

INFLUENCE OF MOISTURE CONTENT ON PERMEABILITY OF FROZEN AND UNFROZEN SOILS

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Gas permeability is studied experimentally in samples of wet frozen and thawing sandy soils with different moisture contents, 3 cm in diameter and 3–5 cm in length, using a specially designed system. The variations of permeability in frozen and unfrozen sand samples at different temperatures and pressures are estimated as a function of saturation with ice or water. Permeability becomes several orders of magnitude lower as saturation exceeds the critical values of 40–50 % and 50–55 % for frozen and thawed samples, respectively. The behavior of the saturation dependence of gas permeability in frozen and unfrozen sandy soils is largely controlled by their microstructure, and especially by the type of ice cement between soil grains which, in turn, depends on moisture content. The permeability difference between the frozen and unfrozen sand samples increases proportionally to saturation.

Gas permeability, sandy soil, frozen soil, thawing, unfrozen soil, moisture content, ice saturation, saturation degree

INTRODUCTION

Gas permeability of frozen and thawing soils has important implications for natural gas emission in the Arctic areas and for chemical exploration in permafrost. Permafrost was presumed to be poorly permeable, and its gas reservoir properties were doubted [Yakushev, 2009]. However, migration and accumulation of hydrocarbon gases were found to continue to some extent in frozen sediments as well, while the permafrost was reported [e.g., Olovin, 1993] to contain permeable zones. On the other hand, the amount of ice in pores influences the reservoir and screening properties of soils, including permafrost [Yakushev, 2009].

There is geochemical evidence for migration of hydrocarbons through shallow permafrost [Glotov *et al.*, 1985; Are, 1998; Glotov, 2005]. Reports of methane venting in the Arctic land and shelf areas [Are, 1998; Shakhova *et al.*, 2010] additionally motivate special experimental studies of gas permeability of frozen and thawing sediments.

According to published data, gas permeability of soils decreases considerably upon their freezing but remains instrumentally resolvable. Specifically, Ananyan *et al.* [1972], who were among the first to estimate gas permeability in experiments, observed that sand filled with ice to 65–75 % remained permeable: 0.36 mD at grain sizes of 0.1–0.25 mm and a saturation of 75 %.

Starobinets and Murogova [1985] investigated the permeability of permafrost with respect to hydrocarbon flow and found out frozen dolomites at –5...–7 °C containing 67 % pore moisture to be two orders of magnitude less permeable than their dry counterparts and about tens times less permeable than the unfrozen ones.

Air permeability of sand samples studied as a function of ice- and water-filled porosity was reported to be lower in frozen samples than in unfrozen ones, at the same saturation [Seyfried and Murdock, 1997].

Experiments on gas and water permeability of dry, moist, frozen, and hydrate-bearing sand samples exposed to hydrate saturation Kneafsey *et al.* [2008] showed that the relative permeability of samples with an initial moisture content of 42 % became more than twice lower after freezing (0.2 against 0.5–0.6 unit fraction), while the reduction was less than 10 % (from 0.68 to 0.62) in drier samples.

Freezing of sand and silt samples saturated with hydrates in laboratory caused up to ten-fold reduction in gas permeability [Chuvilin *et al.*, 2014] as a result of resident pore water freezing and additional hydrate formation.

Experiments of Wang *et al.* [2014] on methane permeability of frozen sand with different ice contents gave the respective empirical relationships.

Soils change in permeability upon pore water freezing, as well as upon cyclic freezing and thawing. For instance, the hydraulic permeability of clay exposed to freezing-thawing cycles increased after one such cycle [Chamberlain and Gow, 1979] because thawing of small ice lenses which formed during the preceding freezing produced microcracks. The cyclically freezing and thawing rocks can be expected to change their permeability with respect to gas as well.

Note however that the effects of pore moisture phase change on permeability, as well as the related processes, remain insufficiently constrained in spite of the abundant experimental evidence.

