Experimental evaluation of changes in permeability of frozen sediments at thawing

Evgeny Chuvilin
Department of Geology, Moscow State University, Moscow, Russia
Skolkovo institute of science and technology (Skoltech),
Skolkovo village, Odintsovsky district, Moscow region, Russia
Sergey Grebenkin
Department of Geology, Moscow State University, Moscow, Russia

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ABSTRACT
Testing of water saturated samples of the fine-grained sediments conducted in a special device under a wide range of temperatures (-15 to +30°C) and pressures (up to 100 bar) allowed to evaluate their gas permeability. It has been found that increase in permeability during thawing of frozen samples depends on moisture content (ice content) and it is associated with the change in the structure of the pore space, the possible redistribution of water in the pores and volume changes during phase transitions of ice – water. At the ice saturation below 40% the difference of values of the gas permeability of frozen and thawed samples was less than one order of magnitude. At an ice saturation more 40% the difference in permeability was more than 3 orders of magnitude. It is noted that the sediment samples are almost impermeable at the high power of pore filling (over 50% for the frozen and 70% for the thawed).

RÉSUMÉ
L'analyse des échantillons de sédiments fins saturés en eau a été réalisée en utilisant un dispositif spécial afin d'obtenir une large gamme de températures, de +20 à -30 °C, et des pressions atteignant les 100 bars. Les résultats obtenus démontrent que lors du dégel des échantillons, l'augmentation de leur perméabilité dépend de la teneur en humidité (glace). Ceci s'explique par les changements dans la structure des espaces interstitiels, par une possible redistribution de l'eau interstitielle et par un changement du volume lors de la transition de phase eau-glace. Ainsi, lorsque la saturation en glace est inférieure à 40 %, la perméabilité aux gaz entre les échantillons gelés et dégelés diminue d'un ordre de grandeur. Lors d'une saturation en glace de plus de 40 %, la différence de perméabilité est de plus de trois ordres de grandeur. Il est à noter que les échantillons de sédiments sont presque imperméables à la puissance élevée de remplissage des pores (plus de 50 % pour les échantillons gelés et 70 % pour les dégelés).

1 INTRODUCTION

Study of the gas permeability of frozen and thawed sediments is of great interest considering the emissions of natural gas in the Arctic. It is also important when analyzing results of the geochemical surveys in the permafrost regions for hydrocarbon exploration. It is considered that frozen sediments are largely impermeable to gas. Specific studies on the gas permeability of frozen sediments are very limited. However, some geochemical studies of subsurface horizons of the permafrost sediments indicate significant migration of hydrocarbons through permafrost (Glotov, et al., 1985, Glotov., 2005, Are, 1998). Data on emissions of natural gas, primarily methane, appears in recent years for the Arctic onshore and offshore areas underlain by permafrost. There is a need for experimental studies to determine the permeability of frozen and thawed sediments (Shakhova et al., 2010).

Review of the published experimental data on permeability of water saturated sediments at freezing indicates that the sediment permeability decreases at freezing and it can be determined experimentally. One of the first researchers who identified the gas permeability of frozen sediments were A.A. Ananyan et al., (1972). They experimentally determined that for the same frozen sediment particle size distribution content the permeability coefficient decreases with the increase in moisture content (W) and ice saturation (S_{ice}). For example, for a sample of sand (the sand fraction of 0.1- 0.25 mm) W = 14.3% S_{ice} =65% and the value of permeability coefficient is equal to 121 mD, while at W = 16% and the quantity S_{ice} = 75% it was 0.36 mD.

I.S. Starobinets and R.N. Murogova (1985) found that the permeability of frozen dolomite in two orders of magnitude lower than the air-dry, and an order lower than non-frozen at 67% water saturation.

