

Experimental and numerical investigation of water freezing and thawing in fully saturated sand

Martina Sobotková^{1*}, Alexandr Žák², Michal Beneš², Michal Sněhota¹

¹ Czech Technical University in Prague, Faculty of Civil Engineering, Thákurova 7, 166 29, Prague, Czech Republic.

² Czech Technical University in Prague, Faculty of Nuclear Sciences and Physical Engineering, Břehová 7, 115 19, Prague, Czech Republic.

* Corresponding author. Tel.: +420 224 354 599. Fax: +420 233 337 005. E-mail: martina.sobotkova@fsv.cvut.cz

Abstract: This paper presents an experimental and numerical study of the freezing-thawing behavior of water in fully saturated sand. A relatively inexpensive and easily replicable experimental procedure was developed to simulate the freezing-thawing cycles in a medium-sized sand sample placed in a modified commercial freezer. By insulating the sides and bottom of the sample well, while allowing good thermal conductivity at the top of the sample, a nearly vertical advance of the freezing and thawing front was achieved. A series of freeze-thaw cycles were performed with higher and lower temperature gradients. A numerical multiphysics model, assuming an axially symmetric geometry based on the transient heat transfer during the phase transition, used a parametric approach to estimate the effective thermal properties of the sand-water-ice system. A good agreement between experimental and modelling results was shown, but slightly different parameter sets were obtained for each temperature gradient. The presented method could be a simple way to characterize the freeze-thaw process in natural and artificial porous materials.

Keywords: Freezing and thawing cycles; Saturated packed sand sample; Modelling.

1 INTRODUCTION

Soil freeze-thaw cycles can cause changes in the geotechnical and hydrological properties of soils, leading to environmental problems such as frost heaving (Huang et al., 2020; Taber, 1929), changes in hydraulic conductivity (Othman and Benson, 1993), soil structure (Ding et al., 2019), deformation of buildings and transportation infrastructure such as pipelines (Landers and Streltskiy, 2023), and methanogenesis from thawing permafrost (Wagner et al., 2003). These effects are becoming increasingly important as human activities expand into areas covered by permafrost. Permafrost covers about one-third of the Earth's surface, mostly in the northern hemisphere. Permafrost is normally permanently frozen material covered by a layer of soil. However, in recent years it has been observed that permafrost is melting and not completely refreezing, even during the winter months (Welch, 2019).

More frequent freeze-thaw cycles (FTCs) can affect ecosystem diversity and productivity by causing changes in soil physical properties and affecting water movement in the subsurface. Therefore, freeze-thaw processes have recently been studied extensively in the field and in the laboratory.

Watanabe et al. (2011) conducted a laboratory study on column samples to investigate changes in water content and pressure under freezing conditions using temperature and time domain reflectometry (TDR) probes. The results showed that the pressure of unfrozen water was lower in the frozen soil than in the unfrozen water. As a result, the water moved toward the frozen layer closer to the surface.

The experiments of Smith and Onysko (1990) were also focused on the development of internal pressures in pores in freezing soil. Nsaif et al. (2019) studied soil behaviour during freezing and thawing cycles at different stress levels up to 1 MPa.

Models related to soil freezing and thawing processes, addressing various levels of complexity of the FTC process, have been developed and reported in the literature above. The

phenomenon of frost heave was first addressed by Taber (1930), stresses during phase transitions were studied by Skempton (1961), and mathematical models of frost heave were developed by Miller (1978). In recent decades, descriptions based on thermodynamic principles of macroscopic poroelasticity have been developed (Coussy, 2005; Hartikainen and Mikkola, 1997; Nishimura et al., 2009). Frémond and Nicolas (1990) presents macroscopic thermodynamics using a nonlinear variational approach. Rempel et al. (2004) and Rempel (2011) describe the effects of interfacial premelting and liquid films. Sweidan et al. (2020, 2022) studied macroscopic thermo-hydro-mechanical modelling of frost action and directional frost penetration. Suh and Sun (2022) simulated the formation of ice lenses. Vogel et al. (2019) introduced a simplified, non-iterative model of approach for modelling water flow and energy transport in a variably saturated, partially frozen soil, applicable to episodic freezing conditions. These models are typically validated by numerical or limited laboratory experiments. This is due to the complexity of the processes involved. Žák et al. (2018a) introduced a pore-scale model, assuming geometrically symmetric pores in 2D. A general pore-scale phase-field model of freezing was then introduced in Žák et al. (2018b) and analysed in detail in Žák et al. (2023). However, the pore-scale models are impractical for solving large-scale problems due to the computational cost.

Since the models for FTC simulation require complicated parameterization, a considerable amount of experimental data is needed to validate these models. Therefore, there is an urgent need for a simple and reliable experimental procedure.

The main objective of this study was to design a relatively simple experiment that would allow the characterization of the freeze-thaw process when coupled with a numerical model, without the need for direct measurement of the unfrozen water content. Freezing series and thawing cycles were conducted on a fully saturated sand sample to evaluate the performance of a numerical model using specifically designed experiments on a simple porous medium. The computer simulation of the

experiments utilized the multiphysics macroscopic model of porous medium solidification, as formulated and analysed by Žák et al. (2013).

2 MATERIALS AND METHODS

A laboratory setup was designed to study the effect of freezing and thawing cycles on sand samples in columns. Cylindrical sand samples were fully saturated with water and placed in a freezer. Heat transfer occurred mainly through the top surface of the sample to achieve a mostly vertical movement of the freezing-thawing front. This was achieved by insulating the side walls and bottom surfaces of the column. A vertical temperature gradient was anticipated to occur as a freezing front descended from the top of the column. To verify the boundary conditions and the effects of vertical temperature stratification in the freezer, introductory experiments were conducted using empty sample containers (refer to Supplementary material A.1) and with sample containers containing only water without sand (refer to Supplementary material A.2). Subsequently, experiments were performed on fully saturated sand samples.

2.1 Sample description

2.1.1 Sand sample

Sand samples (labeled S1 and S2) were prepared using silica sand ST 08 from Sklopisek Strelec in the Czech Republic. The grain size ranged from 0.1 to 0.63 mm and was analysed by sieving. The results of the particle size analysis are shown in Figure 1. The particle density of the sand was 2.65 g/cm^3 . The sand was composed 99.7% SiO_2 and had a pH of 7.3. The sand sample, measuring 15 cm in diameter and 20 cm in height, was prepared by packing layers of approximately 1 cm under water. The resulting porosity was 0.31, and the bulk density was $1,430 \text{ kg m}^{-3}$, which is similar to the parameters found in environmental soils. An aluminum lid covered the surface of the sand column, holding the sand in the container and conducting heat between the sample and the air. The lid was free to move up and down during FTC as the sample expanded and contracted.

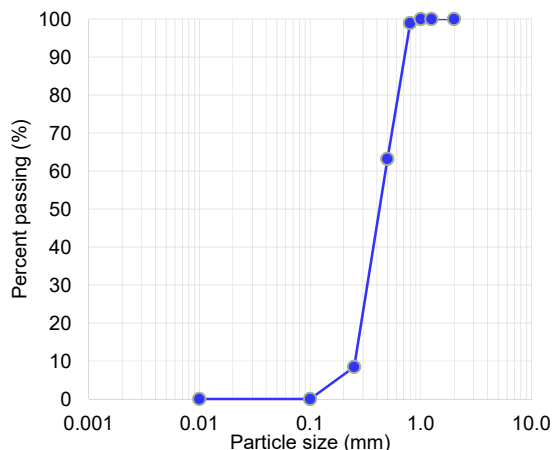


Fig. 1. Particle size distribution of sand.

