



Article Soil Organic Matter in Soils of Suburban Landscapes of Yamal Region: Humification Degree and Mineralizing Risks

Ivan Alekseev^{1,*}, Gleb Kraev², Aleksandr Shein² and Pavel Petrov³

- Otto Schmidt Laboratory for Polar and Marine Research, Arctic and Antarctic Research Institute, 199397 Saint Petersburg, Russia
- ² Yamal-Nenets Center of Arctic Research, 629008 Salekhard, Russia; kraevgn@yanao.ru (G.K.); a.n.shein@yandex.ru (A.S.)
- ³ Institute of Physics and Chemistry, Ogarev Mordovia State University, 430005 Saransk, Russia; petrovps83@gmail.com
- * Correspondence: alekseevivan95@gmail.com

Abstract: Various research issues related to soil organic matter in permafrost soils are still poorly investigated. At the same time, numerous investigations have shown the importance of permafrost soils, as they serve as a huge reservoir of organic matter. This work is aimed at the investigation of permafrost-affected soils in the southern part of the Yamal region, namely at the assessment of composition (fractional, elemental and molecular) of soil organic matter in topsoils formed under different biogeoclimatogenic conditions in tundra and forest tundra. Special attention was given to assessment of potential vulnerability of soil organic matter in the context of Arctic warming. Results showed the predominance of fulvic acids in the humus of the studied soils, which indicates mineralization risks in the humic substances system of Arctic soils under conditions of further warming. The ¹³C-NMR analysis of humic acids revealed that all the studied soils are characterized by higher portions of aliphatic groups of carbon and decreased portions of aromatic groups and revealed early stages of the humification process in studied soils. These results contribute to scarcely distributed research of soil organic matter in permafrost soils of the Arctic. Moreover, our research provided new data on the vulnerability of soil organic matter and its possible mineralization risks under pronounced climate change in the Arctic using the modern instrumental technique.

Keywords: Yamal; permafrost; Arctic warming; soil organic matter; humification; ¹³C-NMR spectroscopy

1. Introduction

Currently, the Russian Arctic is facing an intensification of the anthropogenic forcing rate due to various human activities, e.g., oil and gas exploration and related objects, urbanization, as well as climate change. All these result in soil degradation and further degradation of soil ecosystem benefits. Investigation of Arctic soil functions and soil ecosystem services are crucial for development of strategies for sustainable environmental management in response to anthropogenic disturbances and climate change.

Arctic soils are specifically designated from soils of other landscapes. They are manifested in numerous characteristic features of cryopedogenesis, which is mainly connected with the freezing–thawing processes in the active layer and upper permafrost [1,2]). The depositional characteristics of environments and low rates of decomposition, together with the cryoturbation process, have served as a prerequisite for accumulation of enormous stores of organic carbon in permafrost-affected soils [3,4]. At the same time, a sufficient knowledge gap in understanding soil organic matter of permafrost soils exists; investigation of molecular structure and quantification of stocks, its distribution along the soil profiles as well as along the geographical gradients, dynamics of nutrient pools and their stabilization and biodegradability, greenhouse gas emissions, etc., are all areas that require more study [5].



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Copyright: © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). Although the number of research studies on estimation of carbon pools in permafrostaffected soils in the Russian Arctic region has increased over the last decades [4–8], quantitative estimates of soil carbon stocks and mineralization potential are still under discussion and subject to large uncertainties. This is due to that a high number of existing research studies still do not fully cover many regions and landscapes of the Arctic [9]. Hence, more detailed landscape-scale assessments of soil organic matter in understudied areas are needed [1]. Most of researchers' current believe that Arctic warming will lead to increased CO_2 emissions into the atmosphere due to thawing of permafrost and increased rates of soil organic matter decomposition [2,10]. This would probably lead to even higher rates of warming of the atmosphere based on the principle of positive feedback.

