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SOIL CHEMISTRY

Water-Soluble Organic Acids in Cryomorphic Peat Soils of the Southeastern Bol'shezemel'skaya Tundra

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Abstract—The composition of the water extracts, the pH, and the weight concentrations of the total organic carbon and low-molecular-weight organic acids in seasonally thawed and perennially frozen horizons of cryomorphic peat soils have been determined. The quantitative analysis of the acids converted to trimethylsilyl derivatives has been performed by gas chromatography and chromato-mass spectroscopy. Hydroxypropanoic, propanoic, and hydroxyethanoic acids are the prevailing acids (30–50, 10–20, and 10% of the total acids, respectively). Malic, glyceric, hexadionic, trihydroxybutanoic, ribonic, and other acids have also been detected. It has been shown that the differences in the genesis of the peat deposits significantly affect the composition and content of water-soluble organic compounds in soils on the soil-profile and landscape levels.

Keywords: low-molecular-weight organic acids in soils, cryomorphic peat soils, Cryic Histosols, gas chromatography

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INTRODUCTION

The peat soils, including the cryomorphic peat soils, occupy significant areas (about 10%) of poorly drained watershed terraces and lacustrine-alluvial plains on the southern Bol'shezemel'skaya tundra. The pedogenesis conditions determine the complexity of the soil cover on hilly peat deposits, where peat soils of mound and hollows are distributed. On the mound tops, the lichen cover is frequently disturbed; therefore, bare peat circles appear on the surface [2, 12].

The formation of peat mounds in the permafrost bog complexes of the Bol'shezemel'skaya tundra reflects the complex history of peat formation and accumulation in the bog ecosystems of the tundra and forest-tundra zones under periodic climatic changes during the Holocene. It is believed that the formation of peat mounds began in the main cold periods, i.e., 6.7–7.3 thousand years ago, and the current appearance of frozen peat was formed in the past millennium, during the so-called Little Ice Age [8]. The features of the plant succession during the Holocene caused the heterogeneity of the mound peat layer in terms of age and botanic composition. The presence of permafrost affects the transformation of organic material and the migration of chemical compounds, primarily the most mobile and most water-soluble ones, in the landscapes. No data are available on the composition of the water-soluble organic compounds, including low-molecular-weight organic acids (LMWOAs), in the cryomorphic peat soils and the features of their profile distribution.

The Bol'shezemel'skaya tundra soils, including the peat soils, are presently subjected to increasing loads due to both anthropogenic (exploration and extraction of raw hydrocarbons) and natural (potential global climate changes) factors. This calls for the consistent and thorough study of cryomorphic peat soils, including the components of the soil organic matter.

The aim of this work was to reveal the distribution features of the total content and composition of LMWOAs in the complex of cryomorphic peat soils in the southern tundra zone of northeastern Europe.

OBJECTS AND METHODS

The studies of cryomorphic peat soils were performed within a mound–hollow complex (67°03′ N, 62°55′ E; 10 m asl), 0.6 km^2 in area, on an old lake bed located in the basin of the Seida River (a right tributary of the Usa River) in the southeastern region of the Bol'shezemel'skaya tundra (Vorkuta district, Komi Republic). The detailed characterization of the region under study and the soils of the considered peatland was reported earlier [5]. The following points should be noted. For studying the cryomorphic peat soils, a trench about 10 m in length was dug. The trench begins in the center of a peat mound and successively traverses the bare peat circle and its edge with moss-lichen cover and the hummock periphery with a moss-lichen shrub community typical for hummocky peats (Fig. 1). For revealing the distribution features of the water-soluble components of organic matter in the peat soils, soil samples were taken in three main zones of the trench corresponding to the destructive oligotrophic peat soils (I, peat circle) and typical oligotrophic peat soils (II, circle edge; III, peat mound periphery). The names of the soils are given in accordance with the *Classification and Diagnostics of Russian Soils* [7]. According to the *World Reference Base for Soil Resources* [25], all the soils were identified as Cryic Histosols. The permafrost table depth was 54, 50, and 35 cm when going from the center of the circle to the slope in late August 2013.

