; USDA, USDA textural classification.

Determined by X-ray fluorescence (XRF) and X-ray diffraction (XRD) tests.

FIGURE 1  The gradation curves of the five soils

Air dried for 2 wk and then pulverized and passed through a 2-mm sieve. The basic physical and chemical properties of the five soils, such as the liquid and plastic limits, sand/silt/clay fraction by weight, classification, and mineralogical compositions by dominance are summarized in Table 1. Their respective gradation curves are shown in Figure 1.

For the measurement of SFCC, compacted cylindrical specimens after saturation were used. A certain amount of dry soil and water were hand mixed, passed through a 2-mm sieve, and sealed in plastic bags for achieving uniform water distribution. The specimens were compacted into five equal layers (each layer being 20 mm thick) by the volume control method, with a final height and diameter of 100 and 50 mm, respectively. The dry densities and void ratios of the compacted specimens are shown in Table 2. After compaction, the specimens were securely wrapped with filter paper and cling wrap and then submerged in water for saturation. The specimens were allowed to imbibe water gradually along

TABLE 2  Properties of the soil specimens after compaction and saturation

| Property          | Soil 1 | Soil 2 | Soil 3 | Soil 4 | Soil 5 |
|-------------------|--------|--------|--------|--------|--------|
| After compaction  |        |        |        |        |        |
| \( \rho_{d1}, g \, cm^{-3} \) | 1.926  | 1.954  | 1.631  | 1.621  | 1.843  |
| \( e_1 \)         | 0.391  | 0.377  | 0.662  | 0.697  | 0.476  |
| \( \psi_{opt}, kPa \) | 40     | 260    | 90     | 200    | 190    |
| After saturation  |        |        |        |        |        |
| \( w, \% \)       | 14.6   | 16.0   | 23.4   | 25.5   | 17.5   |
| \( \Delta V, cm^3 \) | -0.4  | 3.1    | 1.0    | 1.7    | 3.3    |
| \( \rho_{d2}, g \, cm^{-3} \) | 1.933  | 1.895  | 1.615  | 1.595  | 1.784  |
| \( e_2 \)         | 0.386  | 0.419  | 0.678  | 0.725  | 0.525  |
| \( \theta, cm^3 \, cm^{-3} \) | 0.281  | 0.304  | 0.378  | 0.407  | 0.312  |
| \( S, \% \)       | 101.1  | 102.8  | 93.5   | 96.7   | 90.6   |

Note: \( \rho_{d1} \) and \( e_1 \), the dry density and void ratio of the specimen after compaction at the \( w_{opt} \); \( \psi_{opt} \), the approximate suction of the specimen after compaction at the \( w_{opt} \), according to Ren (2019); \( w, \Delta V, \rho_{d2}, e_2, \theta, \) and \( S \), the gravimetric water content, volume expansion, dry density, void ratio, volumetric water content, and degree of saturation of the specimen after saturation (i.e., before soil-freezing characteristic curve [SFCC] test), respectively. The mass and dimensions of the specimen after compaction and saturation were measured, and the \( \rho_{d1}, e_1, \Delta V, \rho_{d2}, e_2, \theta, \) and \( S \) were calculated. The degrees of saturation of Soil 1 and Soil 2 are slightly larger than one, which may be due to experimental errors.
their height. The saturation process was stopped when the mass of the test specimens reached equilibrium values. The soil specimens generally had certain expansion during the saturation process, as summarized in Table 2. Table 2 also summarizes the gravimetric water contents of the tested specimens together with their dry densities and void ratios after saturation. In addition, the volumetric water contents and degrees of saturation were calculated from the mass–volume relationships of the five soils and are summarized.

The EC-5 moisture sensor, RT-1 temperature sensor, and EM50 data logger (manufactured by the METER Group) were used for measuring the SFCC of the five soils. The EC-5 sensor is based on the FDR technique. The RT-1 was inserted into one end of the specimen, and the EC-5 was inserted into the other end. The sensors were carefully installed to ensure good contact between the sensors and soil specimen. No plasticine was pasted around the overmolding of the EC-5, as it could contribute to errors. More details regarding the calibration of the two sensors for obtaining SFCC can be found in Ren and Vanapalli (2019). The soil specimens were tightly sealed by cling wrap to prevent moisture loss during the testing period. Three-dimensional freezing and thawing of the specimens (without subjecting them to external stress) were fulfilled by putting them in a freezer. In other words, the SFCC test was conducted under zero external stress conditions. Up to three F–T cycles were imposed on the tested specimens. The unfrozen water content and temperature of the specimens were continuously measured during the applied F–T cycles. All tests were performed by step-freezing and step-thawing, which means that different temperatures were controlled along freezing and thawing paths. The soil specimens were kept at least 12 h under each controlled temperature for achieving uniform temperature conditions. Both the freezing and thawing branches of the SFCCs of the five soils were determined.