Soil organic matter is a central component of biogeochemical cycles [11,12]. Thus, the organoprofiles of soils could reflect the rate of circulation of chemical elements in ecosystems, and therefore the study of quantitative and qualitative characteristics of soil organic matter could provide us a deeper understanding of ecosystem functioning. Previously, numerous studies aimed at the investigation of individual natural (as well as anthropogenic) factors affecting the formation of soil organic matter [13,14]. At the same time, there are practically no comprehensive studies aimed at identifying the main natural and anthropogenic factors that determine the formation of various types of soil organophiles. This problem is especially acute for the soils of the polar regions of the Earth.

Stability and biodegradability are key characteristics of soil organic matter which need to be taken into account for assessment of current and potential stocks of organic carbon as well as for their qualitative assessment and studying the dynamics of changes in the system of organic matter. The stability rate of soil organic matter is related to the degree of humification, e.g., more advanced humification stages include depletion of labile molecules together with an increase in the overall aromaticity level, therefore soil organic matter stability level is increased. Previous studies of soil organic matter in Cryosols of polar regions have revealed generally partial decomposition of organic molecules, which are characterized by preservation of the chemical characteristics of their precursor material due to slow humification [15,16]. Given the fact that a huge amount of carbon from organic compounds is stored in Arctic soils, the possible destabilization in the system of soil organic matter can lead to unpredictable global consequences.

This is why our work is aimed at the investigation of molecular and fractional composition of soil organic matter in permafrost-affected soils of suburban areas of the southern Yamal region to provide new insights into the issue of stability of soil organic carbon pools in the Russian Arctic. For this study, different instrumental and routine laboratory techniques have been used. We hypothesized that the molecular structure of soil organic matter in studied Cryosols would be similar to the case of Histic topsoils, thus we expected a different HA structure in forest–tundra soils where humification precursors have different composition (more aromatic compounds and higher humification degree).

The specific research objectives of this work were:

- To assess the degree of humification of soil organic matter in organoprofiles of permafrost soils in suburban areas of the southern Yamal region;
- To investigate the molecular composition of humic acids in selected topsoil horizons using ¹³C-NMR spectroscopy along the geographical gradient and delineate possible mineralizing risks in conditions of Arctic warming.

2. Materials and Methods

2.1. Regional Setting and Fieldwork

Soil samples for this investigation were collected in the Yamal-Nenets Autonomous region during the fieldwork organized by Arctic Research Centre of the Yamal Autonomous region in August–September 2021. We analyzed 4 soil profiles in vicinities of Salekhard and Novy Urengoy—the most urbanized towns in Yamal-Nenets Autonomous region with populations of 42,494 and 113,254, respectively. Both investigation areas are located in the transitional zone between tundra and forest tundra (Figure 1, Table 1).



Figure 1. Areas of investigation: 1—Gornoknyzevsk; 2—Vicinities of Salekhard (mires); 3—Novy Urengoy, southwestern suburban (palsas); 4—Novy Urengoy, southern suburban (thermokarst).

Table 1. Geograp	hical d	lescription	of the	studied	sites.
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Site	Geographical Coordinates	Landscape Description and Predominant Plant Community	Soil Type (WRB)	
Gornoknyzevsk	N 66,569882 E 66,880857	Flat = tundra site; shrub–moss–lichen tundra	Reductaquic Cryosol	
Vicinities of Salekhard (mires)	N 66,54178 E 66,72707	Wetland, 9 km from Salekhard town; sedge-moss tundra	Histic Cryosol	
Novy Urengoy, southwestern suburban	N 66,167214 E 76,652661	Flat forest-tundra, predominantly flat relief complicated by palsas; forest tundra (<i>Larix sibirica</i>)	Histic Reductaquic Cryosol	
Novy Urengoy, southern suburban	N 66,039521 E 76,608348	Lake thermokarst landscape; forest tundra (<i>Larix sibirica</i>)	Spodic Cryosol	

A notable average air temperature increase has been recognized in the Yamal peninsula over the last 30 years [17]. This is a crucial change for intensification of humification of huge pools of organic matter in permafrost soils of the region as well as for enhancing the risks of greenhouse gas emissions; therefore, the problem of investigation of permafrost soils and soil organic matter is of great interest among researchers [6,8,10].