METHODS

Gas permeability variations in frozen and unfrozen sediments were studied using a method designed by the authors jointly with people from the *Schlumberger* Company for hydrate-bearing rocks [Chuvilin *et al.*, 2013]. The procedure implied gas flushing through samples on a system providing the required temperature and pressure conditions for freezing and hydrate saturation of soils [Chuvilin and Grebenkin, 2015]. The system designed by the *EkoGeosProm* LLC (Fig. 1) can maintain sample temperatures and pressures from -15 to $+30$ °C and up to 100 bar, respectively.

The work included several steps: (i) preconditioning of samples to provide user-specified moisture content and to saturate them with gas in a special pressure vessel; (ii) freezing and thawing of the samples; (iii) testing the samples for gas permeability at different gas pressures and temperatures; (iv) processing the experimental data.

Gas permeability was measured at each cooling and heating cycle while temperature and pressure were monitored continuously during the experiment. Confining pressure in the system was set to ~ 60 bar. The gas permeability measurements were applied first to samples previously frozen in the system to -5 °C and saturated with nitrogen. Then the pressure vessel was heated to room temperature ($+20$ °C), while thermal expansion of oil increased slightly the pressure. Then infiltration tests were performed on the thawed samples after the temperature and pressure had stabilized.

The effective permeability of the sample in the pressure vessel was inferred from pressure drop in receivers with the known gas volume, by solving the differential equation of mass transfer through the sample under the pressure gradient [Chuvilin *et al.*, 2013]:

$$K = \frac{2\eta L V_1 p_1}{S_p p_{10} (p_1^2 - p_2^2)} \frac{p_{10} - p_{1k}}{p_1 t_1},$$

where η is the dynamic viscosity of gas, Pa·s; L is the length of sample, cm; S_p is the cross section area of sample, cm²; V_1 is the receiver volume, cm³; p_1 is the pressure at sensor 1 (Fig. 1) at the time t_1 , bar; p_2 is the pressure at sensor 2 at the time t_1 , bar; p_{10} is the pressure at sensor 1 at the start time, bar; p_{1k} is the pressure at sensor 1 at the end time, bar.

The quality of the gas permeability estimates was checked by gauging against four ceramic samples with known absolute permeabilities [Chuvilin *et al.*, 2013]. The gauging tests estimated effective permeability of standard samples with respect to methane. Our results were compared with those obtained for standard samples in other laboratories using the Klinkenberg correction (K_k). Thus estimated error was within 15 %.

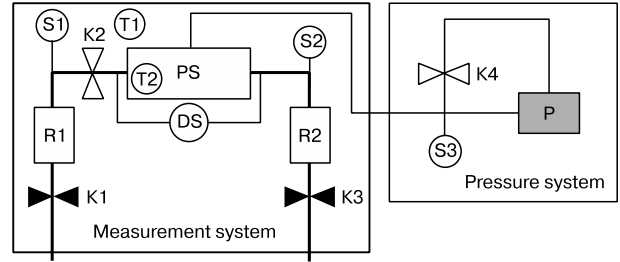


Fig. 1. Sketch of experimental setup for evaluating gas permeability in samples saturated with hydrate.

K1–K3 are membrane valves for gas input; K4 is ball valve of hydraulic system; R1, R2 are gas receivers; S1–S3 are pressure sensors; DS is differential pressure sensor; P is hydraulic pump with an oil tank; PC is pressure vessel; T1, T2 are temperature sensors.

SAMPLES

Gas permeability was measured in sand sampled from marine permafrost (mQ₃) within the South Tambei gas-condensate field in the Yamal Peninsula. The samples were cut from frozen core (samples fs-1 and ss-1) and either left wet or were dried in air and thus disturbed relative to the natural state (most of tests used dry samples). According to grain sizes, the samples were either fine sand (fs) or silty sand (ss) from 36.5–46.0 m and 27–48 m core depths, respectively.

Minerals in the samples were identified by X-ray diffraction (XRD) and the grain sizes were estimated by sieving. Their physical properties corresponded to the Standards of GOST 5180-84 [2005] and Construction Norms and Regulations SNiP 2.02.04-88 [1990] (Table 1).