3 | EXPERIMENTAL RESULTS

Figure 2 shows the temperature–time curves of the specimen of Soil 3 subjected to three F–T cycles. It can be seen that the temperature–time curves are similar for all the three F–T cycles. The temperature of the Soil 3 specimen decreases and stabilizes under each controlled temperature. The pore water in the specimen is supercooled as the temperature decreases. When a certain temperature is achieved, ice nucleates in the pore spaces and a large amount of latent heat is released. As a result, the soil temperature significantly increases from the supercooling temperature to the freezing temperature. The results shown in Figure 2 suggest that the supercooling temperatures of the three F–T cycles are scattered, whereas the freezing temperatures are approximately the same, around −1.0 °C. Table 3 summarizes the supercooling and freezing temperatures of the five soils that were subjected to F–T cycles. It can be concluded that the freezing temperatures of the five soils are relatively stable, whereas their supercooling temperatures are randomly distributed. In other words, the F–T cycles do not show significant influence on the measured SFCC of the five soils. The measured SFCCs of the five soils subjected to F–T cycles are shown in Figure 3. The open circle data point is within the temperature range of 0 to −5 °C. It can also be seen from Figure 3 that the F–T cycles do not have significant influence on the freezing temperature for the five soils.

![Figure 2](image.jpg)

**FIGURE 2** The temperature–time curves of the specimen of Soil-3 subjected to three freeze–thaw cycles

| Soil  | Freezing temperature | Supercooling temperature |
|-------|----------------------|--------------------------|
|       | 1st cycle | 2nd cycle | 3rd cycle | 1st cycle | 2nd cycle | 3rd cycle |
| Soil 1 | −0.6 | −0.6 | NA | −2.5 | −2.8 | NA |
| Soil 2 | −0.6 | −0.5 | NA | −3.8 | −2.8 | NA |
| Soil 3 | −1.1 | −1.0 | −0.9 | −4.4 | −4.3 | −3.3 |
| Soil 4 | −0.5 | −0.4 | −0.4 | −2.5 | −2.3 | −2.8 |
| Soil 5 | −0.7 | −0.7 | −0.7 | −1.7 | −1.8 | −2.3 |

*NA, not applicable.*

Table 3 lists the freezing and supercooling temperatures in each F–T cycle for the five soils. The results show that the freezing temperatures are approximately the same, around −1.0 °C. However, the hysteresis is only significant in the temperature range of 0 to −5 °C. It can also be seen from Figure 3 that the F–T cycles do not have significant influence on the measured SFCC of the five soils. The thawing curves of SFCC are overlapped. The freezing curves show some differences, due to the random supercooling phenomenon. However, it is important to note the relatively large fluctuation in their SFCC within the temperature range of 0 to −5 °C. The
sensors sensitivity should have contributed to the fluctuations in the measured results of the SFCC.

Figure 4 shows the typical soil specimens after SFCC measurement. As can be seen for Soil 1, the adjacent two compacted layers can be separated due to the influence of saturation and F–T cycling. This is because Soil 1 has a high silt content and low clay fraction, as summarized in Table 1. This property can induce large fluctuation in the measured SFCC of Soil 1. Soil 2 and Soil 3 were relatively intact, and Soil 4 had minor cracks. However, Soil 5 cracked when the moisture sensor was inserted into it. The influence of the cracks on the measured SFCC needs more investigation.

4 | DISCUSSION

In the present study, the soil specimens were initially compacted at their optimum moisture contents and were at an unsaturated condition. The compacted specimens had relatively high suction values, as summarized in Table 2.
When the specimens were submerged in water for saturation, water was imbibed by the specimens due to suction gradient. Consequently, the water films around soil particles in the specimens became thicker and the radii of curvature of the air–water interface became larger, indicating a lower suction value. The distance between soil particles was enlarged (i.e., volume expansion) due to the reduction in suction. In other words, the initial compacted structure of the specimens was destructed during the saturation process. As a result, during the F–T cycling process for SFCC measurement, no significant change in soil structure occurred. Therefore, F–T cycles do not show significant influence on the measured SFCC of the five soils. The experimental results on the resilient moduli of the same five soils subjected to wetting and F–T cycles are consistent with this conclusion, according to Ren, Vanapalli, Han, Omenogor, and Bai (2019). They found that for specimens that were initially compacted at the optimum moisture content and then subjected to wetting, the effect of F–T cycles on their resilient moduli was insignificant, due to soil structure destruction during the wetting process.