Studied sites were 50×50 m, each site included six sampling points. Soils were sampled from soil profiles at different depths in 20×20 cm soil pits. Topsoils from each sampling point were collected and classified according to the WRB system [18,19]. The samples were stored in double sterile plastic bags, labelled and transported to the laboratory in Saint Petersburg (Arctic and Antarctic Research Institute). In the laboratory samples were air-dried and sieved (2 mm) before analysis.

The main types of parent material in the Yamal region are loam and sandy loam (sorted silty material or with inclusion of boulder material). In addition, parent material is represented by alluvial origin sands, but this type is less distributed. According to mineralogical point of view, quaternary sediments of the Yamal region are characterized by similar composition. Terrigenous minerals are represented mainly by quartz, feldspars and micas [20]. However, it should be emphasized that some key plots in the area of investigation are quite unique for the Yamal region since they are covered by predominantly sandy deposits. Autonomous conditions of the Arctic tundra subzone are characterized by mosaic vegetation cover represented by herb–lichen–moss, herb–moss–lichen, shrub–moss and shrub–lichen phytocenosis (Figure 2). A big role of permafrost processes in formation of vegetation cover reflects the polygonal and hummock tundra. Herb–moss swamps



are predominated. Meadows are developed in the river valleys and on the bottom of dried-up lakes.

Figure 2. Typical landscapes of the studied sites: **1**—Gornoknyazevsk (shrub–moss–lichen tundra); **2**—vicinities of Salekhard (shrub–moss tundra); **3**—Novy Urengoy, southwestern suburban (dry forest tundra site); **4**—Novy Urengoy, southern suburban (lake thermokarst landscape).

Some soil studies in southern Yamal aimed at different topics (soil microbiome, carbon stocks, geochemical regime) were conducted previously [6,18]. Usually, investigated soils were described as those with acid pH values, low fertility level, high exchangeable and hydrolytic acidity and quite high organic carbon stocks [6,18]. Instrumental evaluation of permafrost table using vertical electrical resistivity sounding depth was also conducted in previous years, with values varying from 70 and 120 cm in the southern Yamal region [21]. Moreover, a comprehensive investigation of microbiome in soils of southern Yamal peninsula was performed previously and delineated the main archaeal and bacterial phyla in permafrost soils as well as their relationship with environmental factors [6].

2.2. Laboratory Analyses

The laboratory analyses for determining the main soil parameters were performed by standard procedures. The pH in water values were measured using a pH150 m (1:2.5 soil:solution ratio). Organic carbon contents were measured using Vario EL III Element Analyzer (Elementar Analyze System). Humic (HAs) and fulvic acids (FAs) for Cha:Cfa analysis were extracted by the method described by Schnitzer [22].

The humic acids (HAs) were extracted from soil samples using the standard procedure proposed by the International Humic Substances Society [23]. The HAs were extracted with 0.1 M NaOH solution (soil:solution ratio 1:10) under N₂. Then, after 24 h of shaking the alkaline supernatant was separated from the soil residue by centrifugation at $1.516 \times g$ for 20 min and acidified to pH 1 by adding 6 M HCl. Fulvic acids (FAs) were separated from the precipitate (HAs) by centrifugation at $1.516 \times g$ for 15 min. Afterwards, HAs

were re-dissolved in 0.1 M NaOH and shaken for 4 h. The HA solution was then acidified with 6 M HCl to pH 1, and separated by centrifugation. The humic acids were then demineralized by shaking 12 h in 0.1 M HCl/0.3M HF (solid:solution ratio 1:1) and washed with deionized water until the was pH 3. The solid humic acids were then freeze-dried. The ¹³C-NMR spectra of the air-dried humic acids were recorded using JNM-ECA 400 NMR spectrometer (JEOL, Tokyo, Japan) with a working frequency of 100.53 MHz using the Cross-Polarization Magic Angle Spinning procedure. The spinning speed was 6 kHz, while the contact time was 5 ms and the recycle delay 5 s. The chemical shifts were referenced to adamantane (29.46 ppm). The processing (quantitative) was performed by numerical integration in the regions corresponding to the functional groups and the molecular fragments with a preliminary automatic correction of the phase and the baseline using a Delta v. 5.0.2. (JEOL, Japan). The data were then corrected for the water and ash content. The oxygen content was calculated by the difference of the samples' mass, gravimetric concentration of C, N, H and ash. We used Leonardite HA standard (1S104H) and the Elliot soil HA standard (1S102H) as standards. The carbon groups on the ¹³C-NMR spectra were determined according to Table 2.

