

PROPERTIES OF FROZEN GROUND AND ICE

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**THE ISOTOPIC COMPOSITION OF DIFFERENT WATER CATEGORIES
IN FINE-DISPersed GROUND**

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Results obtained from the laboratory research of the isotope composition of water occluded in fine-dispersed ground revealed that the water-soil interaction processes are accompanied by ^{16}O , ^{18}O , H, ^2H -isotope fractionation depending on soil structure and properties, with the isotopic composition of bound water being lighter, as compared to free water.

Stable isotopes of water, isotope fractionation, water categories, segregated ice

INTRODUCTION AND PROBLEM STATEMENT

A considerable interest currently addressed to the isotopic composition of ground ices accounts primarily for the advantageous applications of water isotopes from ice cores (stable-isotopes information) as proxy data in paleoclimate reconstructions. Based on the concept of ice bodies forming from solid precipitation, their isotopic composition is correlated to the climate conditions of each period, accordingly [Ekakin, 2016], while the processes of ice formation in soils and its preservation conditions appear to be more complicated. However, a significant progress in ice-wedge research has been achieved by Yu.K. Vasil'chuk, which is amply discussed in his monographs [Vasil'chuk, 1992, 2006, 2011]. Noteworthy is that the successful results were obtained owing to a well-established mechanism of ice wedge growth primarily due to contributions from snow melt water. The isotopic compositions fairly profoundly studied from other types of massive ground ice were discussed in [Arkhangelov et al., 1988; Vasil'chuk, 1992; Mikhalev, 1996; Michel, 2011; Dereviagin et al., 2013, 2016; Sologatin, 2013; Streletskaia et al., 2013]. However, the mechanisms of their formation are either not always clear (e.g. tabular ground ice) or these may be comprehensible but lacking the data on the isotopic composition of water forming them (segregated ice). In addition, the principles applied to their isotope-data interpretation are basically similar to ice-wedge research. We should bear it in mind, though, that this approach is admittedly not always adequate, since ice wedge growth may involve different categories of water, in particular, those featured by molecular fingerprinting reflecting specific bonds between water and soil matrix.

BACKGROUND INFORMATION

A significant heterogeneity of energy inherent in water held within soil pores prompted allocation of categories of soil water that differ in nature, formation conditions, structure, properties, etc. [Trofimov et al., 2005]. Almost a century-long history of study of this phenomenon has resulted in diverse classifications of soil water, both simple and essentially detailed. Nevertheless, soil water basically belongs to two main categories: free and bound, according to the most traditional views [Zlochevskaya and Korolev, 1988]. The latter includes water located in close proximity to the mineral surface of soil particles, which involves interactions between them, affecting thereby both the structure and properties of water. In permafrost studies, a particular interest in properties of bound water arose due to its major role in the mechanism of texture-forming ice aggradation (ice-cement and segregated ice) with water migration to the freezing front and subsequent ice segregation as part thereof [Dostovalov and Kudryavtsev, 1967; Ershov, 2002].

Some valuable data have been accumulated from numerous studies on isotopic composition of texture ices, however their characteristics are interpreted in different ways. A.Yu. Dereviagin and colleagues [Dereviagin et al., 2013, 2016] explain the nature of the isotopic composition of texture ice of the Ice Complex occurring on the coast by its aggradation from meteoric precipitation as primordial water having various isotopic compositions for different climatic epochs, which can be accepted, given the syndeposition of the Ice Complex sequences. This contradicts, however, the inferences made by F. Michel claiming the changes occurring at a great depth in the isotopic

composition of segregated ice in the epigenetically frozen lacustrine sediments to be associated with the Holocene Climate Optimum (HCO), whose time span coincides with the age of lacustrine sediments at locations where this ice was investigated. In this case, ice segregation suggests bound water migration, but this category of water was overlooked by this author.