T.J. Kneafsey et al., (2008) conducted experiments to study the permeability of frozen and hydrate saturated sand. Initially samples had water saturation (S_w) of 42% and relative permeability 0.5 – 0.6 u.f. After freezing permeability decreased by more than twofold and reached 0.2 u.f. At a lower initial water saturation of sample the relative permeability during freezing decreased slightly from the initial 0.68 u.f. to 0.62 u.f. after freezing. Reduced permeability several times (up to 10) was recorded during the freezing artificially hydrate sandy and sandy loam samples (Chuvilin et al., 2014).

Chinese researchers (P. Wang et al., 2014) conducted experiments to study the methane permeability of frozen silty sand. Samples with porosity of 40% were used in experiments. These investigation have been presented that the permeability of soil samples with ice saturation to 45-50% decreases to the order of magnitude, with a further increase of ice saturation gas permeability reduced by several orders of magnitude, when of ice saturation reached 80% frozen sand sample is almost impenetrable.

However, despite the individual experimental data obtained, the gas permeability of frozen and thawed sediments remains very poorly studied. The experimental data on the effect of thawing and ice content on the sediment permeability were not found in the literature.

In this context, the aim of this work is to study the dependence of the sediment gas permeability on the degree of soil pore infill by ice and water.

2 METHOD OF GAS PERMEABILITY STUDY OF CORE SAMPLES

Research methods of the gas permeability during freezing and thawing are based on the study of gas filtration through the soil sample in a special experimental equipment, which provide necessary thermobaric conditions for artificial freezing and thawing of the sediments. This technique includes preparation of a sediment sample with required moisture content, its gas (nitrogen) saturation in a special holder, setting up thermobaric conditions for thawing or freezing, testing the gas permeability at various gas pressures and temperatures and processing of experimental data (Chuvilin et al., 2014).

Equipment, developed by "Ecogeosprom" Ltd. was used to study the gas permeability in the course of sediment freezing and thawing. This device allows to set up thermobaric conditions in soil samples in a wide range of temperatures and pressure: temperatures from -15 to +30°C and pressure up to 100 bar (Chuvilin et al., 2013).

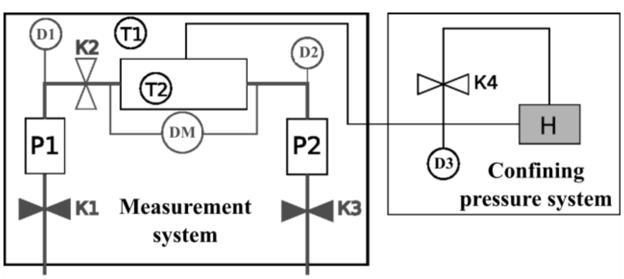

Figure 1. Scheme of experimental equipment. K1-K3 - diaphragm valves of the gas; K4 - ball valve of the hydraulic system; P1, P2 - Receivers gas supply system; D1-D3 - pressure sensors; DM - differential pressure sensor; H - Hydraulic pump with oil tank; T1, T2 - temperature sensors.

The method of experimental studies of gas permeability of frozen sediment samples at thawing included the following steps: cutting pattern with a diameter of 3 cm from frozen core, the measurement of its size and weight, the location in holder, pressurization and evacuation of the vacuum chamber with the sample, gas filling the chamber and measurements of gas permeability in frozen and thawed state.

Sediment samples with disturbed structure were tested (Tab. 1).

Table 1. Characteristics of tested sediments.

Lithology	Particle size distribution, %			Mineral composition, %	Salinity, %
	1-0.25 mm	0.25-0.01 mm	0.1-0.05 mm		
Fine sand	29.3	62.3	8,4	quartz > 90	0.05
Silty sand	14.5	57.8	27,7	quartz- 74.1 Plagioclase (albite) – 12.8 K-feldspar (orthoclase) — 10.0	0.13

Length of samples is of 25 - 35 mm and diameter - 30 mm. The moisture content of samples fs-1, ss-1 was 0-18%, the porosity coefficient of samples was 0.60 – 0.75 u.f. (Tab. 2).

Table 2. Characteristics of tested sediment samples.