Fine sand consisted mainly (62.3 %) of 0.25–0.1 mm grains, which were 93.7 % quartz, 5.1 % albite, and 1.2 % orthoclase, with 2.69 g/cm³ density of the solid component and 0.06 % salinity.

A greater part (57.8 %) of silty sand grains were likewise 0.25–0.1 mm sizes but quite many (27.7 %) were 0.1–0.05 mm (Table 1). Silty sand contained notably less quarts than fine sand (74.1 %) but more albite and orthoclase (12.8 and 10.0 %, respectively). The density and salinity were, respectively, 2.68 g/cm³ and 0.13 %. Silty sand grains had much larger specific surface areas than fine sand (1.37 m²/g against 0.24 m²/g, respectively).

Table 1. Lithology and grain sizes of samples

Lithology	Percentages of grain size fractions, %			
	1–0.5 mm	0.5–0.25 mm	0.25–0.1 mm	0.1–0.05 mm
Fine sand	0.2	29.1	62.3	8.4
Silty sand	0.1	14.4	57.8	27.7

Table 2. Parameters of samples

Sample	Length, mm	Diameter, mm	Water content, %	Porosity, u.f.
<i>Fine sand</i>				
fs-1	30	30	18	0.43
fs-6	29	30	14	0.41
fs-2	30	30	13	0.41
fs-8	29	30	12	0.41
fs-7	30	30	10	0.40
fs-3	24	30	10	0.40
fs-4	34	30	6	0.39
fs-5	29	30	0	0.37
<i>Silty sand</i>				
ss-1	30	30	16	0.38
ss-6	29	30	13	0.38
ss-2	30	30	12	0.38
ss-8	30	30	11	0.37
ss-7	28	30	10	0.37
ss-3	28	30	9	0.37
ss-4	29	30	5	0.36
ss-5	28	30	0	0.35

The samples were 25–35 mm long and 30 mm in diameter (Table 2).

The water contents of natural wet frozen samples were 18 wt.% in fine sand (sample fs-1) and 16 wt.% in silty sand (sample ss-1). The initial moisture of air-dried samples was set within 0 to 14 %, while their porosity was 0.37–0.43 (fine sand) and 0.36–0.38 (silty sand).

Table 3. Gas permeability of frozen samples

Sample	W, %	S_f , %	K, mD
<i>Fine sand</i>			
fs-1	18	74	<0.01
fs-6	14	55	<0.01
fs-2	13	51	<0.01
fs-8	12	51	0.53
fs-3	10	39	25.17
fs-7	10	39	5.13
fs-4	6	23	94.47
fs-5	0	0	288.50
<i>Silty sand</i>			
ss-1	16	80	<0.01
ss-6	13	62	<0.01
ss-2	12	57	<0.01
ss-8	11	52	0.03
ss-7	10	48	2.40
ss-3	9	40	10.36
ss-4	5	24	74.30
ss-5	0	0	177.70

Note. W is water content; S_f is ice saturation; K is effective gas permeability.

GAS PERMEABILITY VARIATIONS IN ROCKS UPON THAWING

In all tests, initial permeability to gas was measured in samples frozen to about -5°C and showed variations in a large range depending on ice saturation (Table 3): tens of mD at 23–24 % of pores filled with ice, a few mD at 50 %, and below the instrumental resolution (<0.01 mD) at >50 %.

Then gas permeability measurements were applied to the same samples thawed at room temperature (Table 4), and the variations were from 0.3 to 291.6 mD depending on moisture contents. The samples became notably more permeable to gas after thawing. For example, it was below the instrument precision (0.01 mD) in intact wet frozen sample of fine sand (fs-1) with initial ice saturation 74 % but increased to 0.27 mD when it thawed (Table 4, Fig. 2, *a*). Or, frozen samples with 51–55 % filled pores changed in permeability from $K < 0.01$ mD (frozen) to 50–60 mD (thawed). The permeability of samples fs-3 and fs-7 with an initial ice saturation of 39 % became 3.4 times higher (84.80 mD against 25.17 mD), but sample fs-4 with 23 % of pore ice became only twice more permeable upon thawing (184.10 mD against 94.47 mD) (Fig. 2, *a*). Thus, permeability increase upon thawing is slower at higher saturation. The permeability of dry samples changed only slightly after thawing: e.g., 288.5 and 291.6 mD at negative and positive temperatures, respectively, in sample fs-5 (Table 4).