Proceeding from theoretical understanding, the isotopic composition of bound water is controlled by interactions between soil water and organo-mineral skeleton, mass-transfer and phase changes. The properties of bound water being largely affected by the ions, atoms and molecules forming the surface soil skeleton differ from the properties of free water, allowing for the presence of disparate molecules (among them, heavy isotopes of hydrogen and oxygen) in its composition. A literature review dedicated to studying water signatures associated with dispersive soils provided in the monograph authored by Yu. Fedorov [1999] has shown that most characteristics include the isotopic composition of water incorporated into the crystal structure of various minerals. However, such water participates neither in migration, nor in ice segregation, whereas studies of the isotopic water composition of water migrating while freezing are extremely sparse [Konishchev *et al.*, 2011]. At the same time, the frozen strata are marked by appreciably large schlieren, or ice lenses (which are found isotopically lighter, than ice in the host sediments) within the “belt-like” cryostructure developing during water migration. This difference ranged from 0.9 to 3.2 ‰ for $\delta^{18}\text{O}$, and from 7.8 to 28 ‰ for $\delta^2\text{H}$ [Dereviagin *et al.*, 2013]. The experimental studies of frozen soils have also shown that water migrating in dispersed soils may be isotopically lighter, than initial water [Konishchev *et al.*, 2014], but these data altogether qualified as sporadic, require clarification.

Theoretically, bonds between soil water and surface of soil particles are determined both by the van der Waals forces (the induced dipole-dipole interaction between atoms and molecules on the surface of these particles) and by the appearance of hydrogen (or ion) bonding between uncompensated charges on their surface, and by H^+ , OH^- , H_3O^+ , ${}^2\text{H}^+$, O^2H^- , H_2 , ${}^2\text{HO}^+$ ions formed during the water dissociation. This allows suggesting both abundant and reduced abundancies of heavy isotopes [Golubev, 2000; Konishchev *et al.*, 2014]. The energy of van Der Waals interactions between isotopically heavy water molecules and atoms and molecules on the surface of mineral particles is found to be 10 % higher, than in the case with isotopically light molecules. Since the dipole-dipole attraction between water molecules is at least an order of magnitude greater, than their interaction with atoms and molecules on the surface of mineral particles, the dominant abundance of isotopically heavy molecules should occur in the volume of free or weakly bound water. However, when consider-

ing soil water in broad sense and given relatively small fraction of bound water, this does not affect significantly the isotopically heavy and light molecules redistribution. Selective interactions between uncompensated charges on the surface of soil particles and H^+ , OH^- , H_3O^+ , ${}^2\text{H}^+$, O^2H^- , H_2 , ${}^2\text{HO}^+$ ions forming during water dissociation, and resulting changes in the isotopic composition of bound and free water can be generally characterized by a differentiated degree of the isotopically heavy and light molecules dissociation and by mobility of the formed ions and associations. Bearing in mind that the ionic product of heavy water is almost an order of magnitude less than that of ordinary water and mobility of its ions and ion associations is also many times lower, the interactions between ions and associations of light water and uncompensated charges should occur much more often and, accordingly, their content in bound water should be higher. Nevertheless, this aspect of the isotopically heavy and light water molecules redistribution proceeding in the course of water – soil interactions is interpreted as fairly small-scaled due to a small fraction of bound water.

The provided data and insights do not offer an unambiguous answer to the question of isotopic compositions forming from different categories of soil water, which ultimately determined the authors' intent to investigate experimentally the consequences of the occurring processes.

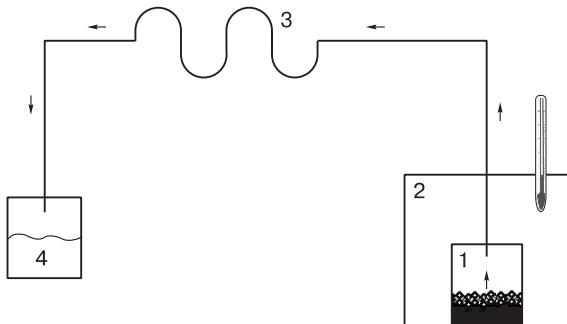
MATERIALS AND METHODS

The model soils used in the experimental determinations of the isotope composition in different types of soil water included: kaolinite (from the Prosvyanovskoe hydrocarbon field), clay-loam (the Igarka subsurface) and yedoma clay-loam (the Duvanny Yar section (Kolyma river)), as well as samples of dispersive soils from Yamal and Gydan regions; their physicochemical properties are discussed in the literature [Pchelintsev, 1964; Ershov, 1979; Vasil'chuk, 2005; Badu, 2018].

