Comparison of Soil-Freezing and Soil-Water Characteristic Curves of Two Canadian Soils

Junping Ren and Sai K. Vanapalli*

The drying–wetting and freezing–thawing cycles significantly influence the soil pore water in the vadose zone in permafrost and seasonally frozen regions. The soil-freezing characteristic curve (SFCC) describes the relationship between unfrozen water content and subzero temperature in a soil at frozen condition. Several studies suggest that the SFCC of a frozen saturated soil is similar to soil-water characteristic curve (SWCC), which describes the relationship between water content and suction for a soil under unfrozen unsaturated condition. In the present study, the similarity between SFCC and SWCC, and possible reasons for the hysteresis of SFCC are succinctly reviewed. The SFCC and SWCC of two Canadian soils were measured and critically interpreted to understand the fundamental behavior of SFCC in comparison with the SWCC. The observed hysteresis of SFCC for the two soils was mainly associated with the supercooling of pore water. The measured SFCC and SWCC of the two soils show quantitatively dissimilarity rather than similarity. This may be attributed to the experimental limitations and possible fundamental differences between drying–wetting and freezing–thawing processes. In addition, several concerns regarding the similarity between SFCC and SWCC are discussed. The present study highlights that rigorous investigations are required for better understanding the SFCC to facilitate its use for cold-region engineering practice applications.

Abbreviations: FDR, frequency domain reflectometry; NMR, nuclear magnetic resonance; SFCC, soil-freezing characteristic curve; SLS, solid–liquid–solid; SS, solid–solid; SSLS, solid–solid–liquid–solid; SWCC, soil-water characteristic curve; TDR, time domain reflectometry; TLC, Toronto lean clay; TSC, Toronto silt clay.

The soil pore water in the vadose zone in permafrost and seasonally frozen regions is significantly influenced by both drying–wetting and freezing–thawing cycles. Unfrozen water and ice coexist in a frozen soil. The amount of unfrozen water and ice is predominantly dependent on subzero temperature. The relationship between unfrozen water content and subzero temperature is commonly referred to as the soil-freezing characteristic curve (SFCC) (Koopmans and Miller, 1966; Spaans and Baker, 1996; Azmatch et al., 2012). The subzero temperature can be converted to cryogenic suction (which is defined as the pressure difference between pore ice and pore water) by using the Clapeyron equation, based on certain assumptions and thermodynamic equilibrium condition between ice and water phases.

The amount of unfrozen water change contributes to the variation of the physical and mechanical properties of frozen soils. For this reason, SFCC can be used as a tool in the interpretation of frozen soil behavior. Several studies highlight SFCC of frozen saturated soil is similar to soil-water characteristic curve (SWCC), which describes the relationship between water content and suction, of unfrozen unsaturated soil (Koopmans and Miller, 1966; Spaans and Baker, 1996). The wide use of SWCC in the prediction of unsaturated soil properties is encouraging researchers and practitioners in recent years to extend similar philosophy in engineering practice for frozen soils using the SFCC.

For example, Azmatch et al. (2012) measured the SFCC of Devon, Alberta, Canada, silt specimens that were consolidated using different pressures. The SFCC of these specimens were expressed as a relationship of unfrozen water content vs. cryogenic suction and
fitted using the well-known Fredlund and Xing (1994) equation. The hydraulic conductivities of the frozen Devon silt specimens were estimated using the fitted SFCC along with a saturated hydraulic conductivity value extending the same philosophy used for unfrozen unsaturated soils.

Ren and Vanapalli (2018) proposed a semiempirical model for estimating the resilient modulus of frozen soils at saturated condition using SFCC as a tool. The unfrozen water content vs. subzero temperature relationship was converted to the relationship between degree of unfrozen water saturation and cryogenic suction. The later relationship was then fitted using the Fredlund and Xing (1994) equation. Experimental data of seven different saturated soils were used to validate the model, and good agreement was achieved between the measured and estimated frozen resilient modulus values.

The reliable determination of SFCC is the primary step to use SFCC in the prediction of frozen soil properties. Two different approaches are commonly used for determining the SFCC. The first approach focuses on the direct measurement of SFCC. Several methods such as the dilatometry, calorimetry, frequency domain reflectometry (FDR), time domain reflectometry (TDR), and nuclear magnetic resonance (NMR), have been used in laboratory and in situ during the past few decades (Anderson and Morgenstern, 1973; Yoshikawa and Overduin, 2005). A significant amount of data is published in the literature following these methods. The second approach focuses on the estimation of SFCC from soil physical properties, or using the similarity between SFCC and SWCC and/or physical and theoretical mechanisms (Anderson and Tice, 1972; Liu and Yu, 2013; Wang et al., 2017; Amiri et al., 2018).

There are limited studies in the literature for better understanding the fundamental behavior of SFCC to facilitate the development of reliable models for the design of civil infrastructure such as pavements, foundations, and pipelines. For this reason, it is important to understand the strengths and limitations of the presently used experimental and empirical methods for the determination or estimation of SFCC. To address these objectives, the SFCC and SWCC of two soils from Toronto, Canada (i.e., Toronto silty clay [TSC] and Toronto lean clay [TLC]) were investigated. Several different testing conditions were imposed to determine the SFCC. Some of the testing scenarios were directed to study the freezing and thawing branches of SFCC to understand the hysteretic characteristics of the two soils. In addition, comparisons were provided between the SFCC and SWCC for both the soils. The results of the study provide valuable information for better understanding the SFCC in general and the two tested soils from Toronto, Canada, in particular.

Background

Similarity between the Two Characteristic Curves

The physical process that a soil undergoes during drying and wetting cycles under unfrozen condition has similar characteristics to the soil that is subjected to freezing and thawing cycles. When an initially saturated soil is subjected to drying, water is gradually removed and replaced by air, leaving the remainder water at an increasingly lower matric potential. In a similar fashion, liquid water in a saturated soil changes phase and gradually becomes ice when it is subjected to freezing. Two different mechanisms contribute to matric potential; namely, the adsorptive force and capillary force. The adsorptive force acts on the mineral–water interface. Water is adsorbed by soil particles as thin films. Therefore, it is considered immaterial whether ice or air is present on the other side of the water film; for this reason, reductions of soil matric potential in frozen and unfrozen conditions due to adsorptive forces can be considered identical (Spaans and Baker, 1996). The capillary force at the interface of different phases, such as air–water, ice–water, and air–ice interfaces, is proportional to the surface energy of the interface. The surface energy of air–water interface is higher than that of ice–water interface (Spaans, 1994). For this reason, reductions of soil matric potential associated with capillary force are different in a soil that is at frozen and unfrozen conditions.