Name	Sediment type	Length, mm	Diameter, mm	Moisture content, %	Porosity coefficient, u.f.
fs-1	Fine sand	30	30	18	0.75
fs-2		30	30	13	0.75
fs-3		24	30	10	0.75
fs-4		34	30	6	0.75
fs-5		29	30	0	0.75
ss-1	Silty sand	30	30	16	0.60
ss-2		30	30	12	0.60
ss-3		28	30	9	0.60
ss-4		29	30	5	0.60
ss-5		28	30	0	0.60

Measurements of permeability of sample sediments were carried out at each stage of cooling and heating, and temperature and pressure recording was carried out during the whole experiment. At the beginning of the test, the permeability of the soil sample was measured after its saturation with gas N_2 at the temperature of 5°C. The permeability of the sample was determined after it thawed reaching room temperature of 20°C.

The calculation of the effective permeability of the sample in the device holder was based on the analysis of the pressure drop in the receivers with the known gas volumes (Chuvilin et al., 2014).

In the course of solving the differential equation of mass transfer through the sample under the action of the pressure gradient, the following formula was used to calculate the effective permeability (Chuvilin et .al., 2013).

$$k = \frac{2 * \eta * L * V_1}{S * p_{1_0} * (p_1^2 - p_2^2) / p_1^2} * \frac{p_{1_0} - p_{1_k}}{p_1 * t_1} \quad [1]$$

When η — dynamic viscosity of the gas
L — length of sample
S — cross-sectional area of the sample
V_1 — value of receiver
p_1 — pressure upstream of the sample at time t_1
p_2 — pressure downstream of the sample at time t_1
p_{1o} — pressure upstream of the sample at start
p_{1k} — pressure upstream of the sample at finish

To assess the accuracy of determination of the permeability of the samples by the method described above, device calibration was carried out for four reference ceramic samples. Values of the coefficients of the absolute permeability were known. The calibration results showed that the measurement error does not exceed 15%.

3 RESULTS

In all experiments, the initial determination of the gas permeability of sediment samples was conducted in frozen state at a temperature of-5°C. The test results are presented in Table 3,

The frozen samples were saturated with nitrogen gas at a temperature of -5°C, and then the permeability measured.

The experimental data shows that the initial permeability of frozen samples with high ice saturation (over 40%) was very small - below the detection limit (0.01 mD) (Tab.3).

Table 3. Permeability of frozen sediment samples.

Name	Sediment type	Moisture content, %	Ice saturation, %	Average coefficient of effective gas permeability K, mD
fs-1	Fine sand	18	74	<0.01
fs-2		13	51	<0.01
fs-3		10	39	25.17
fs-4		6	23	94.47
fs-5		0	0	288.5
ss-1	Silty sand	16	80	<0.01
ss-2		12	57	<0.01
ss-3		9	40	10.36
ss-4		5	24	74.3
ss-5		0	0	177.7

After evaluation of the gas permeability of gas-saturated soil samples at temperatures below freezing had been completed, the core holder with gas-saturated samples was heated to a temperature of +20 °C for 6 hours. At the same time increase in pressure was recorded due to the thermal expansion of the oil. After the temperature and pressure stabilized, filtration tests on thawed samples have been conducted (Tab. 4).

Table 4. Permeability of thawed samples.

Name	Sediment type	Moisture content, %	Water saturation, %	Average coefficient of effective gas permeability K, mD
fs-1	Fine sand	18	68	0.27
fs-2		13	46	59.97
fs-3		10	36	84.8
fs-4		6	21	184.1
fs-5		0	0	291.6
ss-1	Silty sand	16	69	0.6
ss-2		12	52	45.0
ss-3		9	37	69.9
ss-4		5	22	151.3
ss-5		0	0	181.4

It was found that permeability sample fs-1 of frozen fine sand with the ice saturation 74% after thawing have been 0.27 mD (in frozen state permeability was below the detection limit) (Fig. 2).

