

# Equilibrium and non-equilibrium freezing of wet soils

*Boris V. Grigoriev<sup>1,\*</sup>, and Yulia F. Yanbikova<sup>1</sup>*

<sup>1</sup>Tyumen Industrial University, Volodarskogo str., 38, Tyumen, 625000, Russia

**Abstract.** The results of an experimental study of processes of equilibrium and non-equilibrium freezing of various types of wet soils are presented in this paper. Within the research carried out there was determined the behaviour of the content of unfrozen water when decreasing temperature, as well as the minimum time required for freezing soils in terms of thermodynamic equilibrium in the system “ice-water”. The empirical relationships determining the content of unfrozen water on temperature – under equilibrium conditions and temperature and freezing time – under non-equilibrium conditions have been presented.

## 1 Introduction

Key problems encountered when constructing and operating railways and highways, product pipelines, industrial and residential buildings in the territory of permafrost are frost heaving and subsidence as a result of frozen soil thawing, an abrupt change in the strength and rheological properties of soil, a stepwise change in thermal and physical parameters (heat capacity and thermal conductivity), are connected with interstitial crystallization / melting. The enumerated problems require monitoring, analysis and recording all processes and changes taking place in the frozen soil under the influence of engineering constructions in order to avoid deformation and destruction of the foundations of bearing and protecting constructions, reducing the stability of a roadway.

The feature of crystallization of interstitial water is that part of it does not freeze when reaching negative temperature by soil. So-called unfrozen water is formed, the amount of which depends on the value of negative temperature [1, 2]. Thus, soil properties continue to change in a negative temperature area and depend on the content of unfrozen water in soil.

It is necessary to point out that salts dissolved in interstitial water do not have a significant effect on the content of unfrozen water. By temperature reduction a part of salts precipitates, the other one is captured with ice while the third one is pushed into underlying layers [3]. The main factor is soil dispersion and as a result the value of active specific surface of mineral particles  $S_{ac}$ . More disperse soils have a larger area of mineral particles and therefore have a greater ability to bind water contained in pores. That's why the amount of water in an unfrozen condition is always greater in clay and loam than in sand at the same temperature. Nevertheless, at high concentrations of dissolved salts it is necessary to

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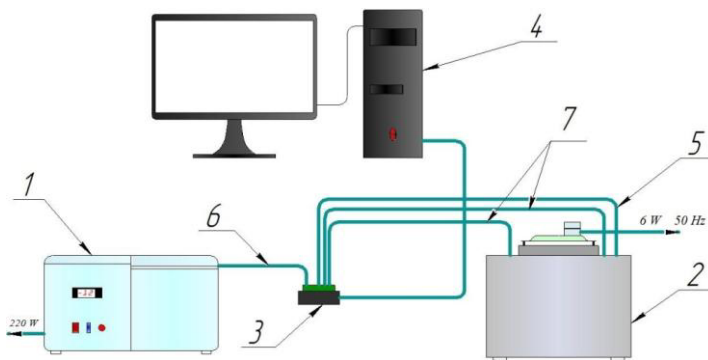
\* Corresponding author: b.v.grigorev@utmn.ru

take into account the influence of salinity on the content of unfrozen water when analyzing the system “ice-water” [4, 5].

An important feature of the process of wet soil freezing is that when freezing the system “ice-water” in soil is in the state of thermodynamic equilibrium only under the condition when the intensity of heat removal is below the accepted one for this type of soil. In case of exceeding this value the ratio of phases in the system “ice-water” is unstable and the content of unfrozen water is higher than the equilibrium one for this temperature [6, 7].

## 2 The description of the experimental setup

To study the processes of freezing and thawing in different types of soils the experimental setup [8, 9, 10] has been developed to determine the amount of unfrozen water with the calorimeter method. The general installation diagram is presented in Fig. 1.



**Fig. 1.** The experimental setup.

The experimental setup includes the following elements (Fig. 1.): thermostat 1; calorimeter 2; platinum resistance thermometers 6, 8, 5; analog-digital converter 3; computer 4, weighing bottles for soil; a set of glasses of different diameters; electronic scales.