Koopmans and Miller (1966) were the pioneers who measured both SFCC and SWCC for three different types of soil (i.e., solid–solid [SS], solid–liquid–solid [SLS], and solid–solid–liquid–solid [SSSL]). The SS soil is free of colloidal materials and represents soils such as sand, silt, or coarse clay fractions that have direct solid-to-solid (SS) contacts between particles. In SS type soils, each particle is wedged with the other adjacent particles and the pore geometry is fixed. A change in water content causes a displacement of the air–water interface within the pore system; however, there will be no change in the bulk volume of the soil. The other extreme scenario relates to the soil particles are always separated by liquid water, designated as the SLS (i.e., solid–liquid–solid) soil. A change in water content is accompanied by corresponding changes in particle spacing and in the bulk volume. Macroscopic cracks may open or close due to changes in water content; however, the air phase does not penetrate the spaces between particles. The schematic representation of the SS and SLS soils is available in Miller (1980). In reality, the behavior of a majority of natural soils lies in between the SS and SLS; for this reason, they are designated as SSSL soil.

It is assumed that adsorptive force dominates the water retention behavior of the SLS soil. The SFCC and SWCC are expected to be identical and do not require any adjustment if soil water is at a similar state in the frozen and unfrozen SLS soil. However, because of the difference between the surface energy of air-water and ice-water interfaces in a SS soil, an adjustment factor is required to relate its SWCC to SFCC (Koopmans and Miller, 1966; Miller, 1980). Figure 1 shows the measured SFCC and SWCC of these three different types of soils. As can be seen, by using an adjustment factor of 2.2 for the SS and SSSL soils and with no adjustment for the SLS soil, good agreement was achieved between the SFCC and SWCC. However, it is of interest to note that a value of 2.2 would lead to erroneous result for the SSSL soil from a theoretical point of view. Investigators were also inspired to find an adjustment
factor that is dependent on temperature, since most soils behave as SSLS soils, which are not predominantly influenced by either capillary or adsorptive force (Kurylyk and Watanabe, 2013). Soil pore water freezes gradually, and the governing force (capillary or adsorptive) may switch with a decrease in temperature. At higher subzero temperature, pore water is mainly present as the result of capillary force, since the curvature of ice–water interface is significant in this situation. However, when there is a further decrease in temperature, most pore water becomes ice and a thin adsorbed film of water is predominantly available. At this stage, adsorptive force dominates and no capillary force is considered (Lebeau and Konrad, 2012; Kurylyk and Watanabe, 2013).

Hysteresis of the Soil-Freezing Characteristic Curve

The SFCC exhibits hysteretic behavior similar to the SWCC. This can be attributed to several possible mechanisms:

1. Supercooling of pore water (Bittelli et al., 2003; He and Dyck, 2013; Tian et al., 2014; He et al., 2015). Soil pore water does not necessarily freeze when its freezing temperature is reached. Instead, pore water remains in a liquid phase and is supercooled to a lower temperature, until freezing is induced by nucleation.

2. The effect of electrolytes (Bittelli et al., 2003; He and Dyck, 2013; Tian et al., 2014; He et al., 2015). Since electrolytes will be excluded from ice when soil is subjected to freezing, the solute concentration in the remainder of pore water becomes larger, which contributes to freezing point depression of pore water and yields hysteresis.

3. Pore geometry (Anderson et al., 2009). Hysteresis may be attributed to differences in ice–water interface curvatures during crystallization and melting, especially in soil that contain a notable component of pores that are cylindrical.

4. Pore blocking (Bittelli et al., 2003; Anderson et al., 2009). A bottle-neck effect (originating from large pores with narrow necks), which is considered a primary cause of the hysteresis commonly observed for air–water phase transition in soil with a wide distribution of interconnected pores of different radii, may similarly lead to the hysteresis for ice–water phase transition in the soil.

5. The effect of contact angle (Liu and Yu, 2013; Zhou et al., 2019). The advancing contact angle during soil freezing is different from the receding contact angle during soil thawing.

6. The change in pore structure. The thixotropic property and aging effects that influence pore size distribution may contribute to hysteresis (Liu and Yu, 2013).

In addition, soil particles typically move from their original positions as soil pores are enlarged due to ice expansion during freezing process. Therefore, there would be more large pores than prior to soil freezing. When soil is subjected to thawing, pore ice melting initiates in small pores first and then progresses successively to larger pores (Tan et al., 2015). For this reason, for the
same unfrozen water content, temperature on the thawing branch is higher than that on the freezing branch. In other words, the thawing branch is below the freezing branch (i.e., hysteresis). Nevertheless, the reasons for hysteresis in porous media such as soils are still not well understood. This may be associated with the complexity of pore structure, which probably comprises various heterogeneous (at the pore scale) pore geometries, a wide range of pore diameters, and varying degrees of interconnectivity (Anderson et al., 2009).

### Specimen Preparation and Experimental Setup

The TSC and TLC were collected from 0 to 3 m below the natural ground surface in Toronto, Canada. The two soils were air dried for 2 wk and then ground and passed through a 2-mm sieve. A standard Proctor compaction test (ASTM, 2012) was conducted on the two soils, whose optimum moisture contents were determined as 13.5 and 12.3%, respectively. The basic physical properties of TSC and TLC are summarized in Table 1. Figure 2 presents their gradation curves.