The isotopic composition of water and ice was determined and analyzed at the Laboratory of Isotope Geochemistry at the Faculty of Geography, Moscow State University (MSU), using the Delta V mass spectrometer with standard GAZ-bench option. The measurements were calibrated against the international standards (V-SMOW, SLAP), and MSU's own laboratory standard – Garabashi glacier snow ($\delta^{18}\text{O} = -15.60 \text{ ‰}$, $\delta^2\text{H} = -110.0 \text{ ‰}$). The determinations (error: $\pm 0.6 \text{ ‰}$ for $\delta^2\text{H}$ and $\pm 0.1 \text{ ‰}$ for $\delta^{18}\text{O}$) were performed by N.A. Budantseva and Yu.N. Chizhova under the guidance of Yu.K. Vasil'chuk.

EXPERIMENTS AND RESULTS

In keeping with the experiment's objective, it is imperative that soil water is squeezed from soil sam-

**Fig. 1. A layout of the experimental unit.**

1 – container with squeezed soil; 2 – thermostat; 3 – condenser; 4 – container with condensed vapor. Arrows show the vapor directions.

ples to be differentiated by the degree of bonding with the surface of constituent skeleton particles. Regardless of the multitude of different methods currently available for determining the amount of bound water in soil, the authors after many failed attempts to find a coherent method in the literature enabling the separation of one category of water from another, have come up with a simple methodical procedure described below. According to the accepted technique, soils were sampled with natural moisture, and the experimental samples were additionally saturated to full moisture capacity with water having isotopic composition specified for each experiment. After that the soil samples were placed in a perforated metal cup sized 48 mm in diameter and 100 mm in height.

Then a screw press was applied with pressure of about 10 kg/cm² to squeeze the water out of the samples with residual moisture content approximately equal to the maximum molecular moisture capacity. The squeezed out portion of water conditionally designated as "free" was intended for measuring the isotopic composition. The water remaining in the sample was extracted by evaporation at a temperature of 150 °C, for which the sample was placed in a looped, leak-tight system consisting of two evaporator chambers connected via a hose (Fig. 1).

The container with the test sample placed in the evaporator chamber was sent to the thermostat to evaporate the water, with the output vapor passing through a spiral condenser tube to another chamber (outside the cabinet), where the vapor was condensed, to be subsequently taken for the isotope analysis. The complete drying of the samples took about 12 hours. At this, dividing the generally recognized separation of soil water into two volumes labeled as "free" and "bound" water (here and elsewhere without the quotation marks) is rather conditional. As such, the system allowed to obtain the amounts of water required for the isotope analysis.

DISCUSSION OF RESULTS

The results obtained are represented in the tables and figures. The experimental data on dry powder kaolin (the samples were saturated with Moscow tap water for the analysis) are illustrated by Table 1 and Fig. 2, a. Determinations of the isotopic composition of initial water were performed in the first five experiments, but afterwards only test samples were measured, inasmuch as no significant changes were observed in the isotopic composition of the initial water. The results revealed a significant differentiation of water types with respect to the isotopic composition in the course of the experiment, with free water being "heavier" and bound water "lighter", than initial water.

A comparison with the global meteoric water line (GMWL) shows that the values of the isotopic composition plots for initial and free waters are located near GMWL, however, most of other plotted values are found to be localized either below (for free water) or above (for bound water) the GMW line.

Table 1. Concentrations of heavy isotopes in kaolin samples saturated with tap water

No.	Experiment No.	Water category	$\delta^{18}\text{O}$, ‰	$\delta^2\text{H}$, ‰	d, ‰	W_s , %	W_a , %
1	1	Initial	-10.54	-74.1	10.22		
2		Free	-9.58	-72.7	3.94	70	38.5
3		Bound	-15.45	-92.7	30.90		
4	2	Initial	-10.54	-68.5	15.82		
5		Free	-9.53	-67.1	9.14	70	33.0
6		Bound	-13.76	-89.8	20.28		
7	3	Initial	-10.59	-71.3	13.42		
8		Free	-10.02	-66.4	13.76	70	37.1
9		Bound	-13.48	-73.0	34.84		
10	4	Initial	-10.01	-70.6	9.48		
11		Free	-8.02	-59.3	4.86	70	38.1
12		Bound	-13.56	-87.7	20.78		
13	5	Initial	-10.54	-68.5	15.82		
14		Free	-9.45	-63.2	12.40	70	43.2
15		Bound	-12.89	-90.9	12.22		
16		Initial	-11.53	-80.3	11.40		
17	6	Free	-9.72	-77.5	0.26	70	40.2
18		Bound	-16.26	-113.9	16.18		
19	7	Free	-9.46	-77.8	-2.12	70	29.1
20		Bound	-11.39	-91.1	0.02		
21	8	Free	-9.94	-78.2	1.32	70	35.5
22		Bound	-14.78	-103.1	15.40		
23	9	Free	-9.84	-79.2	-0.48	70	40.2
24		Bound	-12.80	-87.3	15.10		
25	10	Free	-10.25	-78.2	3.80	70	38.2
26		Bound	-10.97	-84.8	2.96		