2.2 Experimental setup

The experimental setup comprised of an insulated sample container placed in a commercial freezer (KFU 600, KESA s.r.o., Czech Republic) equipped with an automated control unit (KM 32, KESA s.r.o., Czech Republic). The sample container was

composed of inner and outer plastic tubes (refer to Figure 2). The 138 mm inner diameter and 220 mm in height inner tube was constructed using 10 mm thick polypropylene pipe with a thermal conductivity ranging from 0.17 and $0.22 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$, as specified by the manufacturer (Ostendorf – OSMA Ltd., Czech Republic). Fourteen layers of foam insulation (Kaiflex®, Kaimann GmbH) with a thermal conductivity of $0.034 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$, as specified by the manufacturer, were used to surround the inner tube. To insulate the bottom of the sample, a rigid, non-elastic material called cellular glass insulation (Owens Corning FOAMGLAS®, U.S.A.) was placed under the inner column. The insulated inner column was then inserted into an outer cylinder made of polyvinyl chloride (PVC) pipe, which was 326 mm in diameter and 7 mm thick (KGEM, Ostendorf - OSMA Ltd., Czech Republic). According to the manufacturers, the thermal conductivity of the outer cylinder was $0.20 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$. A 136 mm diameter and 20 mm thick aluminum lid with a thermal conductivity of $\lambda = 204 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ was placed on the sand surface. Temperature measurements were taken inside and outside the sample were made using stainless steel temperature sensors (109SS-L Campbell Scientific, U.S.A.) as shown in Fig. 3. The temperature probes have tolerance of $\pm 0.6^\circ\text{C}$ at -40°C , $\pm 0.38^\circ\text{C}$ at 0°C , and $\pm 0.1^\circ\text{C}$ at 25°C . Temperature sensors T1, T2, T3, and T4 were installed at depths of 7.5, 9, 13 and 17 cm, respectively, measured from the top of the sample. Two additional sensors, T5 (top) and T6 (bottom), were placed outside of the sample to monitor freezer conditions. All temperature sensors were connected to a data logger (CR 1000, Campbell Scientific, and U.S.A.) (see Figure 4) and recorded data every five seconds.

2.3 Experimental methods

2.3.1 Experiments with homogeneously packed, fully saturated sand

Two experiments, S1 and S2, were conducted on water-saturated sand samples. In experiment S1, the setpoint temperatures were cycled between $+10^\circ\text{C}$ and -10°C . Each temperature was maintained for 72 hours to allow sufficient time for freezing or thawing of the entire sample volume to occur and for the samples to reach the set temperatures. The duration of the FTC was determined based on previous experiments and preliminary modelling results. After temperature stabilization, two FTCs were conducted over a period of fifteen days. Unfortunately, temperature sensor T1 was not operational due to technical difficulties. Experiment S2 started with setpoint temperatures of $+5^\circ\text{C}$ and -5°C , each maintained for 120 hours to achieve temperature stabilization by the end of each phase.

2.4 Independent determination of the thermal properties of materials used in the setup

To verify the properties declared by manufacturers of materials used in the setup and provide additional information for modelling purposes, we conducted tests on their thermal properties. The thermal conductivity and heat capacity of Kaiflex insulation, glass foam, and fully saturated unfrozen sand were measured simultaneously using an ISOMET 2114 portable heat transfer analyzer (Applied Precision Ltd., Slovakia). The instrument measures heat dissipation from a thin needle in order to determine the thermal conductivity and thermal capacity of the tested materials. Measurements were conducted only on materials that could form a block of sufficient size for needle insertion. The thermal conductivity values obtained were $0.042 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ for Kaiflex insulation, $0.055 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ for the glass foam, and $3.067 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ for the sand. The volume heat



Fig. 2. Photographs of sample assembly (left) and the inner PVC sample holder.



Fig. 3. Photograph of the sample assembly placed in the freezer with temperature sensors connected.

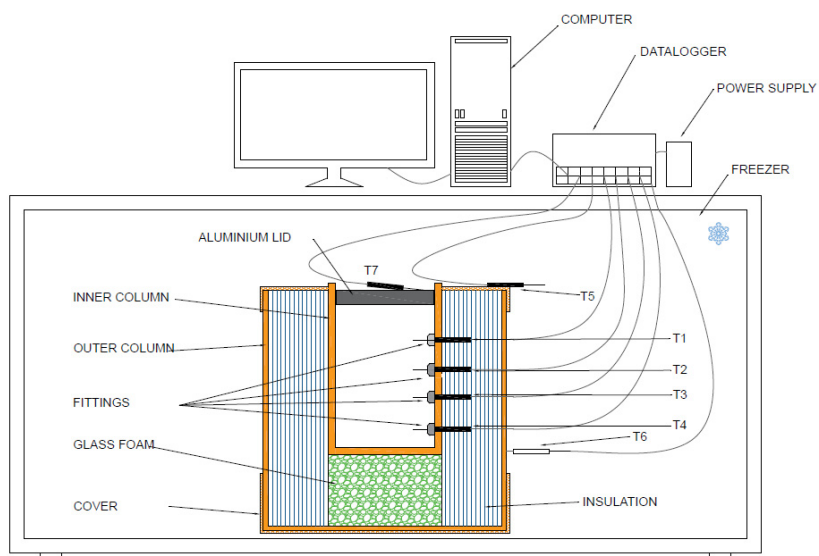


Fig. 4. Experimental setup.

capacity of Kaiflex insulation is $0.119 \text{ MJ}\cdot\text{m}^{-3}\cdot\text{K}^{-1}$, with a specific heat capacity of $1190 \text{ J}\cdot\text{kg}^{-1}\cdot\text{K}^{-1}$ at a density of $100 \text{ kg}\cdot\text{m}^{-3}$. The glass foam has a volume heat capacity of $0.193 \text{ MJ}\cdot\text{m}^{-3}\cdot\text{K}^{-1}$, with a specific heat capacity of $1930 \text{ J}\cdot\text{kg}^{-1}\cdot\text{K}^{-1}$ at a density $100 \text{ kg}\cdot\text{m}^{-3}$. The volume heat capacity of the saturated sand is $2.41 \text{ MJ}\cdot\text{m}^{-3}\cdot\text{K}^{-1}$, with a specific heat capacity of $1685 \text{ J}\cdot\text{kg}^{-1}\cdot\text{K}^{-1}$ at a density $1430 \text{ kg}\cdot\text{m}^{-3}$. The heat conductivity of Kaiflex insulation was determined using both the manufacturer's value and ISOMET. The ISOMET value was approximately 23% higher than the manufacturer's value. Thermal properties obtained from ISOMET measurements or from the materials' manufacturers were used as an initial estimate in modelling.