Soil samples were taken at different depths of the seasonally thawed layer (STL) and horizons of upper permafrost (PFRs): 0–10 (STL-1), 10–30 (STL-2), 105–130 (PFR-1), and 200–240 (PFR-2) cm. The chemical analysis of the objects was performed in the Ekoanalit laboratory certified in the System of Analytical Laboratories of the Federal Agency on Technical Regulating and Metrology (Rosstandart; certificate ROSS RU.0001.511257 of April 16, 2009) and the departments of soil science and the botanical garden of the Institute of Biology of the Komi Science Center of the Urals Branch of the Russian Academy of Sciences. The water extracts were prepared at a soil : water ratio of 1 : 25. The total content of organic carbon in the water extracts from the soils (water-soluble organic carbon (WSOC)), ρ_{WSOC}, was determined by high-temperature catalytic oxidation with IR detection on a TOC-VCPH total carbon analyzer [4] according to the M-02-2405-09 procedure developed for drinking, natural (surface and ground), and waste water. The error of determining the total organic carbon is 12%. The suitability of this procedure for the analysis of water extracts from soils was showed earlier [14]. The weight concentrations of LMWOAs (ρ_{LMWOA}) was assessed by gas chromatography–mass spectrometry (GC–MS) [15–19]. The relative error of the measurements is $\pm\delta \leq 3\%$. Given (ρ_{LMWOA}), the weight concentration of carbon from the identified LMWOAs, $\rho(\mathrm{C_{LMWOA}})$, was calculated. The values of the $\mathrm{pH_{water}}$ were measured by potentiometry [3]; the error of the procedure was 0.1 pH units. The analytical data were processed using Excel 5.0 and Statistica 6.0 software. The similarity dendrograms of the soil horizons and profiles were plotted by the weighted mean method. The Euclidean distance was used as a measure of dissimilarity; the analyzed indices included the $\mathrm{pH_{water}}$, total WSOC, and LMWOAs [10].

RESULTS AND DISCUSSION

The development of the specific microrelief of the peat hummocks—the appearance of bare peat surfaces—is closely related to the cryogenic processes determining the recent heaving processes [11] or the development of erosion [8]. The absence of vegetation on microelevations in the central part of the peat hummock ((I) soil of a bare peat circle) favors the wind transfer of snow in the winter, its redeposition, and accumulation at the periphery of the mound. This fact causes the appreciable differentiation of the mean annual and minimum temperatures in the microrelief elements of the peat mound [9]. The minimum temperatures on the surface of depressions can be lower than those on the mound tops by tens of degrees. The maximum amplitude of the temperature variation in the organic profiles of different tundra soil types is typical for the horizons where recent pedogenesis processes occur: the STL horizons to depths of 20–30 cm [6]. The absence of vegetation and the lower soil temperatures of microelevations favor the destruction (cracking) of the organomineral substrate, its transfer by strong winds (abrasion) [24], and the outcropping of the underlying peat layers. Thus, the well-decomposed surface soil layers in the first segment of the trench (I), which are characterized by a dark cinnamon, almost black, peat color, slightly differ from the analogous horizons of the two other soils.

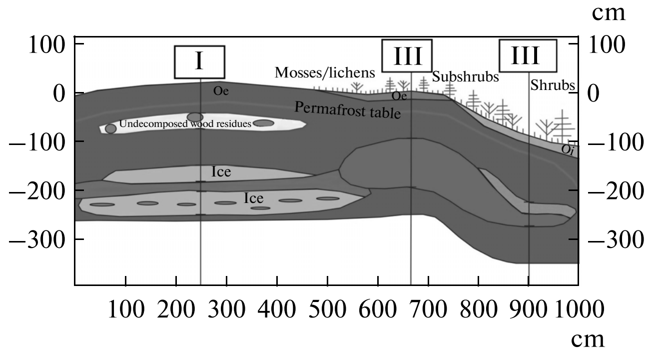

Fig. 1. Morphological structure of the soil–geocryological complex: (I) peat circle soil; (II) circle edge soil; (III) peat mound periphery soil.