Gas permeability in silty sand samples varied in a similar way as a function of pore ice (Table 4).

Table 4. Gas permeability of unfrozen samples

Sample	W, %	S , %	K, mD
<i>Fine sand</i>			
fs-1	18	68	0.27
fs-6	14	50	50.52
fs-2	13	46	59.97
fs-8	12	43	68.64
fs-7	10	36	78.38
fs-3	10	36	84.80
fs-4	6	21	184.10
fs-5	0	0	291.60
<i>Silty sand</i>			
ss-1	16	69	0.60
ss-6	13	57	3.87
ss-2	12	52	45.00
ss-8	11	48	38.46
ss-7	10	44	59.38
ss-3	9	37	69.90
ss-4	5	22	151.30
ss-5	0	0	181.40

Note. W is water content; S is water saturation; K is effective gas permeability.

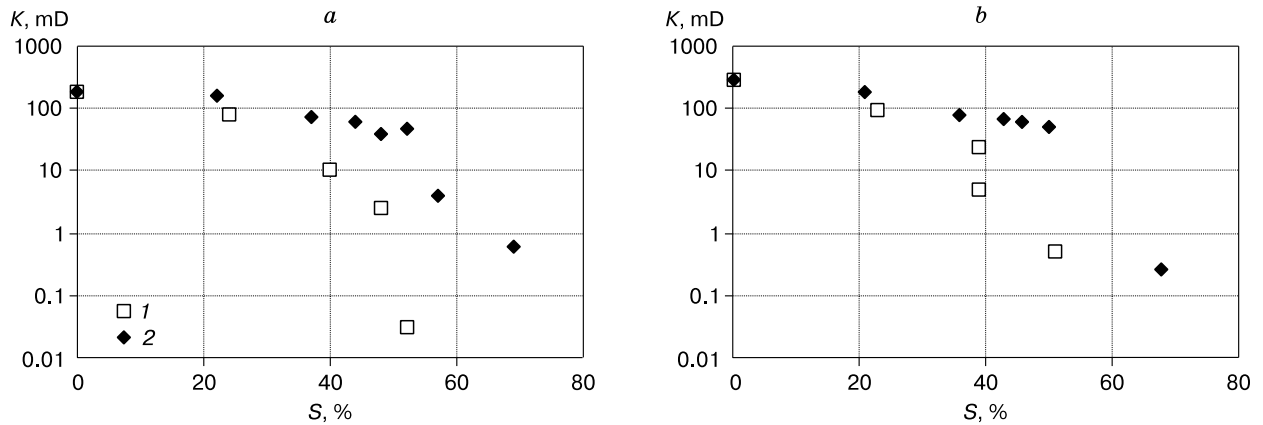


Fig. 2. Saturation (S) dependence of gas (K) permeability in frozen (1) and thawed (2) fine (a) and silty (b) sand.

Namely, it increased from 0.01 mD in frozen sample ss-1 with 80 % ice saturation to 0.60 mD after thawing, and up to 45.0 mD in almost impermeable ($K < 0.01$ mD) dried sample ss-2 with 57 % ice filling (Fig. 2, b). The increase in gas permeability upon thawing was 6.7-fold in sample ss-3 with 40 % ice saturation (from 10.36 to 69.90 mD) but only two-fold (from 74.30 to 151.3 mD) in ss-4 with 24 % pore ice. Thawing of dry sample ss-5 caused almost no gas permeability change (Fig. 2, b).