2.5 Numerical model of phase transition

The water-ice phase transition in the cylindrical sand sample placed in the experimental apparatus shown in Figure 5 was simulated by the in-house computational model. The measured experiments provided data for an optimal adjustment of the model parameters which allowed the reproduction of the described laboratory experiments. The model is then able to predict the dynamics of the laboratory freezing experiments, can serve in the design of future experiments and is versatile in accommodating more effects accompanying the soil freezing such as structural changes and macroscopic water flow in the porous medium.

$$\rho_{0j} c_{0j} \frac{\partial}{\partial t} T(x, t) = \nabla \cdot (k_{0j} \nabla T(x, t)), x \in \Omega_j, t \in [0, t_{\text{fin}}], j = \text{al}, \text{pp}, \text{g}, \text{zz} \quad (1)$$

The corresponding material properties (the density ρ_{0j} , the specific heat capacity c_{0j} and heat conductivity k_{0j}) have values summarized in Section 3.

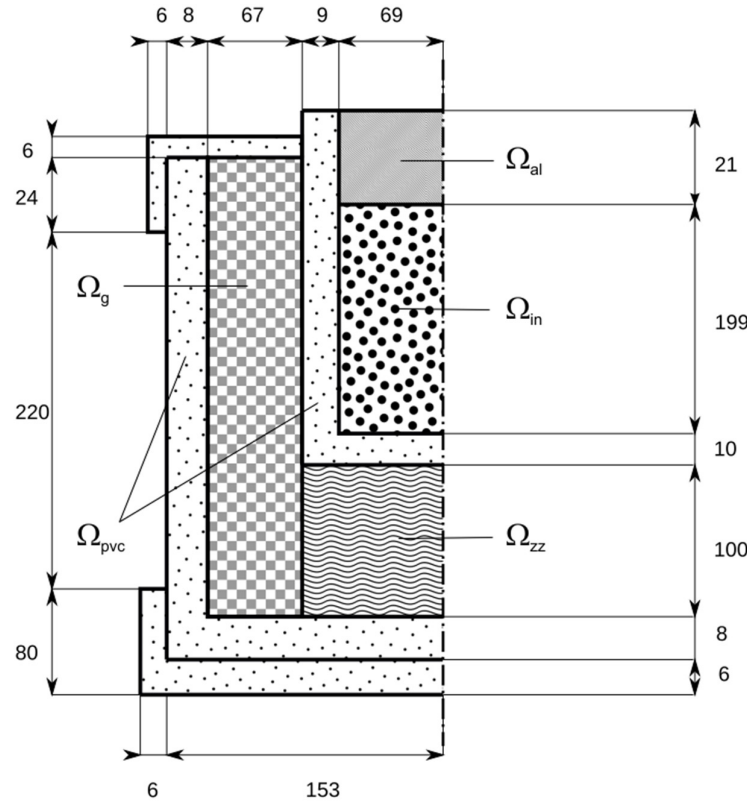


Fig. 5. Cross-section through the experimental apparatus. The entire domain of the cross-section Ω with the outer boundary $\partial\Omega$ is decomposed into several subdomains. The subdomains in which the same system of equation is applied are grouped together and indicated by the same pattern. The scheme is not to scale, the dimensions are expressed in mm.

The model is based on the transient heat transfer during the phase transition in the apparatus controlled by the conditions at its boundary. The computational model is derived from the mathematical model presented in Žák et al. (2013) and adapted for the geometry of the apparatus.

The volume occupied by the apparatus denoted as Ω consists of:

- the saturated sand Ω_{in} ,
- the container cover Ω_{al} ,
- the container walls Ω_{pp} ,
- the side insulation Ω_{g} ,
- the insulation base Ω_{zz} .

The mathematical model provides the space-time distribution of temperature $T = T(t, x) [^{\circ}\text{C}]$ in the apparatus. The temperature and the heat flux are continuous across the interior boundaries of the subdomains of Ω listed above. It is the solution of the heat balance equation inside of each of these subdomains. The form of this balance in all parts Ω_{al} , Ω_{pp} , Ω_{g} , Ω_{zz} except the saturated soil sample Ω_{in} is given by the heat-conduction equation

The heat balance within the solidifying fully liquid-saturated soil Ω_{in} is described using the enthalpy formulation of the heat equation as described in Žák et al. (2013). This model uses the volumetric liquid water content Θ to locate the liquid and solid phases. As in Nicolovsky et al. (2009), we adopt an empirically derived relation between the water content and soil temperature. A commonly used approximation of the water content is based on a power function

$$\Theta(T) = \begin{cases} 1 & \text{for } T \geq T^* \\ \left(\frac{T^*}{T}\right)^b & \text{for } T < T^* \end{cases}, \quad (2)$$

where η is the soil porosity, b is a positive constant, and T^* is the freezing point depression, Θ is the degree of saturation. These parameters are the properties of a given soil. The energy balance in Ω_{in} is then

$$\rho_{\text{in}} c_{\text{in}} \frac{\partial T}{\partial t} + \eta L \frac{\partial \Theta(T)}{\partial t} = \nabla \cdot (k_{\text{in}} \nabla T) \text{ in } \Omega_{\text{in}} \times (0, t_{\text{fin}}), \quad (3)$$

where L is the volumetric latent heat of fusion of water.

From the solution of (3) in Ω_{in} , the ice-water phase interface Γ_{iw} is detected as the level set of the temperature $T = T(t, x)$ given by the value of T^*

$$\Gamma_{\text{iw}}(t) = \{x \in \Omega_{\text{in}} \mid T(x, t) = T^*\}.$$

This approach is used in Žák et al. (2013). At pore scale, the interface Γ_{iw} is not anymore given by temperature. In Žák et al. (2018a), it is identified by a front tracking (related, e.g. to Pauš and Beneš, 2009) and in Žák et al. (2018b) and Žák et al. (2023), the phase-field method is used similar to, e.g., Beneš (2007).

The thermal properties of the sand saturated with water and subject to freezing are expressed parametrically by combining the properties of sand, water and ice based on their volumetric content. The density is

$$\rho_{\text{in}} = (1 - \eta) \rho_{\text{os}} + \eta (\Theta(T) \rho_{\text{oi}} + (1 - \Theta(T)) \rho_{\text{oi}}). \quad (4)$$

The heat capacity is

$$c_{\text{in}} = (1 - \eta) c_{\text{os}} + \eta (\Theta(T) c_{\text{oi}} + (1 - \Theta(T)) c_{\text{oi}}). \quad (5)$$

The thermal conductivity is

$$k_{\text{in}} = k_{\text{os}}^{1-\eta} \left(k_{\text{oi}}^\Theta k_{\text{oi}}^{(1-\Theta)} \right)^\eta. \quad (6)$$

The outer boundary of the experimental apparatus (the domain Ω) is divided into the external surface of the soil-sample cover Ω_{al} denoted as Γ_{al} and the remaining apparatus surface denoted as Γ_{w} .