## 3 The procedure of the experiment

The experiment is carried out in the following sequence: a weighing bottle is filled with investigated soil and a thermometer is inserted at the center of the weighing bottle. Further the weighing bottle is placed into a glass which is in the evaporator of the thermostat filled with antifreeze – tosol cooling liquid. The thermostat is turned on and the required negative temperature is set. Depending on the given intensity of heat removal freezing takes from several minutes to several hours. Then the frozen sample is placed in the calorimeter. The calorimetric experiment includes three stages: primary, main and final ones, during which the temperature readings are carried out by the resistance thermometers. On the basis of the calorimetric experiment ice content in the sample at the given negative temperature is calculated according to the formula 1 that is the consequence of the heat balance equation [9, 10].

$$m_i = \frac{K(\vartheta_0 - \vartheta_n) - |T_{s1} - T_{s2}|(c_s m_s + c_w m_{winput} + c_{wb} m_{wb})}{Q_{ph} - |T_{s1}|(c_w - c_i)} \quad (1)$$

where  $K$  [kJ / K] – the thermal parameter of the calorimeter determined in the course of an additional experiment;  $c_s, c_w, c_i, c_{wb}$  [kJ / kgK] – respectively the capacities of dry soil, water, ice and weighing bottle material,  $m_s, m_{winput}, m_i, m_{wb}$  [kg] – the masses of soil, water input, ice and weighing bottle;  $Q_{ph}$  [kJ / kg] – heat of phase transition;  $|T_{s1}-T_{s2}|, (\vartheta_0 - \vartheta_n^l)$  – the change of sample temperature and calorimetric liquid in the course of the experiment;  $\vartheta_n^l$  – the temperature of the last reference of the main period adjusted for heat exchange found according to the formula Regnault-Pfaundler-Usov [9, 11].

The weight of unfrozen water at the given negative temperature  $m_u$  is calculated on the difference:

$$m_u = m_{winput} - m_i$$

(2)

In the given series of experiments as a test soil there were used fine-dispersed wet sand of mass of 109.115 g, the mass of water in soil is 20.504 g, the mass of barren sample is 88.612 g, the mineralization is 0.687 g / l, clay of mass of 126.03g, weight of the sample after drying is 100.97 g, the mass of water in a wet sample is 25.06 g, the mineralization is 3.558 g / l and peat of mass of 67.05 g, the weight of dry soil is 20.84 g, the weight of water in the sample is 46.21 g, the mineralization is 5.065 g / l.

4 The results of the experiment

To determine the effect of freezing rate on the content of unfrozen water there were carried out a series of experiments for each soil when freezing with different intensity of heat removal. In the result for sand there were obtained 5 curves of unfrozen water, when freezing lasted 3 hours, 1 hour, 35 minutes, 25 minutes and 16 minutes (Fig. 2); for clay – 4 curves with the duration of freezing 5; 3; 1 hour and 16 minutes (Fig. 3); for peat - 4 curves with duration of freezing 4.5; 2.5; 1.2 hours and 40 minutes.