In the overgrown part of the bare circle edge (II), the vegetation consists of cloudberry (*Rubus chamaemorus*), lichens, and hair-cap mosses (*Polytrichum* spp.) with the insignificant presence of dwarf birch (*Betula nana*) up to 20–30 cm high. The surface horizons of the soil in this area are composed of medium-decomposed peat light cinnamon or brown in color. In the peripheral part of the mound (III), which forms a gentle slope toward the hollow, the shrubby layer of the plant community is higher (up to 70 cm). In the moss–lichen layer, along with the presence of hair-cap mosses, the role of sphagnum mosses increases. The oligotrophic peat soil on this area is better protected from wind; it is more wetted and less subjected to seasonal cryogenic processes than the other soils. Its surface horizons consist of low-decomposed sphagnum peat.

The age of the peat deposits in the considered peatland is estimated at about 800 years at a depth of 50–

Characterization of water extracts from the soils

Parameter	Spot peat soil				Spot edge peat soil				Hummock periphery peat soil			
	sample, sampling depth, cm											
	STL-1, 0–10	STL-2, 20–30	PFR-1, 105–122	PFR-2, 201–214	STL-1, 0–10	STL-2, 20–30	PFR-1, 106–120	PFR-2, 220–240	STL-1, 0–10	STL-2, 10–20	PFR-1, 108–127	PFR-2, 225–241
pH_{water}	4.36	4.78	5.20	5.46	4.25	4.36	5.30	4.94	3.94	4.12	5.76	4.98
ρ_{LMWOA}, mg/dm^3	0.4	0.2	0.2	0.1	5.9	0.3	0.4	0.2	20.3	3.3	0.1	0.2
$\rho(C_{WSOC})$, mg/dm^3	80	44	40	68	160	76	48	68	188	172	56	72
$\rho(C_{LMWOA})$, mg/dm^3	0.2	0.1	0.1	0.1	2.4	0.1	0.1	0.1	8.6	1.3	0.03	0.1
$\acute{\omega}$ (C_{LMWOA}), %	0.3	0.2	0.3	0.1	1.5	0.1	0.2	0.1	4.6	0.8	0.1	0.1

(ρ) weight concentration; ($\acute{\omega}$) portion; (WSOC) total water-soluble carbon; (C_{LMWOA}) carbon of low-molecular-weight organic acids.

60 cm and more than 6000 years at depths exceeding 2 m [22]. The peat deposit that is presently preserved in the permafrost layer was developed during the period of the predominant distribution of thin birch and spruce forests over the Bol'shezemel'skaya tundra [1]; therefore, the botanic composition of the peat is significantly variable at different depths of the studied peat mound. The upper part of the peat deposit at depths to 40–50 cm is mainly composed of residual sphagnum mosses; its lower part consists of residual herbaceous (sedges) and woody (various birch and spruce species) plants with a small amount of green mosses [22].

The differences in the botanic compositions predetermine the specific chemical composition of the organic matter at different depths of the peat hummock and the different rates of mineralization and transformation. The decomposition of the organic substrate is also affected by the seasonal cycles of permafrost freezing–thawing [21, 23]. According to Maksimova and Ospennikov [8], the peat mounds 6–7 thousand years old were periodically subjected to thawing and freezing. It cannot be excluded that the deeper layers of the mound peat (PFR-2) remained frozen for a longer time period than the surface peat layers, which reflect the recent period of peat formation, and the peat deposit in the middle part of the profile (PFR-1). This could cause the more active decomposition of plant material in the upper layers of the frozen peat (PFR-1) than in the lower deposits (PFR-2).