The initial temperature distribution $T(x, 0) = T_{\text{ini}}(x)$ is given by the experimental data. The boundary conditions are given as follows. On the boundaries between particular subdomains, the continuity of the temperature and heat flux is considered except for the boundary between the soil sample and

its cover, where we consider a discontinuity of temperature given by

$$-k_{\text{oin}} \nabla T|_{\text{in}} \cdot \mathbf{n}_{\text{lid}} = \alpha_{\text{lid}} (T|_{\text{in}} - T|_{\text{al}}) \text{ on } \bar{\Omega}_{\text{al}} \cap \bar{\Omega}_{\text{in}} \times (0, t_{\text{fin}}). \quad (7)$$

Here, α_{lid} is the lid-sample resistance heat transfer coefficient and $\mathbf{n}_{\text{lid}}$ its outer normal vector pointing from the soil sample to the lid. On the outer boundary the Robin boundary condition is imposed to express the heat exchange between the apparatus and its surroundings. The condition reads as

$$-k_{\text{0al}} \nabla T \cdot \mathbf{n}_{\text{al}} = \alpha_{\text{soil}} (T - T_{\text{amb}}) \text{ on } \Gamma_{\text{al}} \times (0, t_{\text{fin}}), \quad (8)$$

and

$$-k_{\text{0pp}} \nabla T \cdot \mathbf{n}_{\text{wall}} = \alpha_{\text{wall}} (T - T_{\text{amb}}) \text{ on } \Gamma_{\text{w}} \times (0, t_{\text{fin}}). \quad (9)$$

Here, α_{wall} is the side-wall heat transfer coefficient and $\mathbf{n}_{\text{wall}}$ its outer normal vector, α_{soil} the top cover of the soil sample and $\mathbf{n}_{\text{al}}$ its outer normal vector. T_{amb} is the ambient temperature. While the ambient temperature can be readily determined (mostly by direct measurement), the value of heat transfer coefficients is more difficult to adjust. They depend on properties of materials transferring heat and also on their contact.

Therefore, determining the heat transfer coefficient may require a specific experimental measurement or additional verification methods.

The computational simulations using the above-described mathematical model of the experiments presented below were performed by the finite-element code implemented in the *Comsol Multiphysics* simulation software. It used the triangular quadratic Lagrange elements. The time evolution was computed by the time solver based on the implicit second-order accurate method described in Chung and Hulbert (1993). An example of the space-time evolution of temperature and phase composition is shown in Figure 6.

3 RESULTS

3.1 Experiment with fully saturated sand S1 and S2, and results of modelling

Figure 7 displays shows the temperature profiles during **Experiment S1**. The experiment began with freezing at an average temperature of -1.3°C (detailed temperatures in Table 1). The temperatures outside of the sample container, at sensors T5 and T6, were both -1.4°C and -1.4°C respectively. Thaw phase 1 lasted for three days, after which the average temperature inside the sample had stabilized at 9.5°C . The temperature outside the sample reached 10.3°C at T5 and 9.8°C at T6. During the initial freezing phase, the temperatures decreased at the same rate as in the second phase. After approximately twelve hours, all internal temperatures reached around 0°C . The temperature inside the sample dropped to -10.5°C during the first freezing episode. For sensor T2 the temperature decreased after 9.2 hours. It took 16.8 hours and 24 hours, respectively, for sensors T3 and T4 to begin to decrease in temperature. During the second thawing phase, the temperature increased to 0.2°C and reached its maximum value after three days. This phenomenon, in which the temperature stops at 0°C within the freezing or thawing phase was observed by Vogel et al. (2019).

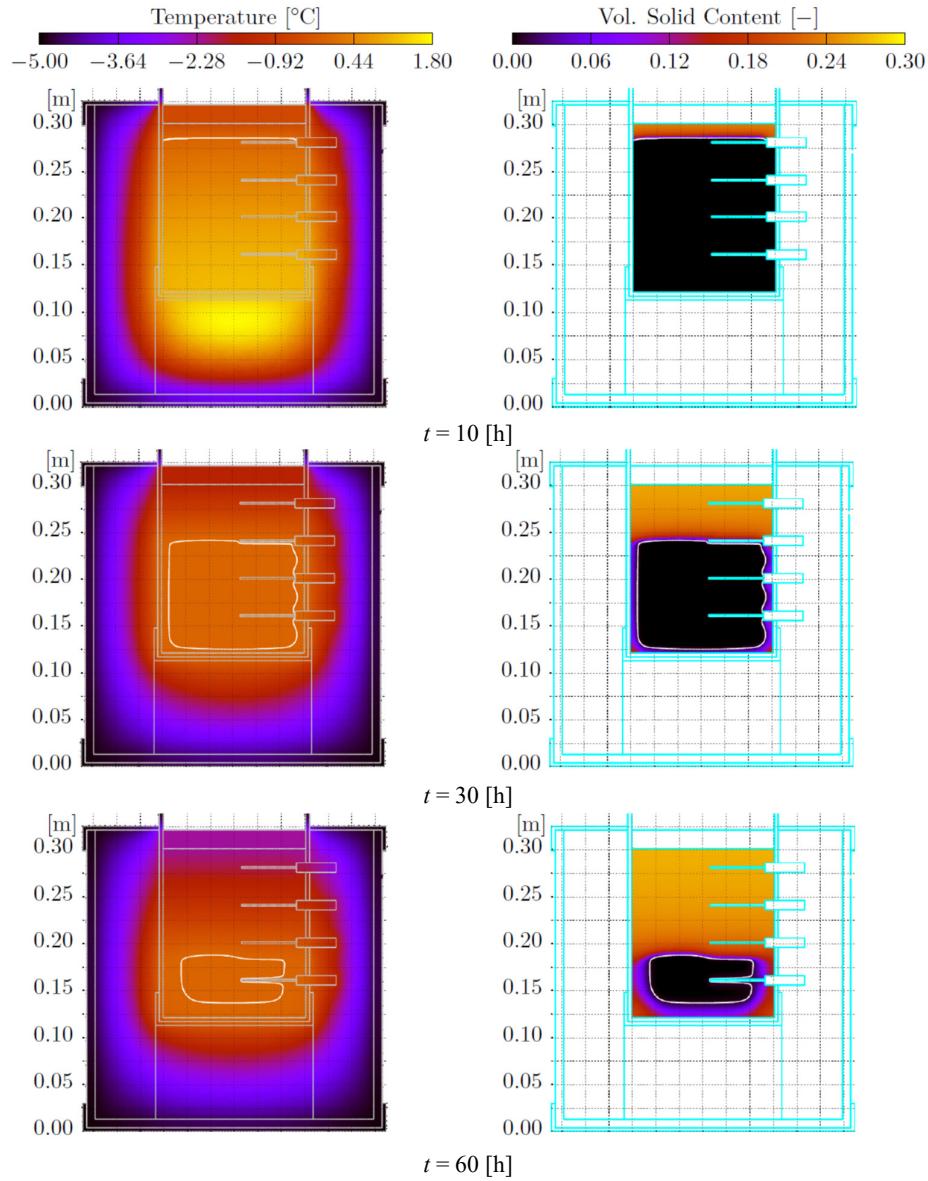


Fig. 6. Example of a numerical simulation of freezing of the water-saturated sand in an experimental apparatus. The left column displays the temperature, while the right column shows the volumetric solid phase content, both in a corresponding color scales. The gray and cyan contours indicate the apparatus components, while the white contours show the interface between the completely melted and at least partially frozen zones.

Table 1. Temperatures measured at the three temperature sensors (T2, T3, and T4) inside the sample container and their standard deviations, and temperatures outside the sample assembly (T5 and T6). Temperatures refer to the beginning (Start) of the experiment and to ends of corresponding phases of the experiment (thaw, freeze) during Experiments S1 and S2.

