

Article

Simulation of Smoldering Combustion of Organic Horizons at Pine and Spruce Boreal Forests with Lab-Heating Experiments

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Abstract: Wildfire is a threat for many boreal ecosystems and induces deep modifications in organic horizons. In this paper, we have considered fire-induced changes to the organic horizon properties. The effect of fire was studied by using a forest litter burning experiment. Sample heating was performed in the lab at fixed temperatures (200, 300 and 500 °C), on a set of O horizons developed under pine (*Flavocetraria-Pinetum* association) and spruce (*Piceetum hylacomium splendens* association) forest litters. Litters were analyzed in terms of pH, specific electrical conductivity, specific surface area, total carbon (C_{tot}) and nitrogen (N_{tot}) content, water-soluble carbon and nitrogen, $\delta^{13}C$ and $\delta^{15}N$ stable isotope analysis and ^{13}C NMR spectroscopy. The mean pH values increased from ~5 to ~8.2 with an increase in the influence of temperature. The specific electrical conductivity and specific surface area properties increased as well from ~255 to ~432 and from 0.42 to 1.84, respectively. C_{tot} and N_{tot} decreased, but at the same time the inorganic carbon content increased. The aromaticity of organic matter after the fire increased. The results of the present study show that organic horizons are changed by wildfire and this discussion made it clear to help with the understanding how fire affects organic matter.

Keywords: wildfires; boreal forest; soil organic matter; combustion experiment; solid-state ^{13}C NMR; $\delta^{13}C$ and $\delta^{15}N$ stable isotopes



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1. Introduction

Wildfire is one of the most important factors of natural cycles, with its frequency and severity depending on human activities [1]. Fires are a global phenomenon destabilizing all the components of the biosphere and regulating their functioning, leading to the formation of new post-fire ecosystems [2–6]. Global warming is expected to increase fire frequency and extent in ecosystems at high latitudes [7–9]. Fires in the European part of Russia are spread more to the North, affecting large areas that used to be less frequently exposed to wildfire events because of climate change in recent years. Over the last decade 2963 wildfires with the total area of 127.51 thousand ha were revealed in the Komi Republic (Northwestern district, Russia) [10]. Most of the wildfires were human-caused or resulting from lightning strikes. The impact of wildfires on the natural environment is multidimensional and complex. By interfering with the carbon, nitrogen, water and other cycles, wildfires frequently upset the ecosystems balance created by decades.

Soil and soil organic horizon are substantial part of ecosystems. Organic horizons are primarily combustible during wildfires and some part of burnt materials is released into the

atmosphere. The unreleased part and plant remains persist on the soil surface and they are subsequently transported down into the horizons by the water movement and layering of new plant material. However, the issue of how organic horizons affect soil organic matter (SOM) in the high latitude boreal forests has been insufficiently studied so far. Given that boreal forests are the largest in the world, this study may result in important information that can be applicable globally. Therefore, it is essential to understand the ways of organic horizon transformation through low intensity surface wildfires. In this paper, we consider only surface fires, as the most frequent and affecting mainly only the organic horizon. It is known that the components of soils and soil organic horizons undergo deep alterations during the fire [11–13]. This is partially caused by a deep transformation of soil physical, chemical and biological characteristics associated with qualitative and quantitative changes in the most functional fraction of soil such as organic matter [14–16]. Severity and intensity of alterations resulting from wildfires depend on the type of fires (crown or surface forest fires), availability, amount, nature, and moisture content of live or dead fuel (ground vegetation and deadwoods), weather (air temperature, humidity and wind speed) and the physiography of the site [17,18]. All of these factors are increasingly altered by human activities and can greatly impact the consequences of a fire [19,20]. Moreover, mechanical, physical, chemical and biological properties of soils and litter can be altered immediately by high temperatures or after vegetation successions [15,21,22].

Major thermal destruction of the entire vegetation cover and soil occurs during intense crown wildfires. The organic component of soil is the one most exposed to fire, in terms of both content and composition [14,23]. Most often fire causes a substantial reduction or even complete removal of the litter layer [16,23,24].

In turn, minor changes in the composition of organic horizons and soils occur during rapid surface wildfires. At the same time, apart from burning and emissions of gases and aerosols [25], the organic matter (OM) of organic horizons and soils may be transformed [15] and can persist in the ecosystems [26,27]. In most forest ecosystems the burnt material remaining on the soil after wildfires mainly comprises non-woody char derived from the organic horizons. Despite the importance of the role of this material in the carbon balance, soil conservation and ecological processes, few studies have examined the properties of this type of charred biomass, which may vary widely in different forest ecosystems and also within burnt areas [28,29].

