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# Solute and water effects on soil freezing characteristics based on laboratory experiments

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### ABSTRACT

Laboratory experiments were conducted to study effects of water and solute on soil freezing using TDR and temperature sensor combination methods. ANOVA methods were applied for analyzing significance for solute influences on soil freezing characteristic curve (SFCC). Results showed that higher initial water content influenced the SFCC by increasing liquid water content at the same temperature due to more water connection with soil pores, and adsorbed by soil particles. ANOVA results showed solute content and solute type all had significant effects (P < 0.001 to P < 0.5) on soil freezing processes. And solute in soil resulted in a lower freezing point of soil, which made more liquid water co-exist with ice at negative temperatures. And solute concentration condensing due to liquid water decline would also impede soil freezing processes by decreasing osmotic potential. Due to the physical and chemical process of soil solution, different ions also presented some differences in SFCC parameter estimation. Based on a trial and error method, a prediction model was also built, and it behaved well in predicting SFCC under different water and solute conditions.

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#### 1. Introduction

Knowledge of soil freezing is of great importance for understanding and modeling the transport of water, heat, and solutes in frozen soils. Water movement in frozen soils plays a significant role in agricultural and engineering activities in cold regions, e.g., in predicting frost heave (Konrad and Morgenstern, 1980), in investigating water and solute transport and redistributions in cold periods (Baker and Spaans, 1997), as well as in waste disposal using artificial ground freezing (Mccauley et al., 2002).

As soil temperature decreases below the freezing point, phase transition will occur and ice forms. Due to absorption of soil particles, crevices between particles, and pores with sufficiently small diameter, some part of water remains unfrozen (Spaans and Baker, 1996). Liquid water can coexist with ice in frozen soils at very low temperature. The relationship between liquid water content and soil temperature below zero is called soil freezing characteristic curve (SFCC).

To obtain soil freezing characteristic, the detection of liquid water under negative temperatures is of necessity. Measurements of the unfrozen water contents in frozen soils have been made by adiabatic calorimetry (Kolaian and Low, 1963), dilatometry (Pusch, 1979), isothermal calorimetry (Anderson and Tice, 1971, 1973), differential scanning calorimetry (DSC) (Kozlowski, 2003a,b, 2004), nuclear magnetic resonance (Tice et al., 1982; Kuyala, 1989), X-ray diffraction

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(Anderson and Hoekstra, 1965) and many other methods (Kozlowski, 1995). All these methods have their limitations in that they could only measure disturbed soil samples or saturated samples, and some are resource-intensive.

Time domain reflectometry (TDR) as a rapid and effective method, is applied widely for in situ measurement of liquid water content continuously at multiple depths (Patterson and Smith, 1980; Topp et al., 1980). Spaans and Baker (1996) combined the TDR probes with multiple temperature sensors to detect seasonal soil water dynamics, and obtained good results. Iwata et al. (2010) monitored water movement during snowmelt infiltration with TDR at multiple depths, and concluded that TDR provided a perfect role in detecting water dynamics in frozen soils.

However, all these experiments were based on non-saline soils, or neglected the influences of solutes in soils. When soil was frozen, soil solution was condensed due to decrease in liquid water content and exclusion of solute from ice (assuming that ice crystal is pure without solutes). The increase in solute concentration would affect soil freezing. The presence of solute in soils would decrease freezing point of soil and eventually induce frost heave (Banin and Anderson, 1974) and the initial water content in soil could also cause differences in SFCC. Kozlowski (2004, 2009a, 2009b) found that the freezing point of soil was influenced by water content, and a power function was obtained by the DSC method to describe the change of freezing point with total water content and plastic limit of soil. Bing and Ma (2011) studied the influences of water content and solute on freezing point of soil, and concluded that the freezing point of soil was raised with increase in initial







water content and declined as solute content increased. Also, Bing and Ma (2011) found that different cations and anions presented different characteristics in soil freezing. However, these studies did not obtain a complete curve for SFCC, and had little discussion on the water and solute influences on the freezing process of soil. Azmatch et al. (2012) obtained the SFCC by TDR combining temperature sensor method and compared the SFCCs between saline soil and non-saline soil, concluding that SFCC for saline soil was relatively different from that of non-saline soil. They also suggested that the hydraulic parameters for saline frozen soils should be carefully studied. However, less study focused on the solute type impact on soil freezing.

This study focuses on the properties of soil freezing characteristic curves under different water and solute conditions and the development of a method for soil freezing characteristic curve prediction for saline, frozen soils.

#### 2. Material and methods

#### 2.1. Soil sample preparation

Soil sample was from agricultural field in Yonglian Experimental Site (40°15′N, 108°37′E), the site is located in Wuyuan County, Inner-Mongolia, China. Soil was dug from a  $2 \times 10^5$  hm<sup>2</sup> agricultural field at the depths of 0 to 40 cm in experimental site. Soil texture was defined as silt loam from USDA soil texture triangle according to sand, silt and clay percentage by sedimentation approach, and the basic characteristics of soil are shown in Table 1. The field is used to plant sunflower and corn from May to October every year, and the soil texture and crops are typical in Hetao Irrigation District. Soil was air-dried and diluted by using deionized water to salt mass content below 0.1%, then sieved by 2-mm sieve. Soil sample was then put into plastic bags and stored in an incubator with temperature of 5 °C for experiments.