Experiment	Phase	T2 (°C)	T3 (°C)	T4 (°C)	σ (°C)	T5 (°C)	T6 (°C)	Set point temperature (°C)
S1	Start	-1.2	-1.3	-1.4	0.05	-1.4	-1.4	—
	Thawing 1	9.7	9.6	9.5	0.07	10.3	9.8	10
	Freezing 1	-10.6	-10.6	-10.7	0.04	-10.5	-10.5	-10
	Thawing 2	9.6	9.5	9.5	0.07	10.5	10.0	10
	Freezing 2	-10.6	-10.6	-10.7	0.04	-10.5	-10.6	-10
	Thawing 3	9.4	9.4	9.2	0.08	10.5	10.0	10
S2	Start	-5.6	-5.7	-5.7	0.07	8.2	3.9	—
	Thawing 1	3.4	3.3	3.2	0.11	5.7	4.7	5
	Freezing 1	-5.6	-5.7	-5.7	0.04	-4.9	-5.4	-5
	Thawing 2	3.5	3.4	3.3	0.11	5.7	4.7	5
	Freezing 2	-5.6	-5.6	-5.7	0.04	-4.8	-5.4	-5

The setup of material parameters in the model of Experiment S1 is shown in Table 4.

Table 2. Table of material parameters in the model adjusted for the experiment S1.

property	value	property	value
ρ_{0i}	915.00 kg m ⁻³	c_{0i}	1950.00 J kg ⁻¹ K ⁻¹
ρ_{0l}	995.00 kg m ⁻³	c_{0l}	4150.00 J kg ⁻¹ K ⁻¹
ρ_{0s}	2000.00 kg m ⁻³	c_{0s}	705.00 J kg ⁻¹ K ⁻¹
ρ_{0al}	2700.00 kg m ⁻³	c_{0al}	915.00 J kg ⁻¹ K ⁻¹
ρ_{0g}	50.00 kg m ⁻³	c_{0g}	400.00 J kg ⁻¹ K ⁻¹
ρ_{0zz}	100.00 kg m ⁻³	c_{0zz}	650.00 J kg ⁻¹ K ⁻¹
ρ_{0pp}	995.00 kg m ⁻³	c_{0pp}	1920.00 J kg ⁻¹ K ⁻¹
k_{0i}	1.98 W m ⁻¹ K ⁻¹	α_{soil}	10.00 W m ⁻² K ⁻¹
k_{0l}	0.65 W m ⁻¹ K ⁻¹	α_{wall}	20.00 W m ⁻² K ⁻¹
k_{0s}	3.18 W m ⁻¹ K ⁻¹	b	1.30
k_{0al}	235.00 W m ⁻¹ K ⁻¹	η	0.372
k_{0g}	0.04 W m ⁻¹ K ⁻¹	T^*	273.15 K
k_{0zz}	0.03 W m ⁻¹ K ⁻¹	α_{lid}	11.00 W m ⁻² K ⁻¹
k_{0pp}	0.25 W m ⁻¹ K ⁻¹		

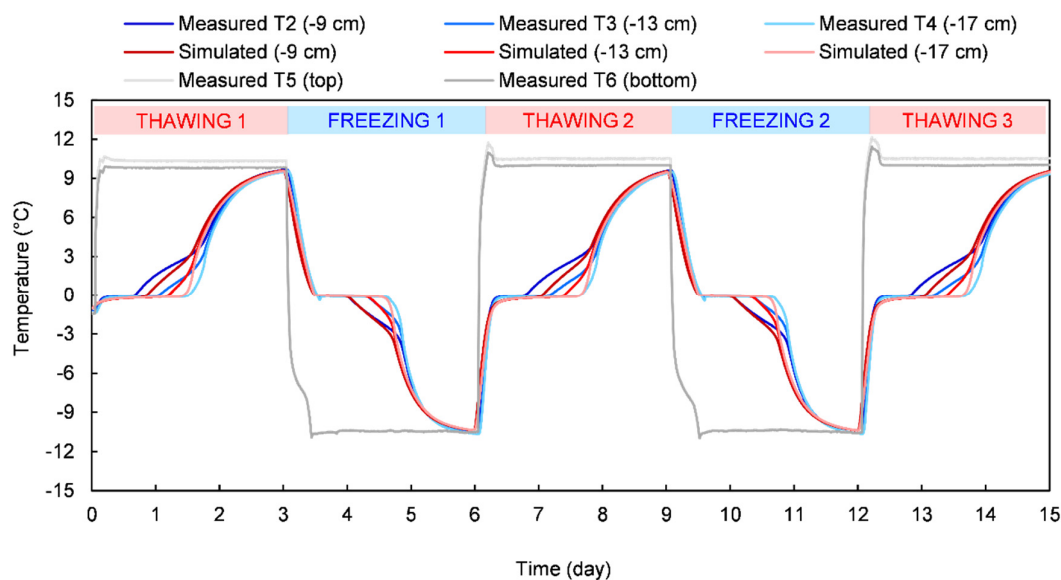


Fig. 7. Time evolution of measured and simulated temperatures at three depths during FTC in sand (Experiment S1).

The results of Experiment S1 showed a strong agreement between the measured and modeled data for the FTC, as depicted in Fig. 7. The temperature changes recorded by sensors T2 and T3 during all thawing phases were slightly faster than the modeled temperatures. However, for T4, the modeled temperature value increased slightly faster than the measured value. During all freezing phases, the simulated freezing front arrived slightly earlier than the measured one.

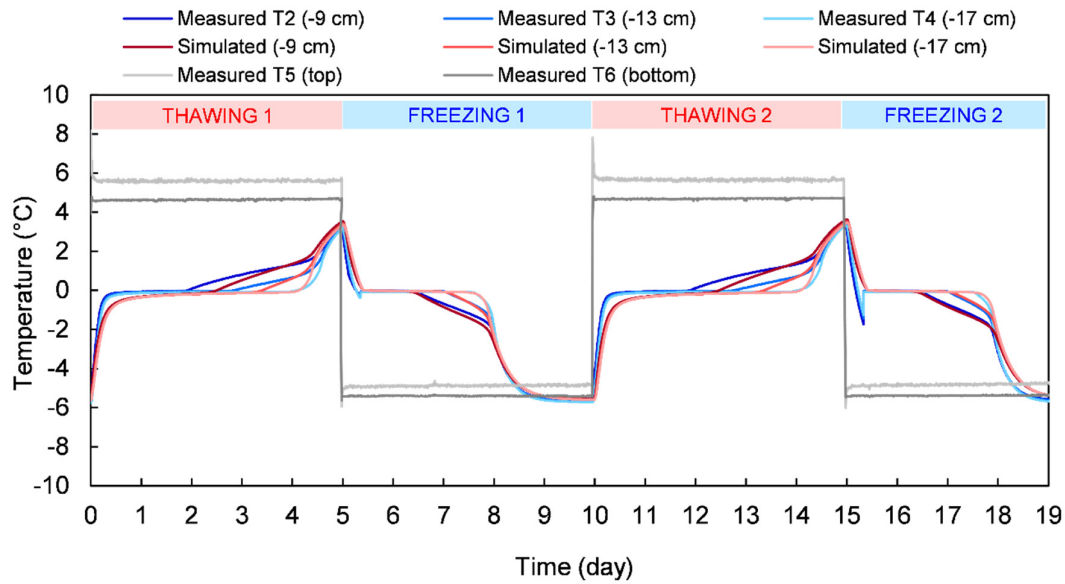
Experiment S2 was conducted using the same column and setup. Figure 8 displays the results. The initial temperature of the freezer was set to +5 °C. The temperatures recorded by the sensors outside the sample ($T_5 = 5.6$ °C and $T_6 = 4.6$ °C) were lower than the initial temperature. This discrepancy may be due to sensor inaccuracy. Freezing phase 1 occurred from day 5 to day 10. The difference between T_5 and T_6 may be caused attributed to temperature stratification inside the freezer or by sensor inaccuracy. During Thawing phase 2, with a set temperature of 5 °C, the temperature inside the sample increased to $T_2 = 3.5$ °C, $T_3 = 3.4$ °C, and $T_4 = 3.3$ °C. After 19.2 hours,