Fires in boreal forests and mires frequently occur in the form of smoldering fires in the soils organic horizons that accumulate in these ecosystems. In contrast to flaming combustion, smoldering is a flameless form of combustion that occurs when oxygen reacts with the surface of solid fuels [30,31]. This form of combustion typically takes place at much lower temperatures than flaming combustion (500 to 700 °C versus 1500 to 1800 °C) [32]. In soil organic horizons combustion is known as pyrolysis with partial oxidation of the fuel resulting in dehydration and charring and char oxidation occurs reducing the pyrolyzed fuel to ash [33].

Some of the most important parameters that are affected by the fires are fractional composition, pH, electrical conductivity and specific surface area, which perform a number of important functions in soils [34–36]. Quantitative and qualitative composition of organic matter plays an important role in the substances' cycle and strongly depends on fires and the content of carbon and nitrogen in different forms as well [37–39]. Generally, studies of the above-mentioned parameters are focused on mineral horizons of soils, with organic horizons left unattended. The reason why studies do not focus on organic soils is that they are often burnt to the ground by wildfire. Here, we attempt to explore how selected forests litter parameters change with temperature in order to determine the impact of fires and eliminate some understanding gaps.

We hypothesized that (1) the main chemical properties of organic horizons will be strongly transformed as a result of the influence of high temperatures, (2) the stable ^{13}C and ^{15}N isotopes will become heavier, and (3) the degree of aromaticity of organic matter will increase. Thus, the aims of this study are: (i) to examine the influence of the heating to

the boreal forest soil organic horizons through conducting a lab-simulation experiment, and (ii) to unravel the role of fire for the organic horizons.

2. Materials and Methods

2.1. Study Area and Combustion Experiment Description

Fires in boreal forests affect large areas and lead to substantial changes of soil and organic horizon properties [22]. The main fire induced changes in soil organic horizons can be identified by studying typical boreal forests. The studied pine and spruce conifer forests are typical for boreal landscapes with a typical composition of organic components [40,41]. The experiment was conducted for samples from the northeast of the European part of Russia (61°40' N, 50°48' E). Samples of the organic horizons were collected in the middle boreal (middle taiga) vegetation zone. The climate is temperate with relatively cold summers and cold, snow-rich winters (Köppen "Dfc") [42]. Modern average annual temperatures are from 0 to −2 °C, and the mean precipitation is 600–800 mm per year [43]. The altitude ranges from 100 to 150 m above sea level with the lowest elevation in the gently undulating eastern part of the study area.

The vegetation of the study area is represented by lichen pine forest (PF) (*Flavocetrario-Pinetum* association) and green feathermoss spruce forest (SF) (*Piceetum hylocomium* association). The studied lichen pine and green moss spruce forests are located on underlying soils typical of boreal forests, on Podzols and Retisols, respectively. The ground vegetation layers are characterized by the predominance of green mosses and lichens: *Pleurozium schreberi* (Willd. ex Brid.), *Hylocomium splendens* (Hedw.), *Cladonia stellaris* (Opiz), *Cladonia arbuscula* (Wallr.) etc. The herbaceous shrub layer is distinguished by the constant presence of *Vaccinium uliginosum* L., *V. myrtillus* L., etc.

Two extensive forests (a pine and a spruce one) were selected for sampling. These forests were selected as typical features of the boreal landscape having undisturbed organic horizons. The organic horizon samples were collected in a multiple site sampling approach at the end of the vegetation season. At each forest site organic horizon samples with an area of 20 × 20 cm were taken in 15 replications. The distance between samples varied from 2.5 m to 20 m by randomization. The randomization was based on an indiscriminate selection of organic horizon samples in the study area. In this paper, organic horizons (O) are soil layers with a high percentage of organic matter. Typically, in boreal forest area there are three distinct organic layers: one of leaves, pine needles and twigs (O_i); underlain by a partially decomposed layer (O_e); and then a very dark layer of well decomposed humus (O_a). The samples had been brought into the air-dry condition before they were split into subhorizons O_i , O_e , and O_a (Figure 1). The subhorizon O_i for each of the forest litters was disassembled into fractions to reveal the mass fraction of individual components. Soil samples were separated and homogenized, sieved (2 mm) and stored at room temperature till the laboratory analysis.