In general, the fine sand samples with ice and water saturations increasing from 0 to 40 % and from 0 to 50 %, respectively, become low permeable, and still less permeable (to below the instrument precision 0.01 mD) as the saturation degree increases further (Figs. 2, a).

The difference in gas permeability between the frozen and unfrozen samples of fine sand and silty sand increases proportionally to their saturation degree (S): less than ten-fold at $S = 40$ % and 3–5 orders of magnitude at $S = 40$ –80 %, while samples at $S > 80$ % are almost impermeable in both frozen and unfrozen states.

Thus, at similar saturations, frozen samples are less permeable than the unfrozen ones and the difference is smaller in less saturated samples.

SATURATION DEPENDENCE OF GAS PERMEABILITY OF SAND SAMPLES

The experimental gas permeability data were used to estimate relative permeability of the samples. The obtained estimates generally agree with other published evidence [Kneafsey et al., 2008; Wang et al., 2014] (Fig. 3), some discrepancy being possibly due to difference in microstructure and composition of the samples.

The general trend of relative permeability for frozen sand samples as a function of ice saturation

(Fig. 3) is that it becomes about an order of magnitude lower as the saturation (S_i) increases from 0 to 40–50 % and then decreases for another order of magnitude upon further S_i increase to 60–70 %. Wang et al. [2014] reported relative permeability of frozen sand samples to be no higher than 0.01 at a saturation of 60–70 %. The relative permeability data points in Fig. 3 fall into two fields: slow reduction with ice saturation increase within $S_i < 40$ –50 % and rapid drop exceeding two orders of magnitude at $S_i > 40$ –50 % (Fig. 3).

The saturation-controlled behavior of gas permeability depends on soil microstructure, especially on the form of ice cementing sand grains. At different amounts of initial moisture in sand, the ice cement can be of contact, film, pore, or basal types [Ershov,

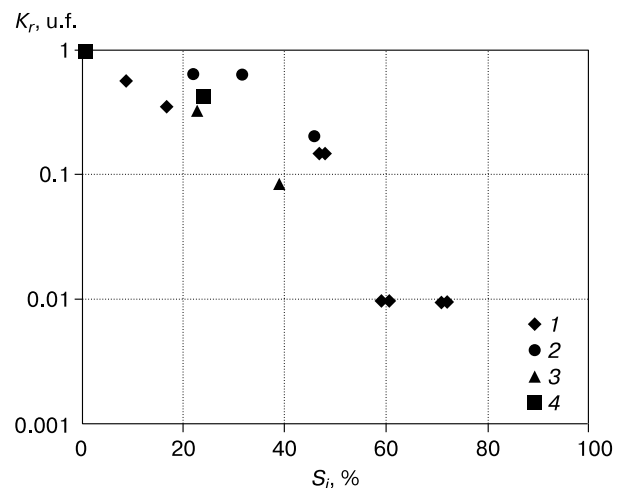


Fig. 3. Experimentally estimated relative permeability (K_r) of samples as a function of saturation with ice (S_i), according to Wang et al. [2014] (1), Kneafsey et al. [2008] (2), this study (3 and 4, for fine and silty sand samples, respectively).