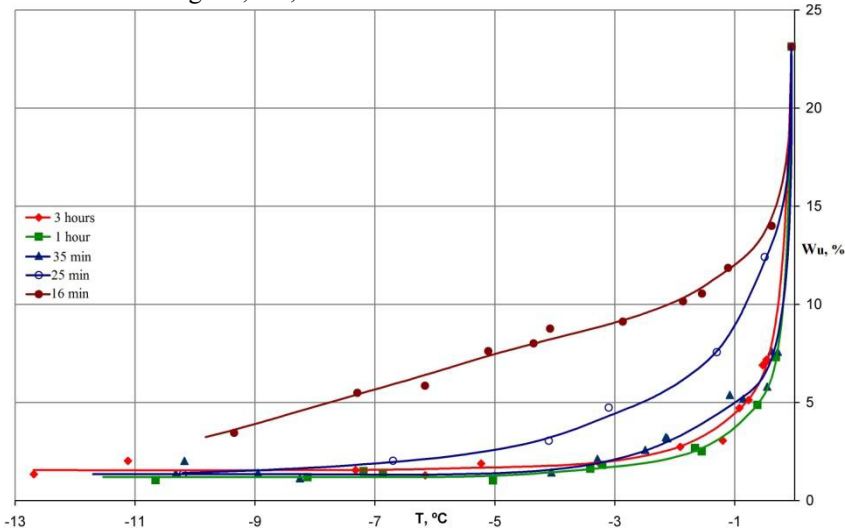
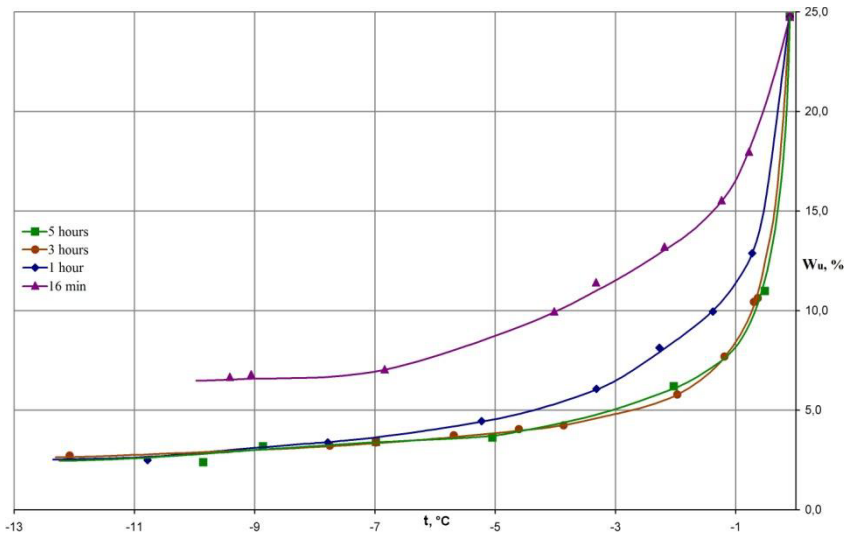


Fig. 2. The curves of unfrozen water content from temperature depending on freezing time (sand).

It can be seen that experimental points of the three curves in Fig. 2 (3 hours, 1 hour, 35 minutes) almost correspond, i.e., any of these three curves are equilibrium, the system “ice – unfrozen water” is in a stable thermodynamic equilibrium. At the same time as shown in

Fig. 2 when freezing during 16 and 25 minutes a remarkable deviation of the system from the equilibrium state is observed. The lower the final freezing temperature is, the smaller the deviation from the state of equilibrium is. It can be suggested that by continuing to conduct the experiment, for the curve “16 min”, for example, when  $T = -11^{\circ}\text{C}$  the obtained value  $W_u$  would correspond to the equilibrium content. This character of the curve according to the authors can be explained in the following way: when quick heat removal from the system “soil-water”, a part of free water does not crystallize turning out into an amorphous structure. At high negative temperatures forces of interaction between molecules of water do not have time to group them into tetrahedrons which is primarily due to high intensity of heat removal for a short period of time and high  $T$ . However, at lower temperatures as described in the article [12], forces of interaction significantly increase and a large number of molecules of water group into tetrahedron with release of latent heat of crystallization, i.e. molecular forces in water at low negative temperature prevail over disoriented effect at this intensity of freezing.