Note. W_s – set moisture content, %; W_a – moisture content of sample after squeezing, %; d – deuterium excess, ‰.

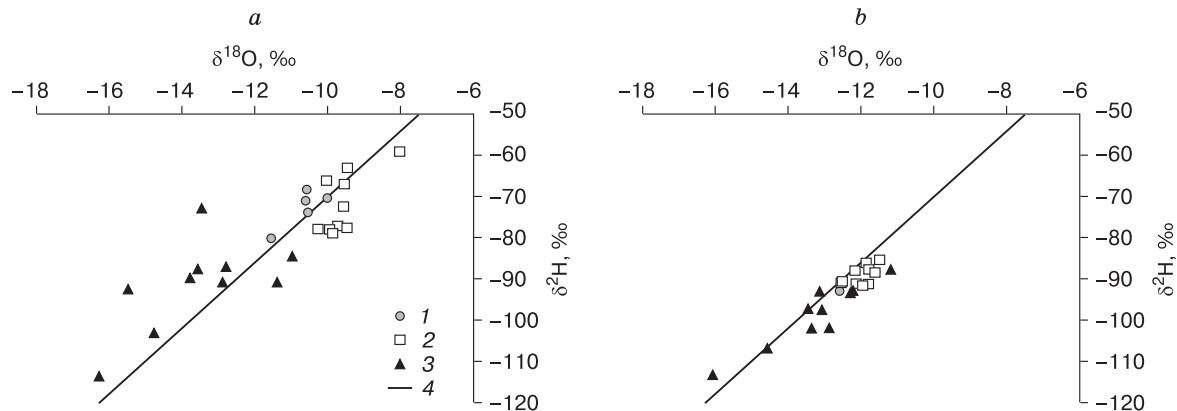


Fig. 2. A ratio between $\delta^{18}\text{O}$ and $\delta^2\text{H}$ isotopes values for free and bound water in the kaolin samples saturated with tap water (a) and distilled water (b).

1 – initial water; 2 – free water; 3 – bound water; 4 – GMWL.

The experiment data on the same type kaolin, whose samples were saturated with distilled water and also isotopically differentiated, with a few exceptions, into a “heavier” (free water) and “lighter” (bound water) are presented in Table 2 and Fig. 2, b. The experiment on samples saturating with distilled water showed that bound water is isotopically significantly “lighter” than free water. However, the distribution of isotopic composition values relative to GMWL is less clear than in the first experiment,

which most likely indicates that the initial water is characterized by a changed isotopic composition (compared to tap water) resulting from the process of distillation.

Results of the experiments with powdered test samples of yedoma clays and Igarka lacustrine clays are given in Table 3 and Fig. 3. These also indicate the

Table 3. Concentrations of heavy isotopes in the Yedoma and Igarka clay loams

No.	Experiment No.	Water category	$\delta^{18}\text{O}$, ‰	$\delta^2\text{H}$, ‰	d, ‰	W_s , %	W_a , %
1	1	Initial	-12.55	-92.9	-24.1	70	37.5
2		Free	-12.11	-91.2	5.7		
3		Bound	-16.06	-113.5	15.0		
4	2	Free	-12.51	-91.0	9.08	70	38.9
5		Bound	-11.15	-87.7	1.5		
6	3	Free	-12.16	-88.3	8.98	70	39.9
7		Bound	-13.45	-97.2	10.4		
8	4	Free	-11.85	-86.2	8.6	70	37.9
9		Bound	-12.29	-93.4	4.92		
10	5	Free	-11.79	-91.3	3.02	70	40.1
11		Bound	-14.56	-106.9	9.6		
12	6	Free	-11.77	-87.9	6.26	70	37.9
13		Bound	-12.18	-92.7	4.74		
14	7	Free	-11.88	-91.7	3.34	70	38.1
15		Bound	-13.34	-102.0	4.7		
16	8	Free	-12.49	-90.7	9.22	70	38.9
17		Bound	-13.06	-97.5	6.98		
18	9	Free	-11.48	-85.5	6.34	70	39.7
19		Bound	-13.08	-92.9	11.7		
20	10	Free	-11.60	-88.6	4.2	70	38.7
21		Bound	-12.88	-102.0	1.04		