The high degree of peat decomposition in the upper layers of the degraded mound part is favored by the current deficiency of fresh plant residues [20, 24]. At the same time, the lower soil temperature on the microelevation can decrease the transformation rate of organic matter in the profile of destructive oligotrophic peat soil at the recent stage of pedogenesis [9].

Thus, the organic material of the seasonally thawed layers in the soils considered significantly varies in the age, composition, and degree of decomposition in the series bare circle → circle edge → mound periphery and differs from the underlying permafrost peat layer.

The complex development and functioning of peat soils are reflected in the variable composition and properties of the WSOC. The analysis of water extracts from peat samples taken at different depths of the peat deposit showed that a general tendency of decreasing acidity down the profile is typical for all the soils considered within the peat mound (table). The values of the pH_{water} reliably increase (by 0.7–1.0 pH units) when going from the STL to the PFR. It should be noted that the difference in the pH_{water} values between the STL surface horizon (STL-1) and the lower part of the permafrost peat (PFR-2) is more manifested in the soil at the mound periphery (III), where dead sphagnum mosses are being accumulated in the upper part of the profile, and less obvious in the samples from the bare peat circle (I). The comparison of the pH_{water} values in the soil series bare spot → spot edge → hummock periphery indicates that some increase in the acidity of the analogous horizons (by 0.4–0.6 pH units) is observed when going from the first to the third profile (table). Thus, the lowest values of the actual acidity are typical for the surface soil horizons, especially the seasonally thawed layer of soil occurring at the edge of the peat mound, which is lower than the central part of the mound.

The actual acidity values correlate with the total WSOC content. The coefficient of correlation between the molar concentrations of hydrogen ions ($c_{\mathrm{H_3O^+}}$) calculated from the values of the $\mathrm{pH_{water}}$ and the total WSOC content (c_{WSOC}) is $r = 0.91$ for $P = 0.95$. The linear regression equation has the form $c_{\mathrm{H_3O^+}} = 7 \times 10^{-6}c_{\mathrm{WSOC}} - 2 \times 10^{-5}$. In spite of the close correlation between these parameters, we consider their relationship as indirect, because different organic compounds (e.g., carbohydrates, esters, alcohols) showing no acidic properties in water solutions are present in the soils [15, 17, 19].

In general, the weight concentration of WSOC in the studied cryomorphic peat soils is in the range of 40–190 mg/dm^3. These values slightly exceed the analogous parameters for the organic horizons of mineral tundra soils, where the weight concentration of carbon in water extracts does not exceed 70 mg/dm^3 [17]. The general tendency for all the considered peat soils is a decrease in the WSOC content down the profile to a depth of 105–130 cm. This parameter varies in the range of 44–188 mg/dm^3 in the seasonally thawed layer (STL-1, STL-2) and 40–56 mg/dm^3 in the middle part of the peat deposit (PFR-1). At depths exceeding 2 m (PFR-2), the weight concentration of WSOC reliably increases by 1.3 times on the average (to 68–72 mg/dm^3). It should be noted that the differences between the ρ_{WSOC} values in the PFR-2 layer are insignificant over the entire area under study, while the analogous differences in PFR-1 are reliable for the extreme profiles in the series.

Thus, the variation range of the total organic carbon in the peat mound decreases with depth. In the seasonally thawed soil layers, this parameter increases when going from the peat circle to the mound slope by 1.4 (STL-1) to 4 (STL-2) times. This can be related to the increase of the peat soil wetting in this direction, which is confirmed by the increase in the portion of sphagnum mosses in the plant cover. The complete similarity of the ρ_{WSOC} values in the PRF-2 layer (200–240 cm) and their partial similarity in the PRF-1 layer (105–130 cm) are apparently due to the higher homogeneity of the organic material. This layer was preserved earlier than the other layers; the considered area during the period of its formation could have had more uniform conditions of peat deposition: a uniform topography and a similar biota composition. At the same time, the lower homogeneity of the material with respect to this parameter in the PRF-1 layer than in the PFR-2 layer can be related to the diverse paleogeographic conditions and the possible periodic inclusion of this layer in the STL [8]. However, the definitive elucidation of the reasons for the variability of this parameter on the soil-profile and landscape levels requires additional studies.