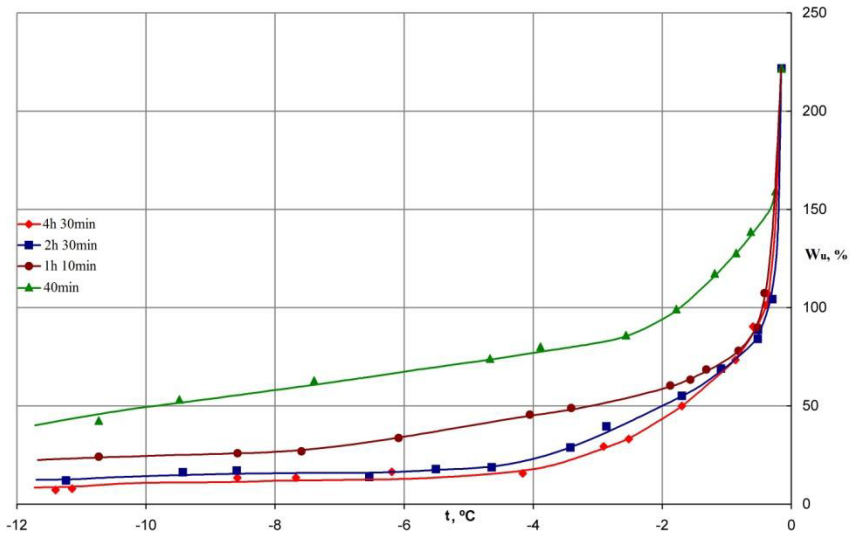
As for the equilibrium curves (Fig. 2) it is seen that the most part of water – 70 % is frozen for a narrow interval of temperatures from  $9$  to  $-0.5^{\circ}\text{C}$  that is explained first of all by a small value of specific active surface of mineral particles in comparison with, for example, clay. Therefore, most part of interstitial water is in a free state and freezes at about  $0^{\circ}\text{C}$ . Water bound with soil particles freezes in the interval from  $-0.5$  to  $-3^{\circ}\text{C}$ , after that there is, apparently, only tightly-bound and highly mineralized water produced in the result of squeezing salts when freezing free and bound water.



**Fig. 3.** The curves of the unfrozen water content from temperature at different times of clay freezing.

According to the given curves in Fig. 3 it is possible to judge about time necessary for phase equilibrium. The difference between the curves “5 hours” and “3 hours” is absent, that’s why it can be assumed that in these cases the equilibrium process of water freezing in soil pores takes place.

The curves “1 hour” and “16 minutes” correspond to non-equilibrium conditions of soil freezing. Frozen soil moisture depends not only on the kind and soil temperature, but also on the intensity of heat transfer from soil to the environment.



**Fig. 4.** The curves of dependence of the unfrozen water content on freezing time (peat).

Fig. 4 shows the effect of the factor of freezing time on the correlation of phases in the system “ice-water” for the same sample. The differences between the curves with freezing time 4.5 and 2.5 hours are not significant, so one can conclude that the lower curve corresponds to the most equilibrium process of water freezing in soil pores. The type of the curves “1.2 hours” and “40 minutes” indicates a significant deviation of the state of the system “ice-water” from the equilibrium one.

5 Approximation of experimental data

The approximation of the obtained experimental curves is necessary for the purposes of mathematical modeling of processes of soils freezing.

The approximation of the equilibrium curves which are selected as the curves “1 hour” for sand, “3 hours” for clay and “4.5 hours” for peat with the method developed by the authors is expressed by the following relationship (Fig. 2):

W(T)=e^{\frac{t}{10}f(t)}\cdot(W\_{initial}-W\_{rem})+W\_{rem} \tag{3}

where  $W_{rem}$  – the value of soil moisture that does not depend on temperature ( $t < 8\div 11^{\circ}\text{C}$ );  $W(t)$ – the target value of moisture at the temperature  $t,^{\circ}\text{C}$ ; the function  $f(t)$  is the following:

f(t)=d\_1t^3+d\_2t^2+d\_3t+d\_4 \tag{4}

The coefficients of the equation 4 are presented in Table 1.  
The absolute error between the experimental data and the values of moisture content obtained according to the formula 3 does not exceed for peat  $\pm 8\%$ , for clay  $\pm 1,5\%$ , for sand  $\pm 2\%$  in the coordinate scale of the given graphs.