### Soil-Freezing Characteristic Curve Measurement

Compacted specimens were saturated and used for measuring the SFCC. Pre-calculated amounts of dry soil and tap water were mixed and statically compacted at the optimum moisture content to achieve the maximum dry density by volume control method. The soil specimen was compacted in five equal layers. Each layer was 20 mm high, and the final soil specimen was 100 mm in height with a diameter of 50 mm. After compaction, the specimen was securely wrapped with filter paper. A cling wrap with small holes was used to cover the filter-paper-wrapped specimen. The specimen was then put into plastic tube (which was drilled with small holes and was used to constrain the lateral expansion of the soil specimen) and was submerged in a plastic tank with tap water for saturation. On top of the specimen, a mass of 2 kg was placed to limit its vertical expansion, as shown in Fig. 3. The specimen imbibed water gradually along its height and achieved a constant mass after a period of 1 wk. At this time, the soil specimens were close to fully saturated condition (i.e., 100%). In spite of careful experimental procedures that were followed to prevent specimen expansion during the saturation process, the soil specimen expanded slightly (for TLC, the changes in height and diameter were approximately 0.8 and 1%, respectively; for TSC, the changes were insignificant).

The EC-5 moisture sensor, RT-1 temperature sensor, and EM50 data logger (all manufactured by the METER Group) were used for measuring SFCC of the two soils. The EC-5 can determine volumetric water content by measuring dielectric constant of the soil using FDR technique. The 70-MHz frequency minimizes salinity and textural effects, making this a reliable sensor for use (with an accuracy better than 0.03 m$^3$ m$^{-3}$ in typical soils). The RT-1 is an easy-to-use instrument for reliably measuring soil temperature and has a temperature resolution of 0.1°C. The dimensions of the two sensors are shown in Fig. 4a. The two sensors were calibrated prior to their use, as summarized below.

The RT-1 was inserted into one end of the specimen, and the EC-5 was inserted into the other end (with its two prongs completely in the specimen). A small amount of plasticine was pasted around the overmolding of the EC-5 (see Fig. 4b). This was aimed to prevent the direct contact, if any, between the EC-5 prongs and air, which may influence the EC-5 reading. The two sensors were then connected to the EM50 data logger. The time intervals for data collection were typically 5 and 15 min. The soil specimen was tightly sealed by cling wrap to prevent moisture loss during the testing period. Three-dimensional freezing of the specimen (under zero external stress) was fulfilled by putting it in a freezer, in which the ambient temperature was controlled. In the present study, 14 specimens were tested, of which seven were TSC (labeled as TSC-1 to TSC-7) and the remainder seven were TLC (labeled as TLC-1 to TLC-7). All these specimens were saturated except for TSC-7, which was kept at its initial optimum moisture content. Table 2 summarizes the imposed testing conditions for these specimens. For achieving uniform temperature condition, the soil specimens were kept for 12 h under each controlled temperature. After 12 h, the controlled temperature was changed to next value, following the sequence shown in Table 2.

![Fig. 2. Particle size distribution of Toronto silty clay (TSC) and Toronto lean clay (TLC).](image)

| Table 1. Basic physical properties of Toronto silty clay (TSC) and Toronto lean clay (TLC). |

<table>
<thead>
<tr>
<th>Soil</th>
<th>Property†</th>
<th>$w_L$</th>
<th>$w_p$</th>
<th>PI</th>
<th>$w_{opt}$</th>
<th>$\rho_{max}$</th>
<th>$G_s$</th>
<th>Sand</th>
<th>Silt</th>
<th>Clay</th>
<th>USCS‡</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>†L, liquid limit; $w_p$, plastic limit; PI, plasticity index; $w_{opt}$, optimum moisture content; $\rho_{max}$, maximum dry density; $G_s$, specific gravity; USCS, Unified Soil Classification System (ASTM, 2011).</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>TSC</td>
<td>19.6</td>
<td>13.6</td>
<td>6</td>
<td>13.5</td>
<td>1915</td>
<td>2.68</td>
<td>3</td>
<td>81</td>
<td>16</td>
<td>CL-ML</td>
<td></td>
</tr>
<tr>
<td>TLC</td>
<td>25</td>
<td>13</td>
<td>12</td>
<td>12.3</td>
<td>1962</td>
<td>2.69</td>
<td>31</td>
<td>50</td>
<td>19</td>
<td>CL</td>
<td></td>
</tr>
</tbody>
</table>

† CL, lean clay; ML, silt.
Two freezing methods (i.e., direct freezing and step freezing) were used to freeze the specimens, and the specimens were then gradually thawed under different temperatures for determining SFCC. For example, specimens TSC-1 to TSC-3 (initially at room temperature, which was around 23°C) were directly exposed to −20°C. The controlled temperature was then gradually increased until it again reached room temperature. The thawing branch of SFCC was measured using this technique. For specimens TSC-4 and TSC-5, the controlled temperature was gradually decreased from room temperature to −20°C and was then increased back to room temperature. In other words, both the freezing and thawing branches of SFCC were obtained. Specimens TSC-6 and TSC-7 were only subjected to step decreasing temperatures, facilitating the measurement of the freezing branch of the SFCC.

Sensor Calibration

For calibrating the EC-5 moisture sensor, four TLC cylindrical specimens were compacted at its optimum moisture content (i.e., 12.3%), with dimensions of 100 mm in length and 50 mm in diameter. After compaction, one specimen was dried and the other three were wetted by covering them with dry and wet filter paper, respectively. The final gravimetric water contents of the four specimens were determined by oven dry method as 14.68, 13.87, 13.01, and 11.96%, respectively. The corresponding volumetric water contents were calculated as 0.280, 0.266, 0.252, and 0.234 m³ m⁻³, respectively, on the basis of the measured dry soil mass and volume of the specimens (i.e., volumetric water content = gravimetric water content × dry soil mass/volume).

The volumetric water contents of the four TLC specimens at room temperature, 15, 10, 6, and 2°C were measured by the EC-5. During this process, the water contents of the four specimens were kept unchanged by tightly sealing them with cling wrap and storing them in plastic container.

From the results summarized in Fig. 5a, it can be observed that under constant water content condition, the measured volumetric water content by EC-5 decreases against the controlled temperature. This suggests that above 0°C, temperature change influences the EC-5 reading. The relationship between the calculated volumetric water content and the measured value for the soil specimen under each controlled temperature can be derived from these results. In the present study, a linear relationship (i.e., calculated volumetric water content = A × measured volumetric water content + B) was used to best fit the data points. The constants A and B and the coefficient of determination (R²) for the five controlled temperatures are summarized in Table 3. For SFCC measurement in the present study, when the temperature of soil specimen was above 0°C, the volumetric water content measured by EC-5 (i.e., total water content) was calibrated by using these relationships. For example, if the soil specimen achieved equilibrium under room temperature (or 2°C), the linear relationship obtained for room temperature (or 2°C) was used to calibrate the EC-5 measured volumetric water content.