Molecular Fragments	Chemical Shift, ppm
Alkyl C group	10–45
Alkyl C group, substituted by oxygen and nitrogen atoms	45–110
Aryl/olefin C group	110–160
Carbonyl/carboxyl/amide C group	160–220

Table 2. Carbon groups and their chemical shifts on ¹³C-NMR spectra.

3. Results and Discussion

Most of the study sites are characterized by predominance of shrub–moss and shrub– moss–lichen tundra with *Betula nana, Vaccinium uliginosum,* and *Empetrum nigrum* as dominants and forest tundra (*Larix sibirica*) landscapes. Histic Reductaquic Cryosols and Histic Cryosols are predominant soil types. Soil profiles were usually characterized by thixotropic features within the upper 20 cm. These highly saturated soils are characterized by peat formation (fibric material) and the presence of quite thick Histic horizon, although at welldrained sites of forest tundra Histic Spodic Cryosols have been also identified (Figure 3).

3.1. Soil Organic Matter Humification Degree

One of the most important functions of humic substances is their participation in the circulation of organic carbon compounds and their stabilization. Due to the accumulation of stable fractions of humic substances, about two-thirds of the organic carbon of present-day terrestrial ecosystems is concentrated in the upper one-meter layer of soils [1], especially in soils of cryogenic ecosystems. Permafrost soils serve as a huge reservoir of organic carbon [4]. This, together with increasingly obvious Arctic warming, requires a more detailed assessment of the dynamics of humus compounds in highly sensitive ecosystems of the Arctic. At the same time, it is crucial to study the process of humification, as well as its dynamics along the geographical gradient in the different bioclimatic and lithological–geochemical conditions of the Arctic. Usually, the organic matter of Arctic soils is characterized by a predominance of fulvic acids [6,7].

The predominance of carbon of fulvic acids (Table 3) observed in the studied soils, together with predominance of low-molecular weight fragments in FA fractions, points out the increased risks of mineralization in the humic substances system of polar soils under conditions of further warming of the Arctic climate.



Histic Cryosol (Surroundings of Salekhard)



Spodic Cryosol (Novy Urengoy)



Reductaquic Cryosol (Gornoknyazevsk)



Histic Reductaquic Cryosol (Novy Urengoy, palsa mires)

Figure 3. Investigated soil profiles (soil names according to WRB classification system).

Although in all samples investigated Cha:Cfa ratios were less than 1, Spodic Cryosol (Novy Urengoy) formed in forest tundra on clayey parent material showed the highest Cha:Cfa value—0.80. This demonstrates that in conditions of litter influx (enriched with lignin compounds) higher rates of humification are observed. The ratio of carbon of HAs to carbon of FAs could be used for a direct assessment of soil organic matter stability levels, as well as degradation and mineralization risks. The lowest Cha:Cfa ratios were found in Histic horizons (0.28–0.58). This further demonstrates the importance of research on soil organic matter (SOM) and its composition, especially in permafrost soils, since they are one of the main sources of unstable soil organic carbon [1].

Soil Depth, cm	pH _{H2O}	Corg, %	Cha, %	Cfa, %	Cha:Cfa			
Histic Cryosol (surroundings of Salekhard)								
0–5	5.43 ± 0.12	32.64 ± 2.02	1.54 ± 0.08	2.65 ± 0.12	0.58 ± 0.08			
Spodic Cryosol (Novy Urengoy)								
0–3	6.01 ± 0.15	17.12 ± 1.08	1.72 ± 0.10	2.16 ± 0.13	0.80 ± 0.06			
Reductaquic Cryosol (Gornoknyazevsk)								
0–7	5.76 ± 0.13	22.62 ± 0.88	0.90 ± 0.07	3.21 ± 0.14	0.28 ± 0.03			
Histic Reductaquic Cryosol (Novy Urengoy, palsa mires)								
0–6	5.12 ± 0.21	33.94 ± 1.76	4.65 ± 0.18	12.05 ± 0.32	0.39 ± 0.04			

Table 3. The pH, organic carbon content and humification in topsoil horizon of selected soils (with \pm standard deviations).