**Table 1.** The empirical coefficients of the equation 4.

|                | Peat   | Clay   | Sand   |
|----------------|--------|--------|--------|
| d <sub>1</sub> | 0.030  | 0.022  | 0.095  |
| d <sub>2</sub> | 0.731  | 0.555  | 2.144  |
| d <sub>3</sub> | 5.748  | 4.629  | 14.567 |
| d <sub>4</sub> | 19.926 | 17.708 | 38.647 |

In these experiments the parameter characterizing non-equilibrium of phase transition “water-ice” in soil is the relation of heat capacity  $q$  [W], removing from mass  $m$  of the frozen soil in the control volume to the thermal capacity  $c$ , i.e.  $q/mc$  [°C / sec]. This ratio determines the rate of soil temperature change. As a parameter of non-equilibrium convenient for processing the experimental data the ratio of time of temperature change to the predetermined amount in non-equilibrium and equilibrium conditions is used:

$$\bar{\tau} = \frac{\tau}{\tau_0} \tag{5}$$

where  $\tau$ ,  $\tau_0$  – time of soil temperature change to  $\Delta t$ , °C in non-equilibrium ad equilibrium conditions.

The experimental data corresponding to the non-equilibrium curves in Fig. 2 and 3 allow obtaining empirical dependence of frost soil moisture content in non-equilibrium conditions [12]:

$$W(t, \tau) = W_{rem} + (W_{initial} - W_{rem}) \cdot \bar{W}(t)^{n(\bar{\tau})} \tag{6}$$

where  $\bar{W}(t) = \frac{W_0(t) - W_{rem}}{W_{initial} - W_{rem}}$ ;  $W_0(t)$  – the dependence of frost soil moisture content on the temperature in non-equilibrium conditions; for peat and clay  $n(\bar{\tau}) = c_1 + c_2 \ln(\bar{\tau})$ , for sand  $n(\bar{\tau}) = c_1 + c_2 \cdot \bar{\tau}$ . The coefficients  $c_1$  and  $c_2$  are presented in Table 2.

**Table 2.** The empirical coefficients.

|       | Peat | Clay | Sand |
|-------|------|------|------|
| $c_1$ | 1.02 | 1.00 | 0.12 |
| $c_2$ | 0.29 | 0.26 | 0.88 |

The absolute error of determining the relative clay moisture content according to the dependence 6 in the regarded conditions does not extend for peat  $\pm 10\%$ , for clay  $\pm 2\%$ , for sand  $\pm 3\%$  in the coordinates of the corresponding graphs.

It is important to add that in mineral soils the amount of unfrozen water, expressed in a percentage by weight of dry soil, is almost independent of the total moisture content of soil and is determined only by the value of negative temperature [13], a specific area of the active surface of mineral particles is a constant. This makes possible to use the same curves of unfrozen water (Fig. 2, 3) for any initial water content in soil (provided that its initial moisture content is higher than its molecular water capacity [14, 15]).

At the same time peat has a large volume of intracellular space, and part of moisture trapped inside a pore does not fall in the field of electro molecular attraction of a pore shell, remaining free. Only part of water in contact with the shell is bound. With increasing

moisture content the degree of interstitial filling with water increases and consequently the area of water layer in contact with the shell increases that results in increase of the amount of bound water. Thus, the value of active surface depends on the current moisture content. Therefore the change of the initial moisture content of peat up to the complete pore filling with water will influence the character of the curves in Fig. 4.

## 6 Conclusion

1. It is shown that under equilibrium conditions the most part of interstitial – up to 70 % freezes in a narrow temperature interval: in sand from 0 to  $-0.5^{\circ}\text{C}$ , in peat and clay from 0 to  $-3^{\circ}\text{C}$ .
2. There have been obtained the experimental data of non-equilibrium process of soil freezing with different intensity of heat transfer parameters. The intensity of freezing has the largest impact on non-equilibrium state of the system “ice-water” at high subzero temperatures.
3. It has been determined that the minimum time sufficient to freeze the equilibrium of soil within the experiments carried out for sand, clay and peat is 35 minutes, 3 hours and 3 and a half hours respectively.

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