On the other hand, when the soil specimen was under subzero temperatures (i.e., frozen condition), no direct calibration was performed due to the lack of reliable equipment for quantifying unfrozen water and ice contents in the soil specimen. However, Smith and Tice (1988) conducted unfrozen water content tests on various types of soils by using TDR and NMR. A general relationship was found between the measured apparent dielectric constant (K_a) by TDR and the measured unfrozen water content by NMR for these soils. This relationship (Eq. [1]) was used in the present study for obtaining the volumetric unfrozen water content (θ_u) under subzero temperatures based on K_a.
of the soil specimen, which can be determined from EC-5 raw output according to the manufacturer:

\[
\theta_a = -1.458 \times 10^{-1} + 3.868 \times 10^{-2} K_s - 8.502 \times 10^{-4} K_s^2 + 9.920 \times 10^{-6} K_s^3 
\]

The calibration of the RT-1 temperature sensor was done by comparing the measured values with two thermometers. A couple of different temperature values were selected and the results are shown in Fig. 5b. It can be seen that the measured temperature values by RT-1 are close to the values measured by the two thermometers (data points are closely located along the 1:1 line), suggesting that RT-1 provided good accuracy.

### Soil-Water Characteristic Curve Measurement

Three different methods, namely, the pressure plate method (ASTM, 2016), vapor equilibrium method (Fredlund et al., 2012), and WP4-T measurement (ASTM, 2016), were used for determining the SWCC of the two soils. For the first two methods, saturated compacted specimens were used. Similar procedure that was used for preparing SFCC specimens (i.e., compaction and saturation) was followed for preparing the required specimens. The dimension of the specimen used for the pressure plate method was 63 mm in diameter and 20 mm in height. A small piece of the saturated specimen was cut and used for the vapor equilibrium method. For the WP4-T measurement, certain amounts of dry soil and tap water were mixed to prepare wet soil aggregates with different initial water contents. The wet soil aggregates (with sizes typically <2 mm) were stored in plastic bags for 2 d to achieve uniform moisture distribution, which were used for measurement.

The ceramic disk with an air-entry value of 500 kPa was used in the pressure plate extractor (Soilmoisture Equipment Corporation). The air pressures ranging from 10 to 300 kPa were applied while the pore water pressure was kept at the atmospheric pressure for gathering SWCC data. Each applied air pressure was maintained at least for 7 d for achieving equilibrium condition. For the vapor equilibrium method, three different solutions were used: CuSO\(_4\)\(\times\)5H\(_2\)O, K\(_2\)SO\(_4\), and NaCl, which approximately correspond to the total suction values of 1.9, 4.4, and 38 MPa, respectively. The saturated specimen was kept in desiccator for 90 d, ensuring the soil pore water equilibrated with the solution vapor. The WP4-T device (METER Group) was calibrated by using the standard KCl solution provided by the manufacturer. The suction measurement for the wet soil aggregates with different water contents was performed at the temperature set in the WP4-T chamber, which was 25°C. The gravimetric water content was determined from these three methods using oven dry method. The osmotic suction of the soil specimens (i.e., 6 kPa, calculated by using the van’t Hoff equation [Fredlund et al., 2012]) was assumed negligible, due to the low solute concentrations in the tap water (Jiang et al., 2017).

### Experimental Results

#### Temperature–Time Curves of the Two Soils

Figure 6a shows the temperature–time curves of TSC-1, TSC-2, and TSC-3, under the direct freezing and step thawing conditions.

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<table>
<thead>
<tr>
<th>No.</th>
<th>Controlled temperatures</th>
<th>Freezing test used</th>
</tr>
</thead>
<tbody>
<tr>
<td>TSC-1</td>
<td>RT, −20.0, −15.0, −10.0, −7.0, −5.0, −3.5, −2.5, −2.0, −1.5, −1.0, −0.5, 0.0, RT</td>
<td>Direct freezing, step thawing</td>
</tr>
<tr>
<td>TSC-2</td>
<td>RT, −20.0, −16.0, −12.0, −9.0, −7.0, −5.0, −4.0, −3.0, −2.0, −1.0, 0.0, RT</td>
<td>Direct freezing, step thawing</td>
</tr>
<tr>
<td>TSC-3</td>
<td>Same as for TSC-2</td>
<td>Direct freezing, step thawing</td>
</tr>
<tr>
<td>TSC-4</td>
<td>RT, −0.1, −0.5, −1.0, −1.5, −2.0, −2.5, −3.0, −3.5, −4.0, −5.0, −6.0, −10.0, −15.0, −20.0, −15.0, −10.0, −6.0, −4.0, −3.0, −1.0, 0.0, RT</td>
<td>Step freezing, step thawing</td>
</tr>
<tr>
<td>TSC-5</td>
<td>Same as for TSC-4</td>
<td>Step freezing, step thawing</td>
</tr>
<tr>
<td>TSC-6</td>
<td>RT, −0.5, −0.8, −1.0, −1.5, −2.0, −2.5, −3.0, −3.5, −4.0, −5.0, −7.0, −10.0, −15.0, −20.0</td>
<td>Step freezing</td>
</tr>
<tr>
<td>TSC-7</td>
<td>Same as for TSC-6</td>
<td>Step freezing</td>
</tr>
<tr>
<td>TLC-1</td>
<td>Same as for TSC-1</td>
<td>Direct freezing, step thawing</td>
</tr>
<tr>
<td>TLC-2</td>
<td>Same as for TSC-2</td>
<td>Direct freezing, step thawing</td>
</tr>
<tr>
<td>TLC-3</td>
<td>Same as for TSC-2</td>
<td>Direct freezing, step thawing</td>
</tr>
<tr>
<td>TLC-4</td>
<td>RT, −0.5, −0.8, −1.0, −1.5, −2.0, −2.5, −3.0, −3.5, −4.0, −5.0, −7.0, −10.0, −15.0, −20.0, −15.0, −10.0, −7.0, −5.0, −3.5, −2.5, −2.0, −1.5, −1.0, −0.5, 0.0, RT</td>
<td>Step freezing, step thawing</td>
</tr>
<tr>
<td>TLC-5</td>
<td>Same as for TLC-4</td>
<td>Step freezing, step thawing</td>
</tr>
<tr>
<td>TLC-6</td>
<td>RT, −0.5, −1.0, −1.5, −2.0, −2.5, −3.0, −3.5, −4.0, −4.5, −5.0, −7.0, −10.0, −15.0, −20.0, −15.0, −10.0, −7.0, −5.0, −4.5, −4.0, −3.5, −3.0, −2.5, −2.0, −1.5, −1.0, −0.5, RT</td>
<td>Step freezing, step thawing</td>
</tr>
<tr>
<td>TLC-7</td>
<td>Same as for TLC-6</td>
<td>Step freezing, step thawing</td>
</tr>
</tbody>
</table>