the temperature at sensors T1, T2 and T3 reached 0 °C. At the end of the phase, after 60 hours, the average temperature was 3.4 °C, $T_5 = 5.7$ °C and $T_6 = 4.7$ °C. During the freezing phase 2, which lasted from day 15 to day 19, the average temperature for all three inner sensors was found to be -5.6 °C, with T_5 and T_6 recording -4.8 °C and -5.4 °C, respectively. The vertical temperature gradient observed in experiment S1 was also present in S2. Table 3 shows the final sets of material parameters in the model of Experiment S2. Table 3 displays the ultimate material parameters in the Experiment S2 model.

The results of experiment S2 show a good agreement between the measured and modeled data for two thawing phases and two freezing phases (refer to Figure 8). The measured temperatures at T1 and T2 increased faster than the modeled temperatures during all thawing phases. However, the modeled temperature for T3 increased faster than the measured value during the thawing phases. In all phases, the simulated freezing front temperature was in good agreement with the measured temperatures.

Table 3. Table of material parameters in the model adjusted for Experiment S2.

property	value	property	value
ρ_{0i}	925.00 kg m ⁻³	c_{0i}	2150.00 J kg ⁻¹ K ⁻¹
ρ_{0l}	990.00 kg m ⁻³	c_{0l}	4275.00 J kg ⁻¹ K ⁻¹
ρ_{0s}	2200.00 kg m ⁻³	c_{0s}	740.00 J kg ⁻¹ K ⁻¹
ρ_{0l}	2750.00 kg m ⁻³	c_{0al}	915.00 J kg ⁻¹ K ⁻¹
ρ_{0g}	50.00 kg m ⁻³	c_{0g}	250.00 J kg ⁻¹ K ⁻¹
ρ_{0zz}	100.00 kg m ⁻³	c_{0zz}	750.00 J kg ⁻¹ K ⁻¹
ρ_{0pp}	945.00 kg m ⁻³	c_{0pp}	1770.00 J kg ⁻¹ K ⁻¹
k_{0i}	2.28 W m ⁻¹ K ⁻¹	α_{soil}	30.00 W m ⁻² K ⁻¹
k_{0l}	0.50 W m ⁻¹ K ⁻¹	α_{wal}	16.00 W m ⁻² K ⁻¹
k_{0s}	2.18 W m ⁻¹ K ⁻¹	b	1.20
k_{0al}	230.00 W m ⁻¹ K ⁻¹	η	0.38
k_{0g}	0.03 W m ⁻¹ K ⁻¹	T^*	273.15 K
k_{0zz}	0.06 W m ⁻¹ K ⁻¹	α_{lid}	10.00 W m ⁻² K ⁻¹
k_{0pp}	0.15 W m ⁻¹ K ⁻¹		

**Fig. 8.** Development of measured and simulated temperatures at three depths during freezing-thawing cycles in sand (Experiment S2).

4 DISCUSSION

General comment. It has been confirmed that the parameters for the freezing and thawing multiphysics model can be obtained through a relatively simple experiment. The experiment involves a cylindrical, fully saturated, sample that is insulated on three sides and placed in an adapted commercial freezer. Only the temperatures are monitored. In contrast to the measurements conducted with dual probe heat pulse sensors by Kojima et al. (2018) for a series of stabilized temperatures, the parameters in this study were obtained in a dynamic experiment over a wide range of temperatures from below to above freezing. Similar to the work of Zhang et al. (2015), the experiment in the current study was performed in a controlled temperature box, but without the need for specialized instrumentation. It does not consider the variation in thermal properties of ice at different temperatures.

On experiments. In Experiment S2, it was observed that during the freezing phases the temperature of the water-saturated sand continued to drop below 0 °C during the freezing phases before suddenly rising to 0 °C. The drop was quite significant

(over 2 °C) in the second freezing phase (Freezing phase 2) but was almost negligible in the first phase (Freezing phase 1). The cause of the observed phenomenon is likely due to the supercooling of soil water followed by abrupt crystallization (Kononov, 2015). It is unclear why the behavior differed in each cycle.

Test measurements taken on an air sample in the sample column indicated the occurrence of temperature stratification. The lowest temperature was found at the bottom of the sample, and the highest temperature was measured just below the top of the sample outside of the column. Temperatures in the distilled water sample and the saturated sand samples were found to be similar. Temperature changes initially occurred at the top of the samples and propagated to deeper layers. In the saturated sand experiment, a vertical temperature gradient was observed. The temperatures within the sample stabilized at 0 °C during the phase transition of freezing/thawing, which is consistent with the findings of Vogel et al. (2019).

Probe accuracy is determined by the interchangeability of thermistors, the accuracy of bridge resistors, and the error of the Steinhart-Hart equation. Interchangeability is the main source of

error. Temperature measurements can achieve an accuracy of ± 0.49 °C for a temperature range of -20 to $+70$ °C). Deviations in temperature measurements inside air and sand samples were small (± 0.08 °C), while an even smaller deviation was found in the water sample (± 0.03 °C). Lara et al. (2020) found larger standard deviations ranging from ± 0.05 °C to ± 0.4 °C. They attributed these deviations to the limitations of their laboratory setup, specifically freezing/melting point depression estimates. In our study, we observed larger temperature variations outside the sample, with deviations of around ± 1.0 °C in all cases.

On modelling. Experiments S1 and S2, which used a saturated sand sample, were simulated using the mathematical model described in Section 2.4. The physical parameters of the model, including the mass and thermal parameters of the container, its insulation, and of the sand and water, were obtained from standard sources. The porous medium mass and thermal parameters were obtained from Kumara et al. (2015) and Kiyohashi et al. (2003), respectively. Ice properties were summarized in Yen (1981). The heat transfer parameters for the boundary conditions were adjusted to values close to the measured data. A manual iterative optimization procedure was used to consider the space of all important parameters and reduce the gap between the simulated temperature data and the measured temperatures. The optimization process was complex and lengthy due to the rather flat profile of the distance. Slight modifications of the material parameters were required, as shown in Tables 4 and 5. The physical parameters obtained are still within the range found in the literature (refer to Section 2.2) and similar to those measured with the ISOMET apparatus. The description of Experiments S1 and S2 mentioned differences between the simulated and measured dynamics. These differences were attributed to additional small-scale effects, such as internal variability of heat conduction parameters of the container and its insulation, the presence of a thin water layer along the internal walls of the container, or heterogeneities in the soil sample.