No.	Experiment No.	Water category	$\delta^{18}\text{O}$, ‰	$\delta^2\text{H}$, ‰	d, ‰	Yedoma clay loam	
						W_s , %	W_a , %
1	1	Initial	-11.63	-79.9	13.14	40	21.3
2		Free	-11.28	-82.0	8.24		
3		Bound	-14.30	—	—		
4	2	Free	-9.12	-79.6	-6.64	40	18.6
5		Bound	-10.90	-91.1	-3.9		
6	3	Free	-9.40	-80.3	-5.1	37	13.1
7		Bound	-14.63	-95.3	21.74		
8	4	Free	-8.79	-73.6	-3.28	37	17.1
9		Bound	-13.32	-99.2	7.36		
10	5	Free	-8.53	-73.3	-5.06	37	18.3
11		Bound	-11.38	-89.7	1.34		
Igarka clay loam							
12	6	Initial	-10.94	-74.0	13.52	65	38.4
13		Free	-9.11	-76.1	-3.22		
14		Bound	-9.78	-81.3	-3.06		
15	7	Free	-8.76	-69.1	0.98	65	41.8
16		Bound	-10.12	-85.9	-4.94		
17	8	Free	-8.93	-73.3	-1.86	65	37.5
18		Bound	-11.30	-87.3	3.10		
19	9	Free	-8.96	-71.9	-0.22	65	38.1
20		Bound	-11.42	-82.4	8.96		
21	10	Free	-9.37	-74.0	0.96	65	37.9
22		Bound	-11.94	-89.7	5.82		

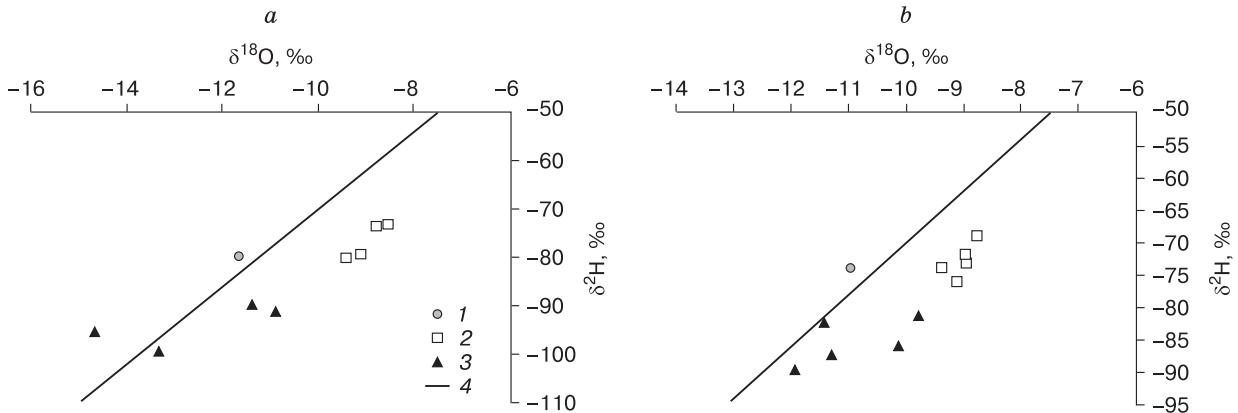


Fig. 3. A ratio between $\delta^{18}\text{O}$ and $\delta^2\text{H}$ values for free and bound water for the samples of yedoma (a) and Igarka (b) clay-loams.