The determination of the content and composition of the water-soluble LMWOAs using the methodological approaches that we developed earlier [15–19] showed the following results. The concentration of LMWOAs in the water extracts from the peat samples was 0.1 to 20.3 mg/dm^3 depending on the sampling depth, and their portion in the total WSOC was 0.1–4.6%. These results generally agree with our earlier data obtained for the organic horizons of mineral tundra soils ($\rho_{\mathrm{WSOC}} = 0.2$–$7.7$ mg/dm^3, $\omega_{\mathrm{WSOC}} = 0.2$–$4.7\%$) [17].

The maximum contents of LMWOAs were found in all the STL profiles of the peat soils; this parameter progressively decreases with depth (table). In the soil of the peat circle, the decrease of ρ_{LMWOA} with depth was insignificant: from 0.4 mg/dm^3 in STL-1 to 0.1 mg/dm^3 in PFR-2. In the circle edge and the mound periphery, the total content of acids within the active layer significantly decreases with depth (by 20 and 6 times, respectively). When going from STL-2 to the PFRs, the decrease in the content of LMWOAs is manifested only in the soil of the mound periphery (by 30 times). The observed differences are related to the changes in the hydrothermal conditions of the soils and the vegetation composition in the series peat circle → circle edge → peat mound periphery. The soil at the mound periphery receives an additional amount of water due to the larger accumulation of snow in the winter than in the central part of the peat mound. Sphagnum mosses dominating in the ground cover of this soil profile accumulate water; this increases the wetting of the substrate and the accumulation of acids regardless of the soil zonality, as was shown earlier [13]. Thus, the microrelief features causing the differentiation of the soil wetting and vegetation composition determine the profile distribution of individual water-soluble organic compounds in the considered series of peat soils.

The highest spatial variability of LMWOAs is typical for the seasonally thawed horizons, where their content increases when going from the peat circle to the peat mound slope by 17 (STL-2) to 50 (STL-1) times. The permafrost layer is homogeneous with respect to LMWOAs, the content of which is 0.1–0.4 mg/dm^3. This allows concluding that the content of LMWOAs determined by GC–MS is a more variable characteristic than the total WSOC content in time (organogenic substrate of different ages) and space (microrelief).

In all the studied peat samples, the LMWOAs contained significant amounts of hydroxypropanoic ($\mathrm{p}K_a = 3.86$), propanoic ($\mathrm{p}K_a = 4.87$), and hydroxyethanoic ($\mathrm{p}K_a = 3.8$) acids. They make up 30–50, 10–20, and 10% of the total acid content, respectively, on the average. Appreciable amounts (up to 10%) of malic acid ($\mathrm{p}K_{a1} = 3.46$, $\mathrm{p}K_{a2} = 5.1$) were identified in the upper horizons of all the profiles; up to 10–20% of hydroxybutanoic acid ($\mathrm{p}K_a = 3.83$) was detected in the lower horizons. Valeric, propanoic, butanedionic, glyceric,

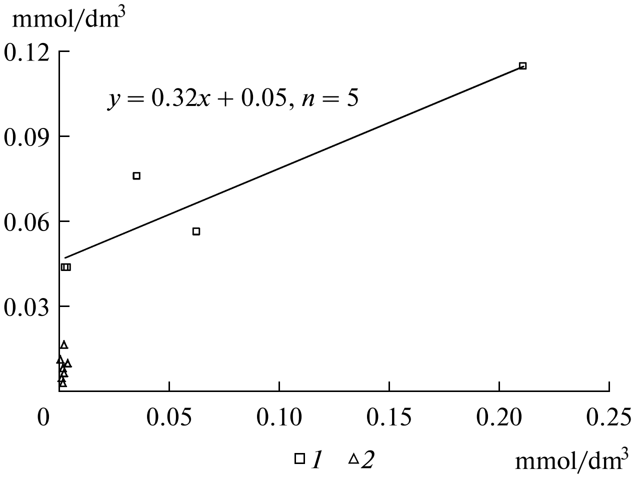

Fig. 2. Concentration of hydrogen ions calculated from data on pH of soil water extracts as a function of the LMWOA concentration in water extracts: (*1*) samples with pH 4.0–4.4; (*2*) samples with pH 4.8–5.5.