4.1 Discussion of the potential application of the theory to address cold region problems. Implications of the results for real-world problems

The article discusses the complexity of thawing processes in soils, which are relevant to the pressing issues of global warming and climate change. Climate change is associated with further changes and damage to the landscape and the release of gases, particularly methane, into the atmosphere, sometimes explosively.

Governments and their relevant agencies have started to pay attention to phenomena that affect construction, transport, and other critical infrastructure. Thawing phenomena are being discussed in the media and are beginning to be part of general scientific interest. However, detailed knowledge of the physical principles of soil freezing and thawing is not yet complete (Hunt, 2022; Kindy, 2021; Olid et al., 2022).

This study contributes to research by developing a novel method for measuring parameters of the freeze-thaw process. These parameters are essential for predicting the thermal behavior and stability of infrastructure and ecosystems in cold regions. The proposed method is general enough to be applied to different soil types and climatic conditions.

5 CONCLUSIONS

This paper presents the results of an experimental and numerical study on the freezing-thawing behavior of water in fully saturated sand. The study includes the development of an

inexpensive and easily replicable experimental procedure to simulate the freezing-thawing cycles in a medium-sized sand sample placed in a modified commercial freezer. The reliability of the setup was tested in experiments on an empty sample container and on a container filled with pure water. By insulating the sides and bottom of the sample well, while allowing good thermal conductivity at the top of the sample, we achieved a nearly vertical advancement of the freezing and thawing front. We conducted experiments with a series of freeze-thaw cycles, using both higher and lower temperature gradients. The in-house developed numerical model utilized a parametric approach to estimate the effective thermal properties of the sand-water-ice system. The parameters were optimized to fit the experimental data, resulting in good agreement between the experimental and modelling results. Only minor parameter changes were necessary between experimental runs, indicating the reliability of the proposed methodology. In conclusion, the developed method that integrates experimental approaches with modelling of the freeze-thaw cycle requires further investigation across a diverse range of natural and engineered soil systems to fully ascertain its applicability and robustness.

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SUPPLEMENTARY MATERIAL

A.1 Introductory experiments on containers filled with air

The interior of the sample container was filled with air only (air sample, marked A1 and A2). To perform the experiment on an air-filled container the setup was slightly modified. Four thin wooden rods were placed along the wall inside the column to support the aluminum lid. The gap between the aluminum lid and the container walls was sealed with transparent adhesive tape to restrict the flow of air from the freezer into the container.

Experiments on container filled with air

The purpose of the empty (air-filled) container experiments (referred to as A1 and A2 in the following text) was to assess the behavior of the experimental setup itself, without a sample. We wanted to check for possible temperature stratification and the accuracy of the air temperature control. For A1, the temperature was set to cycle between 5 °C and 0 °C. Each cycle consisted of setting the temperature to 5 °C and maintaining that temperature for 24 hours (thawing phase), then switching to 0 °C and maintaining that for further 24 hours (freezing phase). This freeze-thaw cycle was repeated three times. Experiment A1 lasted for seven days to complete one thawing phase in order to initialize the setup. Temperature stabilization was then followed by three freeze/thaw cycles. In experiment A2 the temperature was cycled between 17 °C and 20 °C with each temperature being maintained for 24 hours to test the performance of the setup outside of freezing conditions. The total duration of test A2 was seven days.

Results of experiments with air-filled container A1 and A2

Experiment A1 began with set temperature of 0 °C. Three temperature cycles were then conducted with the set point temperature 0 °C and 5 °C. Due to an offset between the set point and actual temperatures, the actual temperatures oscillated above and below 0 °C and are therefore viewed as FTC. The temperature measured inside the sample at the end of the first high phase reached a maximum value of 3.1 °C (T2), 3.0 °C (T3), and 2.8 °C (T4). For the second, third, and fourth high phases,

the average temperature at the end of each phase was 3.2 °C, 3.2 °C and 3.1 °C, respectively. At T5, the maximum temperature measured was 4.7 °C for high phase 1. At the end of all high phases, the average temperature at T6 was 3.1 °C. When the set point temperature was changed from 5 °C to 0 °C, the temperature at the T5 temporarily dropped to −2 °C during the first fifteen minutes and then gradually stabilized at −0.4 °C in all low phases. The average temperature at T6 was −1.3 °C. The differences between temperatures T5 and T6 show the degree of temperature stratification in the freezer even though it was equipped with ventilation. During intervals when the temperature was set to 5 °C, the temperature at the top of the sample T5 stabilized at 4.1 °C after twelve hours. When the temperature was set to 0 °C, the average measured temperature stabilized at −0.9 °C after twelve hours. When the temperature was set at 0 °C, the average temperature inside the column was −1.4 °C. Detailed temperature data are shown in Table S1.

Experiment A2 began with a temperature set of 20 °C. Subsequently, the temperature was lowered to 17 °C resulting in actual temperatures ranging from 16.9 °C at T5 to 16.1 °C at T6 (refer to Table S1). Upon raising the set point to 20 °C, temperatures varied between 18.1 °C at T5 and 20 °C at T6. These fluctuations caused by modifications in the temperature stabilized within a few minutes. When the set temperature was lowered from 20 °C to 17 °C, the temperature inside the sample equilibrated and decreased to 18.1 °C within one day, as shown in Table 1. Temperature stratification was evident. At all intervals, the temperatures inside the sample (T2, T3, and T4) stabilized after one day. The temperature measured inside the sample was consistent with that measured by the external sensor T5.

Conclusions Temperatures measured at the three temperature sensors (T2, T3 and T4) inside the sample container were found almost similar. So, the column was well isolated. Standard deviations were found from 0.08 to 0.12. Temperatures outside the sample container (T5 and T6) were found different. The differences between temperatures T5 and T6 show the degree of temperature stratification in the freezer even though it was equipped with ventilation.

Table S1. Temperatures measured at the three temperature sensors (T2, T3 and T4) inside the sample container and their standard deviations, and temperatures outside the sample container (T5 and T6). Temperatures refer to beginning of the experiment and to ends of corresponding freezing and thawing phases of the experiment during experiments A1 and A2.

Experiment	Phase	T2 (°C)	T3 (°C)	T4 (°C)	σ (°C)	T5 (°C)	T6 (°C)	Set point temperature (°C)
A1	Start	−1.5	−1.6	−1.7	0.08	1.2	−0.3	–
	High 1	3.1	3.0	2.8	0.12	4.7	3.7	5
	Low 1	−1.4	−1.4	−1.5	0.06	−0.4	−1.3	0
	High 2	3.3	3.2	3.1	0.08	4.6	3.7	5
	Low 2	−1.5	−1.5	−1.6	0.08	−0.4	−1.3	0
	High 3	3.3	3.2	3.1	0.08	4.6	3.7	5
	High 4	3.4	3.2	3.1	0.08	4.7	3.7	5
A2	Start	15.2	15.2	15.1	0.07	16.9	16.1	–
	High1	18.1	18.0	18.0	0.07	20.0	19.1	20
	Low 1	15.2	15.2	15.1	0.07	17.0	16.1	17
	High 2	18.1	18.0	17.9	0.08	20.0	19.1	20
	Low 2	15.2	15.1	15.0	0.07	16.9	16.1	17
	High 3	18.1	18.0	17.9	0.07	20.0	19.1	20
	High 4	18.1	18.0	17.9	0.08	20.0	19.1	20