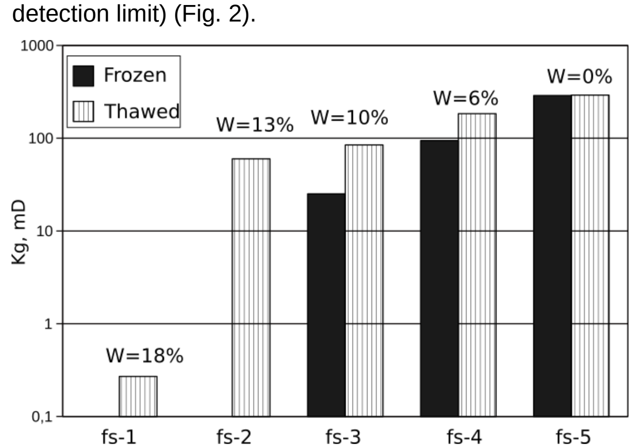

Figure 2. Gas permeability of fine sand samples (K_g) in the frozen and thawed states.

Permeability of sample fs-2 with ice saturation 51% after thawing have been 59.97 mD (Fig. 2). The permeability of the sample fs-3 with ice content 39% after thawing increased by 3 to 4 times - from 25.17 to 84.8 mD (Fig. 2). The permeability of the sample fs-4 with ice content 23% have been increased from 94.47 to 184.1 mD (Fig. 2). The permeability of the dry sample fs-5 at temperatures above and below freezing had approximately the same value – 288.5 and 291.6 mD, respectively (Tab. 3, 4).

Thus, for the fine-grained sand samples with moisture content over 10% permeability increased by more than an order of magnitude, for samples with moisture content of 5-10% - in 2 – 3.5 times, for samples with moisture content below 5% - less than 2 times.

For silty sand, in general, the results were similar. At moisture content more than 10%, the permeability increased by more than an order of magnitude, at a water content of 5-10% - 2 – 6.7 times, at a water content of less than 5% - less than 2 times. The increase in permeability during thawing is associated with the change in the structure of the pore space, the possible redistribution of water in the pores, volume changes during the ice-water phase transitions.

Changes in permeability of silty sand samples depending on ice (water) saturation were similar pattern. For example, the gas permeability of sample ss-1 with ice saturation of 80%, was found to be 0.6 mD after thawing. The permeability of sample ss-2 with ice saturation at 57% was found to be 45.0 mD (Fig. 3). The value of the gas permeability of the sample ss-3 with ice saturation 40% increased in 6 to7 times (from 10.36 to 69.9 md) (Fig. 3). The permeability of sample ss-4 with ice saturation 24% increased from 74.3 to 151.3 md (Fig. 3). The permeability of the dry sample ss-5 at positive and negative temperatures had approximately the same values – 177.7 and 181.4 mD, respectively (Fig. 3).

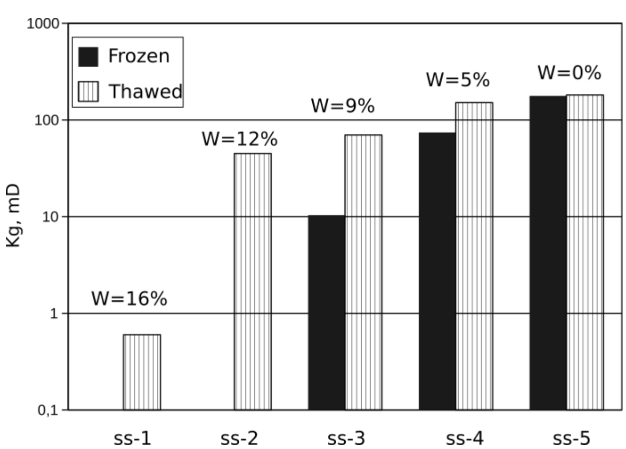

Figure 3. Gas permeability (Kg) of silty sand samples in the frozen and thawed states.

Relationship between permeability and ice (water) saturation of silty sand in the thawed and frozen state (Fig. 3) is similar to the one in fine-grained sand.