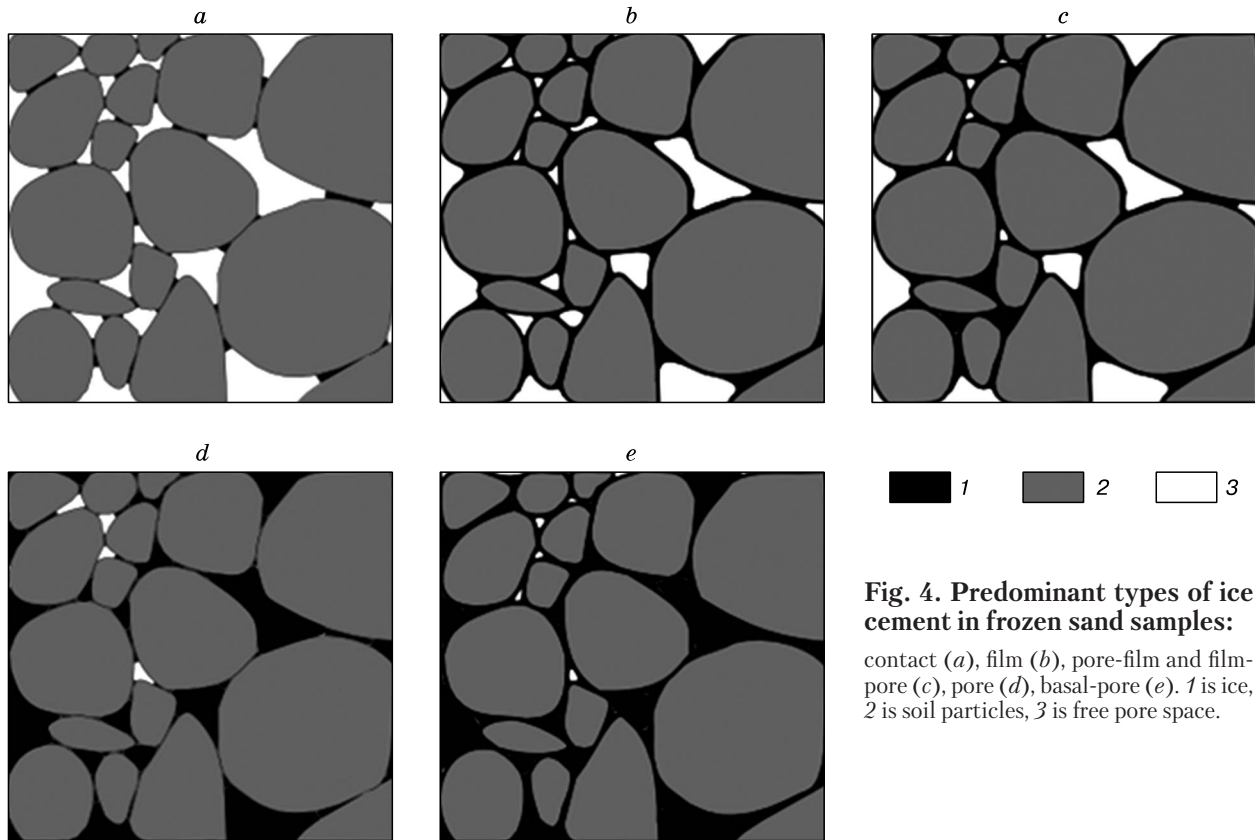


Fig. 4. Predominant types of ice cement in frozen sand samples: contact (a), film (b), pore-film and film-pore (c), pore (d), basal-pore (e). 1 is ice, 2 is soil particles, 3 is free pore space.

1988]. In the absence of cementing ice in dry sand, all pores and voids along grain boundaries are open, and the permeability remains almost invariable as the temperature rises from -5 to 20 °C. Ice filling 30 % of pores is mostly restricted to grain boundaries and cements closely spaced particles along their contacts, while most of pores remain open (Fig. 4). In the case of the contact ice cement, sand samples become obviously less permeable as their ice saturation increases, but no more than ten times. Further saturation increase leads to the formation of film ice cement, and some amount of ice in large pores. The gas permeability curve kinks at ice saturation above 45 %, when pore ice cement becomes prevailing, also in large pores, and the film and pore ice cement blocks most of grain boundaries and markedly reduces the volume of open pores. Increasing pore ice and forming basal ice cement confine the gas component and block its flow through pores thus reducing gas permeability to the minimum. The water saturation dependence of gas permeability in unfrozen samples likewise has two segments of slow (less than ten times) and rapid decrease at saturations below and above 50–55 %, respectively (Fig. 5). Thus, unfrozen samples likewise have a critical saturation value (50–55 %) at which gas permeability reduction accelerates.

Some difference in the critical water saturation values between fine and silty sand may be due to different structure, size, and volume fraction of the pore space. These parameters control redistribution of pore moisture in soils upon thawing and the action of gas pressure gradient across the sample. However, this issue requires special research which is beyond the scope of the reported study.