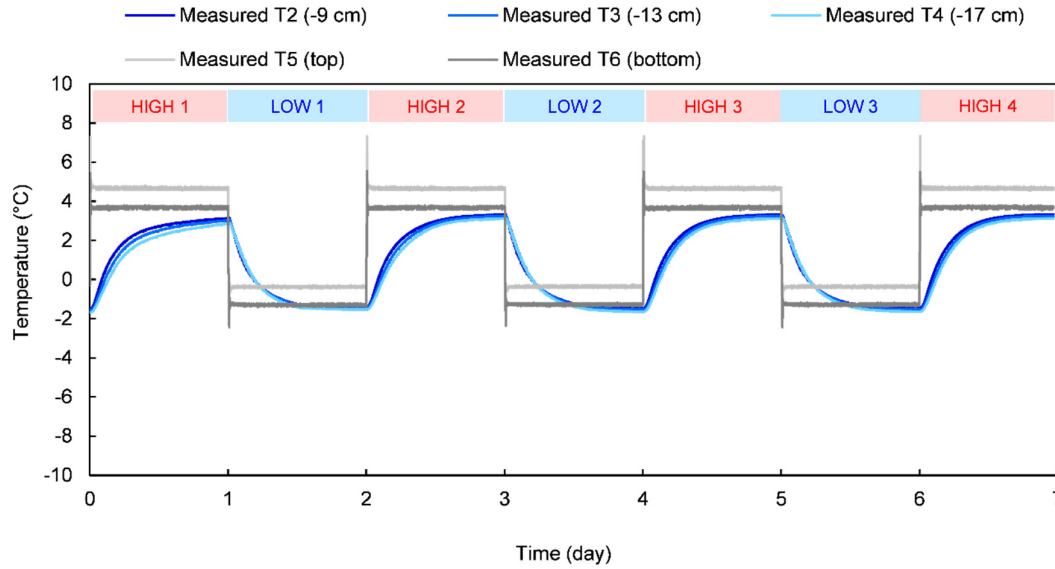


Fig. S1. Development of measured temperatures at three depths within freezing-thawing cycles on air (A1).

A.2 Introductory experiments on container filled with distilled water

An experiment was carried out on container filled with water (without porous media) was performed (referred to as W1 in the following text). The interior space of the container was completely filled with demineralized, boiled water to avoid the formation of bubbles. The surface of the sample was covered with an aluminum lid. The total volume of water in the container was 2.95 L, less than the volume of the sample column in order to allow for expansion.

Experiment on sample container filled with water

Similar to experiment A1, the experiment with demineralized water (referred to as W1 in the following text) was carried out with temperature cycles between 5 °C and 0 °C. The temperature was changed every 48 hours. Thus, four thawing cycles and three freezing cycles were performed.

Experiment with distilled water W1

Fig. S2 shows the temperature profiles during **Experiment W1** with distilled water. The average temperature inside the column ($t = 0$ day) was found to be -0.3 °C. All temperatures for the different cycles are shown in Table 2 in detail. The

differences between the stabilized temperatures at the top ($T5 = 4.6$ °C) and the bottom ($T6 = 3.6$ °C) of the column were about 1 °C for the first thaw phase. After two days, the set temperature was changed from 5 °C to 0 °C and the first freezing phase, which lasted two days, was initiated. During this phase, temperatures dropped to -0.2 °C at T2, T3, and T4, to -0.4 °C at T5, and to -1.3 °C at T6. All temperatures stabilized after one day. Thaw phase 2 started at $t = 4$ days. The average temperatures inside the column were similar to those seen during the first thaw phase (-0.1 °C). After six days, the temperatures inside the sample, calculated as the average of T2, T3, and T4, had increased to 2.7 °C, and to 4.6 °C at T5, and 3.6 °C at T6. The second freezing phase started at $t = 6$ days and the temperatures had stabilized at -0.2 °C after two days. When the temperature set point was changed from 0 °C to 5 °C, the values within the sample remained constant and increased at the same time.

Conclusions The experiments were carried out in order to determine the tightness of the experimental assemblies. Also, the temperature stratification was determined. It was found that temperatures inside the samples varied from the top and bottom temperature measured outside. The respond of temperature sensors was checked by these experiments. It was found the set point temperature higher than measured temperature inside the sample.

Table S2. Temperatures measured at the three temperature sensors (T2, T3, and T4) inside the sample container and their standard deviations, and temperatures outside the sample container (T5 and T6). Temperatures refer to beginning (Start) of the experiment and to ends of the freezing and thawing phases of Experiment W1.

Experiment	Phase	T2 (°C)	T3 (°C)	T4 (°C)	σ (°C)	T5 (°C)	T6 (°C)	Set point temperature (°C)
W1	Start	-0.2	-0.2	-0.3	0.02	-0.4	-1.3	-
	Thawing 1	2.9	2.9	2.8	0.03	4.6	3.6	5
	Freezing 1	-0.2	-0.2	-0.3	0.03	-0.4	-1.4	0
	Thawing 2	2.7	2.7	2.7	0.03	4.6	3.6	5
	Freezing 2	-0.2	-0.2	-0.3	0.03	-0.4	-1.3	0
	Thawing 3	2.7	2.7	2.6	0.03	4.6	3.6	5

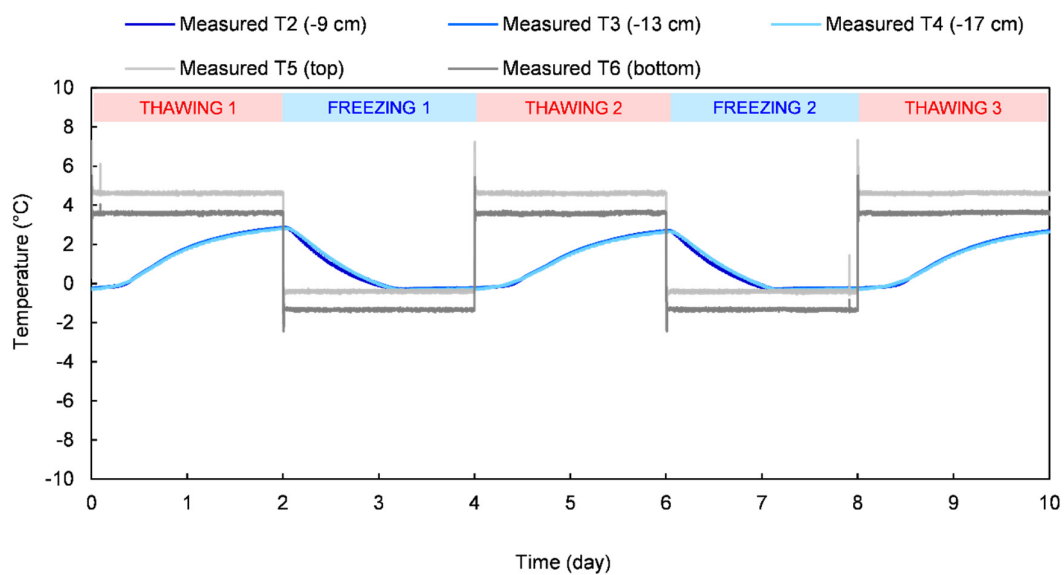


Fig. S2. Development of measured temperatures in three depths within freezing-thawing cycles on Experiment W1 with distilled water.