For notations see Fig. 2.

same difference in the isotopic composition of both free and bound water, however, among all the analyzed samples this distinction appears the greatest for yedoma clays (i.e. free water was isotopically “the heaviest” and bound water “the lightest” relative to initial water), against other soils. The experiment results for these soils show a greater spread of the isotopic composition values relative to initial water, with almost all values plotted below GMWL. The revealed distinctions in the isotopic composition between the studied soils are interpreted as stable oxygen and

hydrogen isotope composition of bound water being much “lighter” for yedoma clays, than for the Igarka lacustrine clays. Probably, the reason for this should be sought in some specific properties of yedoma clays.

Table 4 and Fig. 4 provide data on the study of natural soils samples with their intact moisture content, showing the same pattern for isotopic composition of free and bound water, despite the observed different ranges of water isotope values for different soils. In most cases, $\delta^{18}\text{O}$ of bound water is a few ppm

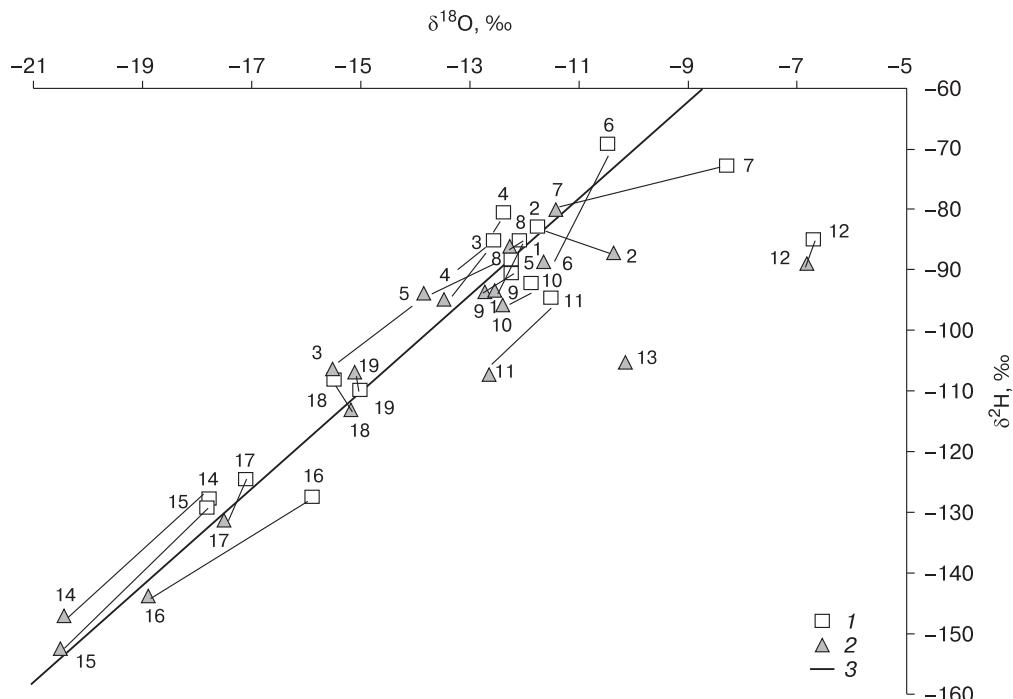


Fig. 4. A ratio between $\delta^{18}\text{O}$ and $\delta^2\text{H}$ isotopes values for free and bound water in the natural soil samples.

1 – free water; 2 – bound water; 3 – GMWL. Numbers next to the symbols are the experiments' numbers.