Divergence of permeability values increases with the powers of pore filling at the same initial permeability (Fig. 2). This factor can be explained by the varying degrees of mobility water and ice components. Redistribution of water in soil pores is possible, when gas moves through water-saturated pore space. Whereas, change of ice configuration is impossible when gas moves through the frozen soil sample. Thus, active porosity of a thawed sample is higher than active porosity of a frozen sample at the same powers of pore filling.

The nature of depending the gas permeability from ice saturation caused the microstructure of frozen ground and dependence of ice-cement type from the moisture content. Depending on the initial moisture content of the sand rocks formed contact, film, porous and basal types of ice-cement (fig.5) (Yershov, 1998).

4 DISCUSSION

Our experimental data for estimation the relative permeability of the ice saturated sandy rocks generally in good agreement with other author’s data. Some deviations are apparently caused by the different composition and structure of the frozen sand samples (Fig. 4).

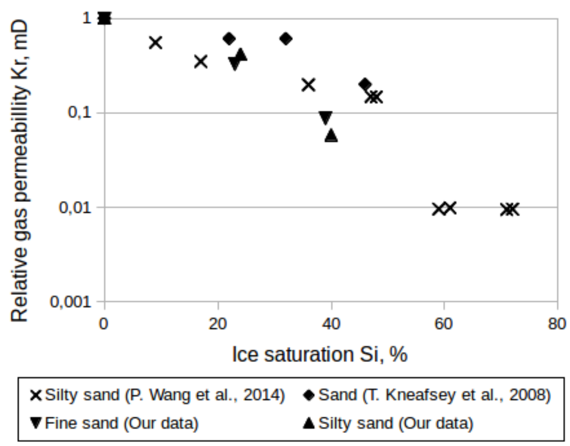

Figure 4. Relationship between the relative permeability (K_r) of frozen sand samples and ice saturation (S_i).

Comparing of the experimental data presented in Figure 4 makes it possible to trace the general dependence of the relative permeability of sandy soils from ice saturation.

When analyzing a diagram below, two sections can be identified. The first section has powers of pore filling up to 40-50% and is characterized by logarithmic decrease relative permeability with increase in water and ice saturation. The second section is characterized by a sharp decline in the relative permeability with a further increase in water and ice saturation.

For frozen samples in the first section, decline in the relative permeability is evident at ice saturation 0 - 40% not more than one order (Fig. 4). In the second section (Si > 40%), the decline in the relative permeability is greater than two orders of magnitude (Fig. 4).

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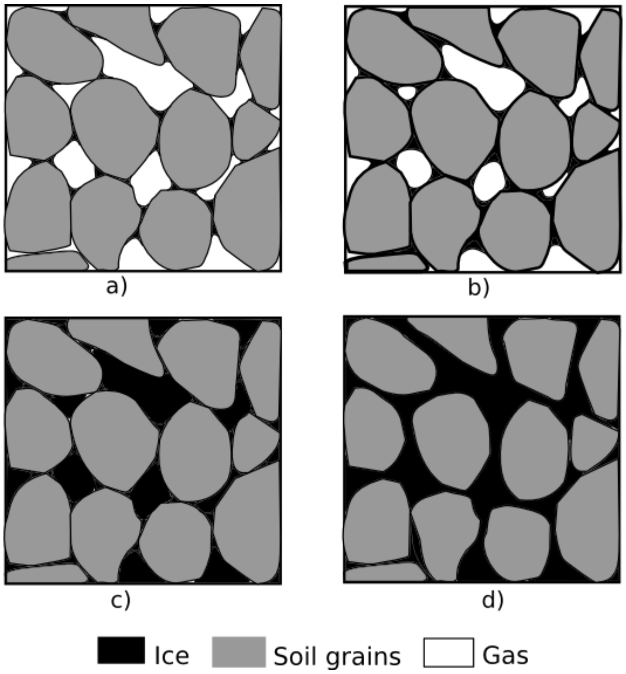

Figure 5. General types of ice-cement in frozen sand samples: a) contact; b) film; c) porous; d) basal.