3.2. Elemental Composition of Humic Acids

The elemental composition of humic acids from the studied soils are given in Table 4. Elemental composition of humic acids provides crucial information on the humification degree of soil organic matter, condensation of humic acids, as well as oxidation. We found low variabilities (CV < 9.50%, Table 3) in both elemental composition of humic acids of investigated soils and their corresponding elemental ratios. The lowest coefficient of variability was found for carbon content (CV = 2.95%), while the average value for all soils was 51.42%. The highest variability was found in H/C ratio values (CV = 9.50%), with the highest value in topsoil horizon from Spodic Cryosol from the suburban area of Novy Urengoy (0.12) and the average value of 0.10. The highest C/N ratio was found in topsoil of Histic Cryosol from surroundings of Salekhard (11.94) and the lowest in Spodic Cryosol (10.23). The O/C ratio was characterized by the average value of 0.69 with the highest ratio in Reductaquic Cryosol (0.73). In general terms, these data are comparable with that previously reported for Arctic soils [2,6,8,24]). Soil humic acid (HAs) compositions are widely described to be driven by the quality of the precursor material together with climatic features of the region [25]. This explains similar elemental composition of humic acids in our study and previous works from southern Western Siberia and adjacent areas.

Table 4. The elemental composition of the studied humic acids from studied topsoils and standard materials.

Soil ID	C, %	H, %	N, %	O, %	C/N	H/C	O/C	Ash, %
Histic Cryosol (surroundings of Salekhard)	50.50	5.12	4.23	34.23	11.94	0.10	0.68	5.00
Spodic Cryosol (Novy Urengoy)	51.26	6.13	5.01	35.21	10.23	0.12	0.69	5.00
Reductaquic Cryosol (Gornoknyazevsk)	50.30	5.11	4.43	36.51	11.35	0.10	0.73	5.00
Histic Reductaquic Cryosol (Novy Urengoy, palsa mires)	53.60	5.21	5.12	36.40	10.47	0.10	0.68	5.00
CV, % *	2.95	9.16	9.25	3.03	7.20	9.50	3.27	0.00
Leonardite HA standard (1S104H)	63.81	3.70	1.23	31.27	51.88	0.06	0.49	2.60
Elliot soil HA standard (1S102H)	58.13	3.68	4.14	34.08	14.04	0.06	0.59	0.90

* CV—coefficient of variation.

3.3. ¹³C-NMR Spectroscopy

The ¹³C-NMR spectra of investigated topsoils are summarized in Figure 4. Histic Cryosol (surroundings of Salekhard) showed the presence of an intense peak in the area of a chemical shift of 30–32 ppm, which can be attributed to carbon atoms of methylene. Methylene carbon atoms, as some researchers suggest, may be the result of the accumu-

lation of lipids [26,27]. In the ¹³C-NMR spectrum of this soil, there is also a peak with a chemical shift of 175 ppm (carbon of carboxyl groups). The presence of a peak with intensity of 72 ppm of Histic Stagnic Cryosol from suburban areas of Salekhard also explains the predominance of CH(O) groups of ring carbon atoms in carbohydrates [28]. The signal in this region of the spectrum comes from different HC-OH groups of cellulose and other carbohydrate fragments [27]. Despite this, signals from carbon in the α position in polypeptides (C(O)–C*(R)H-NH–)n might be observed in this region as well [27,29]. In the region of the carboxyl group (164–183 ppm), the most intense peak is observed in the area of 170–174 [26]; however, this might also characterize the carbonyl group of amides and polypeptides [27].



Figure 4. The ¹³C-NMR spectra of investigated topsoil horizons.