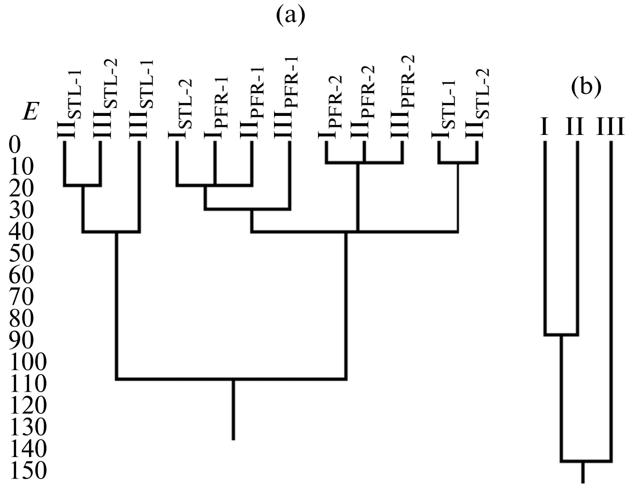

Fig. 3. Similarity dendrograms of (a) horizons and (b) soils: (*E*) Euclidian distance; (I) peat spot soil; (II) spot edge soil; (III) peat mound periphery soil.

hexanedioic, 2,3,4-trihydroxybutanoic, and riboic acids were also detected.

The molar concentration of any acid is directly proportional to the concentration of hydrogen ions in the solution ($c_{\mathrm{H_3O^+}} = \sqrt{K_a c_{\mathrm{HA}n}}$, where K_a is the acidity constant, and $c_{\mathrm{HA}n}$ is the molar concentration of the acid). Two groups of the studied samples are clearly distinguished in the plot of the hydrogen ion concentration (calculated from the pH$_{\mathrm{water}}$ values) as a function of the identified acid concentration (Fig. 2). The first group includes the STLs of all three soils, except for STL-2 of the circle soil. These objects are characterized by the high coefficient of correlation between the discussed parameters ($r = 0.93$ for $P = 0.95$), acid content of 2 to 27 mg/dm^3 ($\omega_{\mathrm{WSOC}} = 1$–$14\%$), and pH = 4.0–4.4. The second group includes the samples of PFRs and the lower STL of the circle soil. For them, no correlation is found between these parameters; the concentration of acids corresponds to the range of 0.3–0.4 mg/dm^3 ($\omega_{\mathrm{WSOC}} = 0.7\%$ on the average); pH = 4.8–5.5. The similarity of the properties of STL-2 and the PRF layers indicates the rise of the profile.

This shows that the compounds identified by GC–MS determine the acidity of the water extracts in the STLs, where recent pedogenesis processes occur. In the second group, the essential sources of actual acidity are probably undetected weaker, probably aromatic, acids [15], as well as water-soluble HMWOAs. The different character of the acidity in these two groups of horizons is related to the difference not only in the age (i.e., the degree of humification) of the material but also in the material composition of the original organic substrate, as was noted above. The absence of radical differences between the lists of acids in the STLs and the PFRs can be related to the fact the procedure used can identify only narrow groups of organic compounds [15].