† RT, room temperature, around 23°C.
condition. There is a distinct supercooling phenomenon, as can be seen from the inset of Fig. 6a. However, the phase change part is not well defined because only a certain amount of pore water can transform into ice at the freezing temperature of the soil specimen. The corresponding latent heat released therefore could not fully counteract the cooling effect exerted by the controlled temperature, which is low (i.e., −20°C). The temperature–time curves of TLC-1, TLC-2, and TLC-3 under the direct freezing and step thawing condition are similar to those of the TSC specimens. For the direct freezing and step thawing condition, the volumetric unfrozen water content of soil specimen under each thawing temperature was measured. As a result, the thawing branch of SFCC was obtained.

Figure 6b shows the temperature–time curves of specimens TSC-4 to TSC-7 under the step freezing–step thawing condition. For TSC-4 and TSC-5, their temperature–time curves appear to be the same (due to the same controlled temperatures), except for the different supercooling temperatures and the time when ice starts nucleation. Similarly, TSC-6 and TSC-7 have approximately the same temperature–time curve, in spite of the latter being at the optimum moisture rather than the saturated condition. However, it should be noted that TSC-7 has lower supercooling and freezing temperatures than TSC-6. In addition, the temperature increment of TSC-7 is lower than that of TSC-6. This is reasonable, since initially more water existed in the soil pores of the saturated specimen and therefore larger amount of latent heat was generated and contributed to the temperature rise within the soil specimen. Similar conclusions can be drawn for the four saturated TLC specimens (i.e., TLC-4 to TLC-7) under the step freezing and thawing condition.

Table 3. EC-5 calibration constants A and B.

<table>
<thead>
<tr>
<th>Temperature</th>
<th>A</th>
<th>B</th>
<th>R²</th>
</tr>
</thead>
<tbody>
<tr>
<td>°C</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Room temperature</td>
<td>0.5200</td>
<td>0.1134</td>
<td>0.99</td>
</tr>
<tr>
<td>15</td>
<td>0.5158</td>
<td>0.1331</td>
<td>1.00</td>
</tr>
<tr>
<td>10</td>
<td>0.5983</td>
<td>0.1217</td>
<td>0.99</td>
</tr>
<tr>
<td>6</td>
<td>0.6347</td>
<td>0.1182</td>
<td>0.95</td>
</tr>
<tr>
<td>2</td>
<td>0.6372</td>
<td>0.1223</td>
<td>0.94</td>
</tr>
</tbody>
</table>
Under the step freezing and thawing condition, both the freezing and thawing branches of SFCC were measured.

In the present study, the freezing temperature of soil specimen was determined based on its temperature–time curve (i.e., the highest temperature after supercooling). Under the direct freezing condition, the soil specimen (initially at room temperature) was directly exposed to $-20^\circ C$. Therefore, the temperature change in the soil specimen was dramatic during the initial period of freezing. Since the time interval for data collection was 15 min for some tests, it was probable that the RT-1 did not capture the actual freezing temperature (the highest point after supercooling) but measured the temperature values that were close to the freezing temperature. On the other hand, the controlled temperature in the step freezing condition was gradually decreased (i.e., temperature change rate was low). There was a relatively well-defined phase change period (the soil specimen maintained at the same temperature for ~1 h). This indicates that the freezing temperature under the step freezing condition can be reliably determined.

The supercooling and freezing temperatures of the specimens under the step freezing condition are summarized in Table 4. The supercooling temperatures of the two soils are scattered due to the random nature of supercooling. The saturated TSC specimens show fluctuation with respect to freezing temperature. This may be attributed to specimen structure change during the saturation process and crack formation when inserting sensors into the specimen, which contribute to differences between specimens. On the other hand, the freezing temperatures of TLC specimens are more consistent with a value around $-0.6^\circ C$ (see Fig. 7). This suggests that better integrity was achieved with TLC during the testing process, and hence its experimental results experienced relatively limited fluctuation.

Results of Soil-Freezing Characteristic Curve Measurement

The measured SFCC of TSC and TLC are summarized in Fig. 8 and 9. The corresponding open data point is the initial volumetric water content of each specimen calculated based on its gravimetric water content and maximum dry density before testing. Figures 8a and 9a show the thawing branch of SFCC of the two soils, respectively. It can be seen that the unfrozen water content changes rapidly in the temperature range from 0 to $-5^\circ C$, after which the unfrozen water content gradually levels off. The rapid change in unfrozen water content is attributed to the phase change of capillary and adsorbed water in relatively large pores. With a further decrease in temperature, most of the pore water is frozen, and the strongly adsorbed water and the water confined in small pores (which have low matric potential) gradually transform into ice. It is also noted that the measured thawing branches of TSC are not consistent, which may be partly attributed to the structure change of TSC specimen, and its poor ability of maintaining water during experimental process. On the other hand, the measured thawing branches of TLC are relatively consistent, especially for the low temperature range. This is reasonable, since the TLC specimen was typically intact and did not crack during the testing period.