Spodic Cryosol (Novy Urengoy, forest tundra) as well as Histic Cryosol was also characterized by the most intense peak of 175 ppm (carboxyl group). Previously, it was discussed that the carboxyl groups could be partly derived from the polyuronic acid moieties associated with the polysaccharides [30]. Thus, humification in these soils is carried out more intensively, which leads to the formation of humic acids with a more developed peripheral aliphatic part. Less intense peaks were found in the aliphatic zone of a spectrum at 23 ppm and 28 ppm (acetyl group, CH₃CO–).

The ¹³C-NMR spectrum of Reductaquic Cryosol (Gornoknyazevsk) showed the most intense peak at 174.6 ppm (carboxyl group). Less intense peaks are observed at 19.98 ppm (primary alkyl—methyl group). The abovementioned group is usually described to be originated from the recalcitrance and accumulation of waxes, lipids, cutin, and suberin polymers from plants [27,31]. The Histic Reductaquic Cryosol (Novy Urengoy, palsa mires)

spectrum showed a quite different picture. The two most intense peaks were found in a methyl carbon group (19.25 ppm) and carboxyl group (173.86 ppm). The second peak at the carboxyl group characterized the carboxyl carbon but could be also attributed to carbonyl C from amides and polypeptides.

The humification process in organic materials has been previously studied in detail using various methodologies—¹³C-NMR, ¹H-NMR, high-pressure size exclusion chromatography (HPSEC), as well as Fourier transform infrared spectroscopy (FTIR) [8,32,33]. Results of these studies showed that the humification process could be associated with increased aromatic condensation and the conjugation degree [32]. Generally, humification has been found to be related to both an increase in the aromaticity (mainly in phenol groups), and a reduced amount of oxygen containing functional groups (mainly carboxylic groups). Previously, some research on soil organic matter in Arctic soils of Northwestern Siberia found aromatic compounds of humic acids as more stable compared to aliphatic [5]. Different authors also showed that urbanization could impact soil organic matter molecular composition, usually showing the trend of increased aromaticity due to more intense aeration, oxidation and accumulation of nitrogen in the aromatic compounds in topsoils [34,35]. Interestingly, these features of HAs were not found in any of the studied topsoils in our work. This confirms that more research on soil organic matter in urbanized territories of the Arctic is needed to specify the stability and biodegradability features of this region.

4. Conclusions

It is previously discussed that the degree of humification of organic residues in studied soil areas depend on the plant community's composition, as well as lithological and geochemical conditions [6,8,12]. Most of the studied soils from suburban areas in southern Yamal are characterized by quite low Cha:Cfa ratios, especially in the case of Histic horizons. However, in conditions of litter influx in forest tundra sites, which is enriched with lignin compounds, higher rates of humification are observed. Since most of the studied soils are characterized by predominance of carbon of fulvic acids, further mineralization of humus in conditions of predicted global warming is expected to lead to intensified emissions of greenhouse gases to the atmosphere.

Humic acids of topsoil horizons in different bioclimatogenic conditions of the southern Yamal region were investigated by ¹³C-NMR spectroscopy to reveal their specific molecular structure. All the studied samples were found to have the most intensive peaks on ¹³C-NMR spectra around 175 ppm, which corresponds to the carboxyl C group. This explains more intense humification in the fine earth of these soils together with the formation of a more developed peripheral aliphatic part in humic acids. Results obtained from ¹³C-NMR spectroscopy revealed only small differences in molecular composition of humic acids across the geographical gradient of the studied sites in the southern Yamal region. This can be connected with the homogeneous composition of vegetation and similar climatic conditions, as well as the soil hydrological regime in most of the studied soils. However, some previous investigations reported that Arctic soils could have more stable aromatic parts compared to aliphatic ones, but we did not find such a feature in the suburban soils of the southern Yamal region. Therefore, more research needs to be performed in different locations of the Arctic region to collect further data on the molecular composition of soil organic matter and its variability across biogeoclimatogenic gradients. We could conclude that the studied soils of the southern Yamal peninsula are at the early stages of humification, since the aliphatic carbon groups are prevailed in the ¹³C-NMR spectra of humic acids.

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