The similarity dendrogram of the objects (Fig. 3a) confirms the homogeneity of the permafrost layers at depths exceeding 2 m. The PFRs of the three soils at a depth of 100–130 cm are also highly similar. Being homogeneous in space, these two layers are significantly different. The following facts argue for the outcropping of the lower layers and the removal of the surface horizon in the circle soil: first, the presence of the lower STL of this soil in the group of the PFR-1 horizons and, second, the similarity of the peat circle STL-1 to the circle-edge STL-2. The hydrothermal conditions and vegetation composition of the soil in the periphery of the peat mound favor the formation of a surface layer differing from those of the two other soils. Thus, the dendrogram drawn from the experimental data showed that the STLs of the three soils reveal the maximum variability of the selected parameters. The same depths are characterized by the highest frequency of the freezing–thawing cycles and maximum variability of temperatures, which confirms the earlier results [6, 9].

From the similarity dendrogram of the horizons, the scheme of the soil profile structures was drawn, in which the similar objects in terms of the studied parameters (Fig. 3a) are similarly colored. The scheme clearly demonstrates the manifestation of cryogenic processes: heaving and abrasion (Fig. 4). The similarity dendrogram of the profiles (Fig. 3b) indicates the similarity of the first two profiles: the soils of the circle and its edge. Their hydrothermal conditions and biota distinguish them from the hollow soil.

CONCLUSIONS

The study of water-soluble organic compounds in the STLs and PFRs of cryomorphic peat soils in the

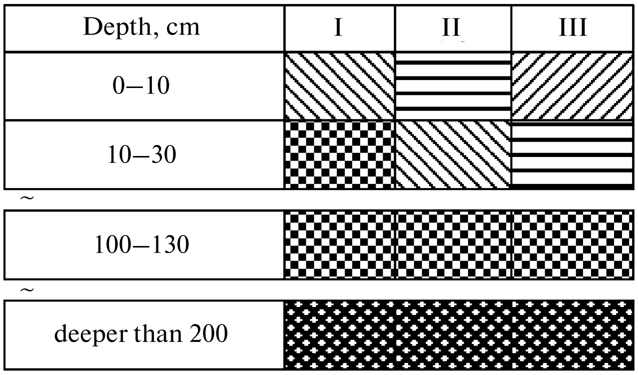

Fig. 4. Scheme of soil profile structures (from the similarity dendrogram of horizons). The explanations are in the text.

southeast of the Bol'shezemel'skaya tundra showed the following.

(1) The content of total WSOC in the soils is 40–190 mg/dm^3, and that of LMWOAs is 0.1–20.3 mg/dm^3 (less than 5% of the total carbon in the water extracts). All the soils contain large amounts of hydroxypropanoic (30–50%), propanoic (10–20%), and hydroxyethanoic (~10%) acids.

(2) The spatial variability of the WSOC in the STLs of the soils on the plot depends on the microrelief, which develops under cryogenesis conditions and determines the differentiation of the soil wetting and vegetation composition. The content of the total organic carbon increases when going from the peat circle to the mound slope by 1.4 (STL-1) to 4 (STL-2) times, and that of the acids increases by 17 (STL-2) to 50 (STL-1) times. The permafrost layer is homogeneous in terms of these parameters within the plot.

(3) Regardless of the soil position in the relief, the content of total WSOC decreases down the profile to a depth of 105–130 cm and then increases from a depth of more than 2 m by 1.3 times on the average. In the soil of the peat circle, the weight concentration of acids insignificantly changes down the profile (0.1–0.4 mg/dm^3). Within the active layer in the circle edge and on the hummock periphery, this parameter decreases with depth by 20 and 6 times, respectively. When going from STL-1 to the PFRs, the decrease in the content of LMWOAs is manifested only on the periphery of the peat mound (by 30 times).

(4) The cluster analysis based on the parameters of the water extracts from the soils (pH$_{\text{water}}$ and weight concentrations of total organic carbon and LMWOAs) substantiated the manifestation of cryogenic processes: heaving and abrasion. The similarity dendrogram of the profiles indicates the similarity of the soils in the peat circle and its edge. Their hydrothermal conditions and biota differentiate them from the hollow soil.

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