The freezing and thawing branches of SFCC of the two soils are shown in Fig. 8c and 9b, respectively. It can be seen that the unfrozen water content is relatively stable (i.e., around the measured initial water content, since no ice was formed) until the supercooling temperature is achieved. As ice crystals start to nucleate and continue to grow in soil pores, the quantity of unfrozen water drops dramatically. Finally, when most capillary and adsorbed water turns into ice, unfrozen water content decreases with a gentle slope and gradually levels off. On the contrary, the thawing branch of SFCC is smooth and does not show abrupt change in unfrozen water content or superheating phenomenon (Tan et al., 2015). Therefore, significant hysteresis is observed between the freezing and thawing branches. For the two soils under the present experimental conditions, hysteresis occurs mainly in the temperature range from 0 to $-5^\circ C$. After $-5^\circ C$, hysteresis is insignificant.

Figure 8b shows the freezing branch of SFCC of TSC. This is similar to the freezing branch shown in Fig. 8c. It is interesting to note that the freezing branch shown in Fig. 8c. It is interesting to note that the freezing branch of SFCC is supposed to

<table>
<thead>
<tr>
<th>Soil†</th>
<th>Supercooling temperature</th>
<th>Freezing temperature</th>
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<tbody>
<tr>
<td>TSC-4</td>
<td>-3.8</td>
<td>-1.2</td>
</tr>
<tr>
<td>TSC-5</td>
<td>-4.1</td>
<td>-1.0</td>
</tr>
<tr>
<td>TSC-6</td>
<td>-3.9</td>
<td>-0.5</td>
</tr>
<tr>
<td>TSC-7‡</td>
<td>-4.8</td>
<td>-2.0</td>
</tr>
<tr>
<td>TLC-4</td>
<td>-3.2</td>
<td>-0.6</td>
</tr>
<tr>
<td>TLC-5</td>
<td>-3.1</td>
<td>-0.6</td>
</tr>
<tr>
<td>TLC-6</td>
<td>-3.6</td>
<td>-0.6</td>
</tr>
<tr>
<td>TLC-7</td>
<td>-3.6</td>
<td>-0.5</td>
</tr>
</tbody>
</table>

† TSC, Toronto silty clay; TLC, Toronto lean clay.
‡ TSC-7 was at the optimum moisture content.
behave as the line shown with dashes in Fig. 8b. It means that soil specimen temperature would change to its freezing temperature (i.e., ice-entry value [IEV], at which ice first begins to form in the largest soil pores) from the supercooling temperature due to the release of latent heat. The freezing temperature is theoretically maintained for a short period while unfrozen water content plunges. Afterward, unfrozen water content gradually decreases with the decrease in soil temperature. However, in reality, as ice crystals form in large soil pores, the amount of latent heat released is small and not able to maintain soil temperature at the freezing temperature (for a period that is long enough to ensure an equilibrium condition that facilitates reliable measurement), since the ambient temperature inside freezer at this time is relatively low (i.e., around the supercooling temperature). Instead, the soil temperature keeps decreasing until an equilibrium condition is achieved under the current controlled temperature (e.g., see Fig. 7). As a result, the unfrozen water content during this period could not be reasonably measured, as equilibrium condition is not fully established. In other words, the theoretical freezing branch shown in Fig. 8b is not measurable in the present study.

Results of Soil-Water Characteristic Curve Measurement and Comparison with the Soil-Freezing Characteristic Curve

The SWCCs of the two soils are shown in Fig. 10. It can be seen that the data points measured by the three methods are consistent and form a smooth curve (especially for TLC) in spite of some scatter in the data. This suggests that the measured SWCC
is reliable. Comparisons between SWCC and the three thawing branches of SFCC of the two soils (i.e., TSC-1 to TSC-3, and TLC-1 to TLC-3) are shown in Fig. 11. The gravimetric water content obtained in SWCC measurement is converted to volumetric water content by multiplying by the maximum dry density of each soil shown in Table 1 (assuming that the volume of specimens was unchanged during SWCC measurement). The measured subzero temperature is converted to cryogenic suction by multiplying by 1225 kPa °C⁻¹, as suggested by Ren and Vanapalli (2018). It can be observed from Fig. 11 that the measured SWCC does not agree well with the SFCC. The suction value obtained from SWCC is lower than that obtained from SFCC at the same unfrozen water content. The disagreement cannot be compensated by multiplying the cryogenic suction by an adjustment factor of 2.2, because it introduces more discrepancy to the two retention curves. In other words, there is only qualitative but no quantitative similarity between SWCC and SFCC for the two soils under the investigated experimental conditions. The discrepancy can be partly attributed to the slightly different initial water contents of the specimens used for SWCC and SFCC measurements, and to possible errors associated with the sensors.

The measured SWCC and SFCC were fitted using the Fredlund and Xing (1994) equation. The fitted curves are shown in Fig. 11 and the fitting parameters (i.e., $a_f$, $n_f$, $m_f$, and $y_r$) are summarized in Table 5. It can be seen that the Fredlund and Xing (1994) equation provides good fitting for both the SWCC and SFCC of the two soils. However, the fitting parameters for the two retention curves of the same soil are different, especially with respect to the slope of the retention curves (controlled by parameter $n_f$) and the residual suction value ($y_r$).

![Fig. 10. Measured soil-water characteristic curve (SWCC) of (a) Toronto silty clay (TSC) and (b) Toronto lean clay (TLC).](image1)

![Fig. 11. Comparison between the thawing branch of the soil-freezing characteristic curve (SFCC) and soil-water characteristic curve (SWCC) of (a) Toronto silty clay (TSC) and (b) Toronto lean clay (TLC).](image2)

<table>
<thead>
<tr>
<th>Soil</th>
<th>Curve</th>
<th>Parameter</th>
<th>$a_f$</th>
<th>$n_f$</th>
<th>$m_f$</th>
<th>$y_r$</th>
<th>$R^2$</th>
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</thead>
<tbody>
<tr>
<td>TSC</td>
<td>SWCC</td>
<td>665</td>
<td>0.81</td>
<td>1.41</td>
<td>904</td>
<td>0.96</td>
<td></td>
</tr>
<tr>
<td></td>
<td>SFCC</td>
<td>895</td>
<td>11.91</td>
<td>0.27</td>
<td>1447</td>
<td>0.92</td>
<td></td>
</tr>
<tr>
<td>TLC</td>
<td>SWCC</td>
<td>1217</td>
<td>0.59</td>
<td>1.67</td>
<td>2718</td>
<td>0.99</td>
<td></td>
</tr>
<tr>
<td></td>
<td>SFCC</td>
<td>876</td>
<td>3.34</td>
<td>0.45</td>
<td>5189</td>
<td>0.95</td>
<td></td>
</tr>
</tbody>
</table>