#### 2.2. Laboratory soil freezing experiment

TDR measures the dielectric coefficient of soil medium, and converts it to volumetric water content using empirical equation calibrated in advance. Solute in soil changes the electrical conductivity of soil water solution, which will influence the dielectric constant of soil, and cause deviations in measuring water content. In this study, traditional TDR probes were adapted by painting 1-mm ethoxyline around the middle probe of 3-probe TDR to eliminate the influences of solute, as shown in Fig. 2.

The calibration of adapted TDR was conducted in the laboratory. Soil sample was collected using a brass cylinder with diameter of 3.5 cm and height of 5.0 cm. The brass cylinder was placed inside a plastic tube with diameter of 5.0 cm and height of 10.0 cm. Then the tube was sealed and located in a bottle filled with the ice crystals of the 2.0 mol/L sodium chloride at -7.6 °C (Fig. 1). Another bottle was filled with mixture solution of ice and water at temperature zero. A thermocouple was used to measure temperature difference between soil sample and the ice and water mixture. One end of the thermocouple was put inside the mixture solution of ice and water, and another end of the thermocouple was put inside the soil sample. The two bottles was located in a cryostat (a

Tuble 1			
Soil hydraulic	parameters	and	texture.

Table 1

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	Texture <sup>a</sup>	Particle distribution				Hydraulic parameters <sup>b</sup>				
		Clay	Silt	Sand	α	n	$\theta_r$	$\theta_s$	l	Ks
		%			1/m	-	cm <sup>3</sup> /cm <sup>3</sup>		-	$\times 10^{-6}\text{m/s}$
	Silt loam	16.22	60.84	22.94	0.44	1.71	0.07	0.44	0.5	6.25

<sup>a</sup> Soil texture determined using USDA's texture triangle.

<sup>b</sup> Estimated by RETC.

Thermocouple V Voltmeter 200 Ice crystals 00 00 10 g D Ð Mixture of water 00 ō bD οD -0 and ice at 0 0 00 D temperature zero O D Ð 00 Ð D Soil sample D 0 Ð 00 D D D D Ð D Dry sand ſ bο ΠD

Fig. 1. TDR calibration setup.

device to maintain low temperature). A digital voltage meter with range of 0 to 20  $\mu$ V was used to measure the voltage caused by the temperature difference in the two ends of the thermocouple. Then the temperature was obtained using the calibrated standard curve of temperature and voltage. Simultaneously, adapted TDR was used to measure liquid water content at the same temperature of soil sample. Calibration results showed good correlation ( $R^2 = 0.9998$ ), indicating that the adapted TDR could be used in determining unfrozen water content in frozen saline soil.

Soil freezing experiment considers the effects of different solute and salinity levels. Five kinds of solute (NaCl, KCl, CaCl<sub>2</sub>, MgSO<sub>4</sub>, and MgCl<sub>2</sub>) combining five solute content levels (with 0.1%, 0.3%, 0.5%, 0.7%, and 1.0% g solute contained in per g of dry soil) were set in determining SFCC. All tests have an initial volumetric water content of 30.0%, and solute was mixed with deionized water to obtain the set water and salinity level. Besides, soil freezing experiment for different initial water content was conducted to study initial water content effects on SFCC. Four initial water content levels were set, i.e., 15%, 20%, 25%, and 30% in volumetric water content, with one repetition for each.

Before starting the freezing test, soil sample was packed into a  $10 \times 10 \times 20$  cm<sup>3</sup> acrylic soil column, with bulk density of 1.50 g/cm<sup>3</sup>. Soil sample was compacted in lifts of 5-cm depth to get uniform bulk density. The packed height of soil sample was 20 cm. TDR probes and temperature sensors were inserted in the middle of soil column and tested after packing. Freezing tests were conducted in a freezing cell (Fig. 2), which could control temperature with accuracy of 0.1 °C. Unfrozen water content and temperature were collected with data-loggers in 4-h intervals, and temperature of freezing cell was stepped from 0 to -25 °C during freezing test. To detect the phase transition around freezing point carefully, temperature interval was set as 1 °C from 0 to -5 °C, then set as 2 °C between -5 and -25 °C. Due to the limited precision of probes and the fast phase transition between 0 and -1 °C, this test procedure might not capture the SFCC nature well between these temperature zones. Thus the obtained data mainly had temperature ranging from -1 to -25 °C.

#### 2.3. SFCC prediction model

The SFCC was often described with a power function as below:

$$\theta_l = a|T|^{-b} \tag{1}$$

where  $\theta_l$  is the liquid water content, cm<sup>3</sup>/cm<sup>3</sup>; *T* is the soil temperature, °C; *a* and *b* are the fitting parameters.

Research had proved that the influences of initial water content and salt content of SFCC had the property of additivity (Xu et al., 1985).

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