On the other hand, large fluctuation in the measured SFCC was observed for several specimens, especially for Soil 5. Such a behavior may be attributed to the cracks in Soil 5 specimens that arise during the testing process, as can be seen from Figure 4e. It is also possible that the performance of the EC-5 moisture sensor (e.g., its stability) contributed to the fluctuation in the measured unfrozen water content. This may overshadow the actual effect of F–T cycles, if there are any, on the SFCC. The FDR technique used in the present study has the merits of low cost, fast acquisition, portability, and suitability for both laboratory and field measurements. On the other hand, the FDR sensor is greatly dependent on salt content and soil type, and air gaps around the sensor can profoundly influence its readings. In summary, the F–T cycles do not show significant effect on the measured SFCC of the five soils, based on the testing methods used in the present study.

There is no significant difference between the measured SFCC for different F–T cycles in the low temperature range (i.e., lower than −5 °C). Such a behavior may be attributed to the adsorptive forces that are dominant when the soil temperature is low, since the clayey particles on which unfrozen water is mainly adsorbed were not influenced by F–T cycling. This may partly explain why Kozlowski and Nartowska (2013) did not observe significant F–T effect on the SFCC because they used highly plastic bentonites (with large specific surface area), whose water retention capacity is dominated by adsorptive forces. In the relatively high temperature range of 0 to −5 °C, the different supercooling temperatures in different F–T cycles resulted in differences between the freezing branches of SFCCs. However, this difference should not be attributed to the effect of F–T cycles on SFCCs.

In the present study, the saturated compacted soil specimens were not subjected to any stress conditions during SFCC measurement. In other words, the effect of stress on SFCCs is not considered. Mu et al. (2019) investigated the effect of confining stress on the SFCC of a lean clay and a silty sand. The cylindrical soil specimens with certain dry densities (void ratios) were prepared by compaction, saturation, and consolidation. The SFCCs of the two soils were measured under constant confining stresses, along a three-dimensional freezing and thawing path. They found that the specimen under a higher confining stress was able to retain more unfrozen water than that under a lower stress. Such a behavior can be attributed to the reduction of the pore sizes in the soil specimen under higher confining stress. As a result, more unfrozen water can be retained due to enhanced capillarity. They also found that the stress effect on the SFCC was more significant for clay than for sand. It was reasoned that the stress-induced change in pore size distribution was larger in clay due to its higher compressibility.

Soils in the vadose zone of cold regions are not only subjected to temperature fluctuation, but also to various stress conditions. Therefore, the combined effect of F–T cycles and stress levels on the SFCCs of soils at various saturation levels merits further investigations. It is generally acknowledged that F–T cycles can contribute to a significant change in soil pore structure, or void ratio, or density. This tendency of change in pore structure may be balanced by external stresses, which can compress or consolidate the soil specimen. However, the relative contribution to the change in soil structure (and therefore in SFCC) by F–T cycles and external stresses may be related to factors such as the saturation level of the soil specimen, the freezing and thawing conditions, and the magnitude of the external stresses.

FIGURE 4 Photos of the five soils after soil-freezing characteristic curve (SFCC) test

(a) Soil-1: weak bond between adjacent compacted layers;  
(b) Soil-2 and (c) Soil-3: relatively intact;  
(d) Soil-4 and (e) Soil-5: slightly cracked. 
The hole in (b) was for RT-1; and that in (c), (d), and (e) was for EC-5.
5 SUMMARY

Many important physical and mechanical properties of frozen soils that are of interest in agricultural and engineering practice applications can be estimated using the SFCC. In addition, the SFCC is essential in modeling the water and solute dynamics in the vadose zone of cold regions. For this reason, the SFCC has attracted continuous attention of researchers and engineers during the last five decades. However, the effect of F–T cycles on the SFCC has not been well investigated or understood in the literature.

The effect of F–T cycling on the SFCC of five Canadian soils with low to moderate plasticity were investigated in the present study. The SFCCs of the five soils were measured by FDR technique, and up to three F–T cycles were considered. The experimental results show that the effect of F–T cycles on the measured SFCC is not significant. This is mainly attributed to the destruction of soil structure during the previous saturation process, and the effect of F–T cycles on soil structure (and therefore on SFCC) was not clearly observed. The hysteresis of the SFCC is observed for all the five soils under each F–T cycle. The changes in SFCCs under the effect of both F–T cycling and stress levels should be investigated, as in situ soils in cold regions are not only subjected to F–T cycles, but also to various stress conditions.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

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