† $y_r$, residual suction value.
Discussion

Measurement of the Soil-Freezing Characteristic Curve

The reliable measurement of SFCC depends on the accurate determination of temperature and unfrozen water content. The difficulties in accurately measuring temperature are not only associated with the resolution and precision requirement for the temperature sensor (the resolution of RT-1 is 0.1°C, which is considered sufficient for an accurate temperature measurement), but also with the time required to achieve uniform temperature within the bulk soil specimen. If this is not achieved, the measured temperature may only be the temperature of soil surrounding the temperature sensor. Moreover, during the freezing process, once ice has started nucleation, soil temperature changes relatively quickly under the low controlled temperature (see Fig. 6 and 7). Therefore, during this period, the reliable measurement of unfrozen water content is difficult, since an equilibrium condition is not fully established.

A number of concerns must be addressed carefully with respect to the measurement of unfrozen water content. First, temperatures above 0°C were observed to have influence on the EC-5 reading. Hence, the total water content needs to be calibrated taking into account the influence of temperature at which it is measured. In addition, the EC-5 was calibrated by using four TLC specimens at different water contents. The obtained calibration curves were used for both the TLC and TSC specimens. However, specific calibration for TSC is suggested for reliable determination of its SFCC. Second, the measurement of the dielectric constant in frozen soils is influenced not only by water content, but also by other parameters, which include ice content, soil structure, and bulk density that influence the sensor’s performance (Yoshikawa and Overduin, 2005). The water content at which soil specimen is compacted, imparting a particular soil structure, would have significant influence on the measured SFCC. Third, unfrozen water content in high subzero temperature range (e.g., from 0 to −5°C) shows significant change with respect to temperature. For this reason, more precautions are required in this temperature range such that the measured SFCC is reliable. Fourthly, the accuracy of the EC-5 sensor is ~0.03 m³ m⁻³, which may result in considerable difference between the measured and actual unfrozen water content, especially when the soil temperature is low. In addition, the use of plasticine for covering the overmolding of EC-5 should be avoided in future studies, since the plasticine has high water content, which can probably contribute to additional experimental error. Finally, pore water supercooling during the freezing process causes significant hysteresis of SFCC. The hysteresis measured in the present study is also mainly attributed to this reason. It is difficult to obtain representative freezing branch of SFCC, since the degree of supercooling may be influenced by many factors such as the freezing rate, presence and concentration of solutes, and electromagnetic fields exerted by EC-5. For this reason, comparisons were not provided between SWCC and the freezing branch of SFCC in the present study.

Concerns Regarding the Similarity between the Curves

Several studies have shown similarities between SFCC and SWCC for several soils (Koopmans and Miller, 1966; Spaans and Baker, 1996; Liu et al., 2012; Schafer and Beier, 2017). Good agreement was also observed between the measured soil water potential or subzero temperature and the one obtained from the Clapeyron equation (Watanabe et al., 2012; Caicedo, 2017). However, several investigators have also obtained results showing dissimilarity between SFCC and SWCC.

For example, Ma et al. (2015a) measured both the SWCC and SFCC of a silt and a clay. The soil specimens were statically compacted at specified water contents and to targeted dry densities. The unfrozen water content was obtained by using NMR. To avoid the effect of supercooling, only the thawing branch of SFCC was measured. The soil water potential of frozen soil is derived from a generalized Clapeyron equation. The results show that at the same unfrozen water content, the soil water potential of an unsaturated soil is significantly lower than that of its frozen counterpart, which means that there is no similarity between SFCC and SWCC. In the study by Azmatch et al. (2012), the SWCC and SFCC of Devon silt specimens with and without salinity were measured. The specimens prepared from slurry were consolidated under different pressures. The unfrozen water content was measured by using TDR. The results display significant discrepancy between SWCC and SFCC of the specimen with salinity. This suggests that the tap water used in the present study may have contributed to the observed discrepancy between SFCC and SWCC of the two investigated soils due to influence of various ions (i.e., water salinity).

In the present study, the slightly different initial water contents of the specimens used for SWCC and SFCC measurements, and the accuracy of EC-5 may be the major reasons for the dissimilarity between the measured SFCC and SWCC. In other words, the limitations associated with testing sensors prohibit a reliable characterization of the quantitative similarity between the two retention curves. However, besides the experimental limitations, some additional concerns with respect to the similarity between SFCC and SWCC are summarized below.

First, it is important to understand the temperature range within which the similarity between SFCC and SWCC could be achieved. The classic work by Koopmans and Miller (1966) experimentally showed the similarity within a narrow temperature range (i.e., from 0 to −0.2°C). It should also be noted that their results were based on certain assumptions (Miller, 1980), in addition to following careful specimen preparation and experimental procedures. Williams (1976) stated that the free energy of soil unfrozen water, calculated from the Clapeyron equation, is essentially equivalent to the negative pore water pressure before temperatures lower than −1.5°C. This suggests that using the Clapeyron equation to convert subzero temperature to cryogenic suction and adopting the latter for the comparison of SFCC and SWCC is applicable, in the temperature range between 0°C and about −1.5°C. Brown
and Payne (1990) also concluded that in the absence of solutes, the Schofield (1935) equation (which is a simplified form of the Clapeyron equation) relating cryogenic suction to subzero temperature can be used at least down to \(-0.73\,^\circ\text{C}\) and almost certainly to several degrees lower without serious error. It has also been shown that in a fine glass capillary tube at temperatures near \(0\,^\circ\text{C}\), the ice–water interface has a hemispherical appearance with zero contact angle at the walls (Skapski et al., 1957). This is the same as the air–water interface in a capillary tube (Miller, 1980). Ma et al. (2015b) pointed out that the Clapeyron equation is valid for phase equilibrium state where pressure and temperature remain constant with respect to time. Sufficiently slow freezing or thawing rate is required to ensure an equilibrium condition. Freezing or thawing at a relatively fast rate will lead to significant discrepancy between SFCC and SWCC (Liu et al., 2012). In addition, fast freezing may cause ice crystals to form in smaller pores first (Colbeck, 1982), rather than ice filling larger pores first in frozen soils as is typically assumed (Williams, 1999).