In the case of gas filtration through the dry sand sample all channels and pores are open. At low ice saturation up to 30% occurs contact ice-cement, which is located in the contact area of particles and blocked only the individual throats, but pores mostly remain open. At predominance of contact type ice-cement in sand sample gas permeability linear decrease with increasing of ice saturation. And reducing of gas permeability was not more than an order of magnitude. A sharp bend on the curve occurs when the gas permeability of the ice saturation greater than 45% when the begins to dominate the film ice-cement, which blocks most of the channels, and a significant decrease in the volume of open pores. With the predominance of pore ice-cement and emergence basal ice-cement (fig. 5c, d) in the sample most pores almost completely filled by ice. At the same time there are frost heaving areas of rocks with the presence of basal ice cement. This greatly reduces the filtration of gas through the sample and results in an essentially complete loss of permeability.

Microstructure of impermeable fs-1 sample characterized by typical frame texture, which is composed mainly sand particle size of 0.1-0.3 mm. Small and fine sand fraction of frame particle is medium rounded, generally isometric or slightly elongated with irregular shape. Ice component is presented in the form of ice-cement of pore type and basal.

Microstructure of impermeable ss-1 sample characterized by frame texture, which is mainly composed of sand particles of 0.05-0.15 mm. Sand grains are well and medium rounded, isometric or slightly elongated with irregular shape. There is some differentiation of sandy material, often smaller particles are arranged around the larger. Ice component is presented in the form of ice-cement of pore type and basal.

For thawed samples in the first section decline of the relative permeability at water saturation 0 — 50…55% is not more than one order (Fig. 6). In the second section (S_w > 50%), the decline in the relative permeability is greater than 2 orders of magnitude (Fig.6).

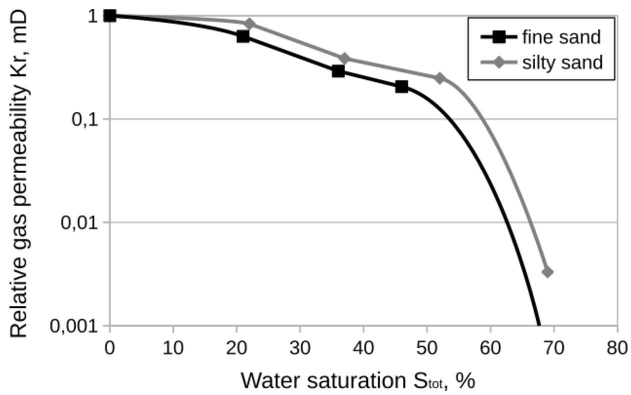

Figure 6. Relationship between permeability (K_r) of thawed sand samples and water saturation (S_w).

Thus, we can identify for frozen and thawed sediment samples some critical value of the powers of pore filling above which gas permeability of the sediment samples extremely reduced.

For frozen samples of fine and silty sand critical saturation is equally due to similar mechanisms of ice formation in the pore space, but with an increase in dispersion of sediments critical value of ice saturation can vary, but this requires further investigation.

Some difference in the critical water saturation value of fine and silty sand may be associated with different pore sizes and porosity, which in turn affects the ability of water to redistribute in the pore space of the sample during thawing and at the pressure gradient of the gas.

CONCLUSIONS

Thus, the gas permeability of frozen sediments was found lower than thawed sediments at the same powers of pore filling. At sediment thawing, the increase in permeability attenuates with decrease in the powers of pore filling. At the powers of pore filling less than 20%, the gas permeability increased by twofold. At powers of pore filling 20-40% permeability increased by 2 to7 times.
At more 40% - permeability increased by several orders of magnitude. It is noted that the sediment samples are almost impermeable at the high power of pore filling (over 50% for the frozen and 70% for the thawed). However the sediment samples are almost impermeable at the high power of pore filling (over 50% for the frozen and 70% for the thawed).

As the result of study of permeability of sediments at varying water and ice content was found that the gas permeability of the sediment samples was strongly dependent on the phase state of the pore water. In the thawed state, the gas permeability of the samples was significantly higher than in the frozen state at the same powers of pore filling.

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