Table 4. Concentrations of heavy isotopes in natural soil samples

No.	Experiment No.	Water category	$\delta^{18}\text{O}$, ‰	$\delta^2\text{H}$, ‰	d, ‰	W_i , %	W_a , %
<i>Bovanenkovo, Gas Production Facility (GPF)-1, 4-th open pit, depth: 1.8 m, silty clay-loam</i>							
1	1	Free	-12.12	-85.13	11.83	27.7	9.5
2		Bound	-12.56	-93.45	7.03		
3	2	Free	-11.77	-82.97	11.19	33.2	10.1
4		Bound	-10.36	-86.98	-4.10		
5	3	Free	-12.56	-85.13	12.15	29.4	9.2
6		Bound	-15.51	-106.09	17.99		
7	4	Free	-12.39	-80.51	18.61	32.7	9.7
8		Bound	-13.46	-94.69	12.99		
9	5	Free	-12.23	-88.52	9.32	29.7	8.5
10		Bound	-13.84	-93.76	16.96		
<i>Bovanenkovo, BCS (boosting compressor station) GPF-1, depth: 1.0 m, silty sand-loam</i>							
11	6	Free	-10.45	-69.10	14.90	19.9	7.8
12		Bound	-11.64	-88.52	4.60		
13	7	Free	-8.28	-72.80	-6.56	21.7	7.9
14		Bound	-11.42	-79.89	11.47		
15	8	Free	-12.10	-85.13	11.67	24.3	7.3
16		Bound	-12.26	-86.05	12.03		
17	9	Free	-12.25	-90.68	7.32	20.5	6.8
18		Bound	-12.73	-93.45	8.39		
19	10	Free	-11.87	-92.22	2.74	21.0	7.0
20		Bound	-12.38	-95.61	3.43		
21	11	Free	-11.50	-94.38	-2.38	21.8	7.1
22		Bound	-12.66	-106.9	-4.81		
<i>Bovanenkovo, GP-1, Seyakha mound 2.6 m a.s.l., silty clay-loam</i>							
23	12	Free	-6.70	-84.82	-31.22	25.5	8.1
24		Bound	-6.84	-88.83	-34.11		
25	13	Free	-8.81	-	-	24.5	7.9
26		Bound	-10.14	-104.86	-23.74		
<i>Gydan, Mys Kamenny, Peschanoe oilfield, silty clay-loam</i>							
27	14	Free	-17.80	-127.88	14.52	22.3	8.3
28		Bound	-20.42	-146.99	16.37		
29	15	Free	-17.82	-129.09	13.47	25.2	8.4
30		Bound	-20.51	-152.14	11.94		
31	16	Free	-16.36	-127.88	3.0	23.1	8.1
32		Bound	-18.88	-143.65	7.39		
33	17	Free	-17.13	-124.54	12.50	22.1	7.9
34		Bound	-17.50	-131.22	8.78		
35	18	Free	-15.48	-108.17	15.67	24.1	8.0
36		Bound	-15.18	-112.72	8.72		
37	19	Free	-15.03	-109.68	10.56	23.2	7.8
38		Bound	-15.10	-106.95	13.85		

Note. W_i – initial moisture, %; W_a – moisture content in the soil sample after squeezing, %; d – deuterium excess, ‰.

lighter, than for free water. A greater variation (up to 25 ‰) of $\delta^2\text{H}$ between free and bound water may indicate different degrees of isotopic fractionation during the water – soil interactions.

The simplest explanation for the revealed distinctions between the isotopic composition of the two categories of water would be a suggestion that water molecules which are isotopically lighter have an advantage in the formation of bound water, since they are more readily captured by the force field of dispersed clay soil particles. The deuterium (d) excess analysis is illustrated by a more complicated picture: a noticeable increase in d-excess (with exception of only 12 % of measurements) for bound water as compared to free water, was observed in experiments with artificial soils saturated with tap water with a notably low heavy isotope content (Tables 1, 3). This indicates a stronger fractionation of oxygen isotopes (compared to deuterium) occurred during the water and soil molecules interactions.

The use of distilled water in the second series of experiments illustrated by Table 2, and for natural soils (Table 4) showed that the deuterium excess was almost equally greater, and lesser – for bound water, against free water. The reason for such isotope pattern is unclear, but it might as well be prompted by the soil composition and structure. This positively necessitates more research in order to provide further elucidations.

CONCLUSIONS

1. Bound water is isotopically “lighter” (according to the experimental data, in the range from 0.5 to 9.8 ‰ for $\delta^{18}\text{O}$ and from 1.3 to 29.4 ‰ for $\delta^2\text{H}$), than free water.

2. The water isotopes differentiation into volumes of free and bound water does not depend on the isotopic composition of initial water, however it is largely governed by the composition and origin of soil.

3. As it was shown experimentally, distribution of the plotted hydrogen and oxygen heavy isotopes values differ significantly from the global meteoric water line (GMWL). In this regard, the interpretation of stable isotope data of ground-ice, whose origin differs from ice of atmospheric origin, in the perspective of their applications to paleogeographic reconstructions should take into account the intrasoil transformations of the isotopic composition of soil water.

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