Second, it is difficult to precisely control temperature around \(0\,^\circ\text{C}\) from typical experimental studies. For this reason, there are difficulties associated with the reliable SFCC measurements at high subzero temperatures (i.e., small suction range). In addition, the determination of SWCC becomes increasingly inaccurate as soil dries (i.e., large suction range). Therefore, the comparison between SFCC and SWCC in these two ranges is questionable. For example, coarse-grained soils generally have an air-entry value of a few kilopascals. According to the Clapeyron equation (e.g., using a value of 1225 kPa \(^\circ\text{C}^{-1}\) for conversion), a suction value of 8 kPa corresponds to a temperature value around \(-0.007^\circ\text{C}\), which is difficult to be accurately measured or controlled. This causes difficulty in comparing the air-entry value and ice-entry value.

Third, Black and Tice (1989) showed that the SFCC and SWCC data of Windsor (mixed, mesic Typic Udipsamments) sandy loam could be best fitted by one single equation. They achieved better results when the soil specimens used for SFCC and SWCC measurement had approximately the same densities. However, worse results were obtained when there was a difference between the densities. It was also suggested that SFCC of frozen saturated soil and SWCC of unfrozen unsaturated soil are comparable only for the same soil at the same bulk density and with similar histories. In other words, theoretically, only freezing and drying curves or thawing and wetting curves can be compared. In addition, the soil must meet similar structure requirements as proposed by Miller and Miller (1956). Konrad (1990) concluded that unfrozen water content of a given soil is strongly dependent on the soil structure. This is similar to the influence of soil structure on the SWCC (Vanapalli et al., 1999). Therefore, the difference between the structures of the SWCC and SFCC specimens in the present study (due to slight variation in compaction water content and uncontrollable structure changes during the saturation process) is possibly another important factor that contributed to the dissimilarity.

Finally, some fundamental differences may also contribute to the dissimilarity between the SFCC and SWCC. Williams (1967a) showed that there are at least three distinct processes by which air may replace water in soil pores: (i) a process explained by normal capillary equation for air–water interface, (ii) a process involving diffusion of external air to entrapped air bubbles, which are then slowly enlarged and tend to penetrate smaller pores, and (iii) a process of slow advance of air–water interface into the soil. Additionally, the nucleation of air bubbles was not experimentally observed in his study (Williams, 1967a). However, spontaneous ice nucleation and the associated replacement of water by ice may occur at anywhere inside soil pores, if the freezing condition is favorable (e.g., at sufficiently low temperature for a long freezing time). The ice may then percolate further and extrude into adjacent pores. No “ice diffusion” process corresponding to the second process of the replacement of water by air (i.e., air diffusion) is expected. Rempel (2012) pointed out that the strengths and nature of the dominant wetting interactions that cause liquid films to coat soil particles in the unsaturated zone are different from those that cause premelted films to separate soil particles from pore ice (i.e., the former involve intermolecular forces between particles, liquid, and vapor, whereas the latter involve intermolecular forces between particles, liquid, and ice). The existence of solutes further complicates the problem.

From a theoretical point of view, there is similarity between the SWCC of unfrozen unsaturated soil and the SFCC of the same soil at frozen saturated condition. However, researchers also showed that the similarity between SWCC and SFCC of variably saturated soils. For example, Zhou et al. (2014) determined the SWCC on an initially saturated specimen, whereas the unfrozen water contents (i.e., SFCC) were measured at different depths of an initially unsaturated soil column subjected to unidirectional freezing with water migration and varying total water content. Except for some dissimilarities when soil temperature was around \(0^\circ\text{C}\) (e.g., higher than \(-0.2^\circ\text{C}\), the results showed that there was good agreement between the SWCC and SFCC of variably saturated soils. These investigators suggested that the dissimilarity was partly because unfrozen water content is dependent on both temperature and total water content in the high subzero temperature range. An adjustment factor with value of 1.0 was obtained for the unsaturated soil used in their study. However, extensive experimental investigations are required for better understanding the similarities and differences between SWCC and SFCC of variably saturated soils.

\textbf{Summary}

The SFCC and SWCC are physically similar, since both of these two retention curves describe the relationship between the amount of pore water and its energy state in a soil. Some of the possible reasons for the hysteresis of SFCC proposed in the literature (e.g., the effects of pore blocking and contact angle, and the change in pore structure) are based on their similarity. In the present study, the SFCC and SWCC of two Canadian soils (i.e., TSC and TLC) were experimentally determined and analyzed.
Many factors have a significant influence on the reliable measurement of SFCC, including the sensors’ resolution and their stability, specific sensor calibration for each soil under investigation, thermodynamic equilibrium condition, and continuous soil structure changes that arise during freezing–thawing processes. The observed hysteresis of SFCC for the two Canadian soils is mainly attributed to the supercooling of pore water.

The measured SFCC and SWCC of the two soils show significant differences, indicating their quantitative dissimilarity. Many factors may contribute to the dissimilarity—for example, variations in the structure of specimens during the compaction and saturation processes, and during freezing–thawing processes, and crack formation when sensors were inserted into the specimens. The performance and calibration of sensors would also significantly influence the comparison between SFCC and SWCC.

In other words, the limitations associated with testing sensors prohibit reliable characterization of the quantitative similarity between the two retention curves.

In addition, some fundamental differences may exist between drying–wetting and freezing–thawing processes. For example, the way that ice replaces water may be different from the way that air replaces water in a soil (Williams, 1967b). The dominant wetting interactions that cause liquid films to coat soil particles in the unsaturated zone are different from those that cause premelted films to separate soil particles from pore ice. The existence of salinity further complicates the problem. The present study highlights that rigorous investigations are required for better understanding the fundamental behavior of SFCC of fine-grained soils.

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References