The effective permeability ratio of frozen and thawed sand samples (K_f/K_{th}) varies with moisture content (Fig. 6): decreases from 1 to 0.1 at less than 10 % of moisture but drops for several orders of magnitude as its content exceeds 10 % (Fig. 6). This behavior may result from difference in the physical properties of frozen and unfrozen fluids and in the mechanisms of pore filling upon phase change.

The reduction in air permeability in frozen soil relative to that in unfrozen soil, at the same degree of saturation, may result from internal redistribution of water [Seyfried and Murdock, 1997]: freezing begins with water in large pores and then draws water from small pores. Thus the larger and more conductive pores freezing first become filled with ice at the account of smaller and less conductive pores which supply moisture in the absence of external in-

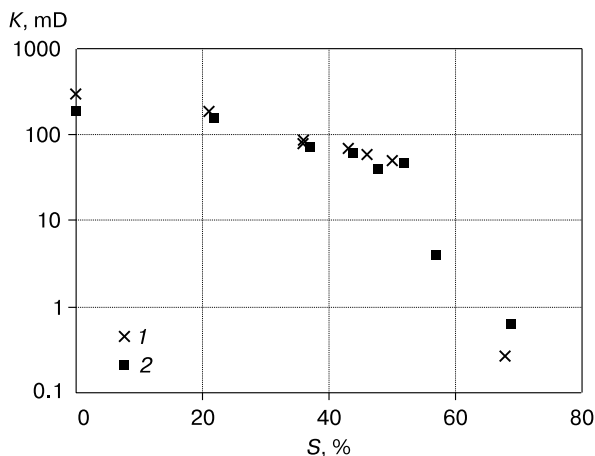


Fig. 5. Saturation (S) dependence of effective gas permeability (K) of unfrozen samples of fine (1) and silty (2) sand.

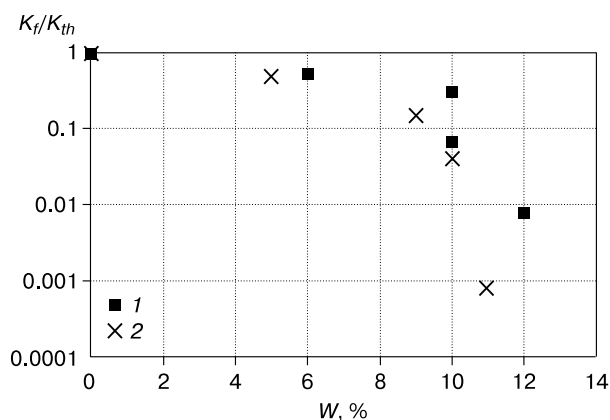


Fig. 6. Water content (W) dependence of effective gas permeabilities (K) of frozen and thawed samples (K_f/K_{th}).

1 – fine sand; 2 – silty sand.

put. Ice blockage by this mechanism more strongly affects the air permeability of soils than mere expansion of water when frozen. Air permeability in thawing soils may be expected to increase significantly as some water will leave large pores and return to small pores.

CONCLUSIONS

The reported experiments showed how the moisture contents in frozen and unfrozen sandy samples influence their permeability to gas.

The behavior of gas permeability of frozen fine sand and silty sand samples depends on ice saturation (S): the permeability reduction is within ten times as the saturation increases from 0 to 40 % and reaches several orders of magnitude at $S = 40\text{--}50\%$; the gas permeability of samples with saturation about 50 % is as low as a few mD, while that at saturation more than 60 % reduces to below the instrumental resolution (0.01 mD).

As the experiments demonstrate, thawing leads to higher gas permeability, and the difference increases proportionally to saturation (S), being less than ten-fold at $S = 40\%$ and 3–5 orders of magnitude at $S = 40\text{--}80\%$.

The behavior of the saturation dependence of gas permeability in frozen and unfrozen sandy soils is largely controlled by their microstructure, and especially by the type of ice cement which, in turn, depends on moisture content.

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