Under pine forest, the O_i (fresh litter subhorizon) layer with thicknesses between 1 and 4 cm was composed of recent, poorly transformed vegetation remains. Underneath the O_i , O_{e+a} subhorizons (1–4 cm thickness) were observed, mainly formed by fragmented plant residues that were, generally, of coniferous-lichen origin including well decomposed vegetation remains. In the pine forest, O_e and O_a are combined into one due to the impossibility of accurately separating the subhorizons. The organic horizons of spruce forest displayed different structures with thinner O_i subhorizon (1–2 cm thickness), followed by potent and thicker O_e and O_a subhorizons (1–3 cm). Lower subhorizon O_a consisting of well-decomposed organic matter (1–3 cm).

Organic horizon samples were placed in porcelain crucibles and covered with aluminum foil. Each of the porcelain crucibles was oven-heated for 3 h in ashing furnace LV9/11 P330 (Nabertherm, Lilienthal, Germany) equipped with a thermometer at a specific temperature (t): 200, 300 and 500 °C. A heating time of 3 h was selected in accordance with recent studies [34,44]. The greatest transformation is generally experienced under conditions of steady surface fires with great masses of large wood debris, which are charac-

terized by a long time of burning. It is known that the temperature under these conditions may reach 500–700 °C [45]. Therefore, we selected 200, 300 and 500 °C temperature values in order to simulate low intense fire to be able to observe changes in the forest litter.

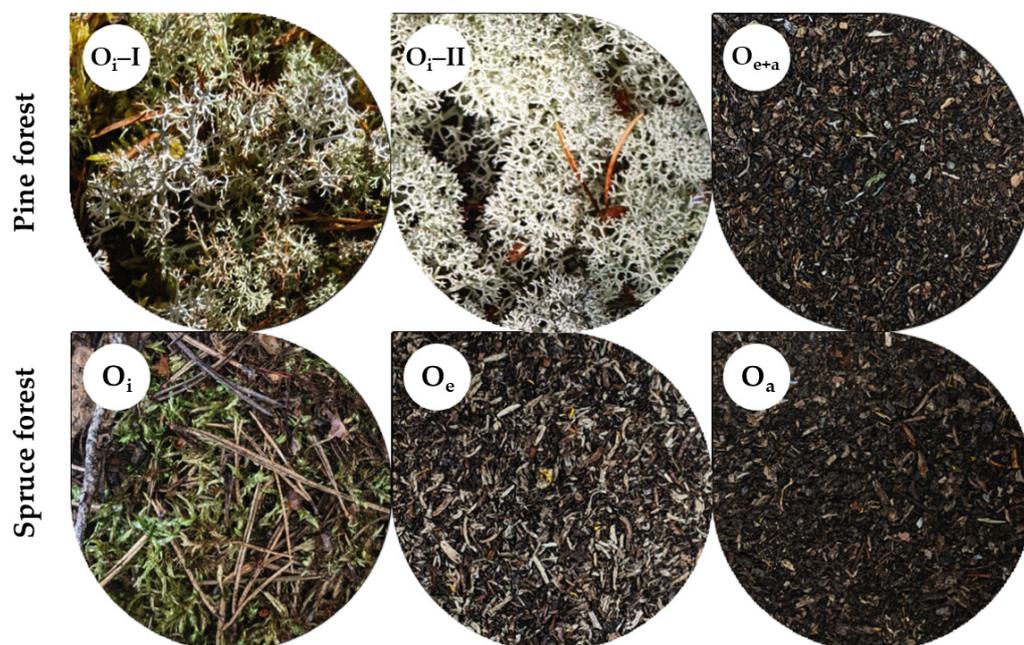


Figure 1. Photographs of the O-layers that were sampled in the lichen pine (**up**) and green moss spruce (**down**) forests. Abbreviations: O_i , O_e , O_a —subhorizons of the studied organic horizons.

2.2. Organic Horizons Analysis

2.2.1. General Analysis

The study samples of forest litters were divided into subhorizons. Litters from pine (PF) and spruce (SF) forests were divided into O_i , O_{e+a} and O_i , O_e and O_a layers, respectively. The subhorizons were separated according to the degree of decomposition of organic matter. At each site litter layers O_i were divided into plant fractions. As a result, we have received the fractional content of plant remains by weight. The weight loss was calculated in the dry condition of organic horizons for various thermal conditions: 200, 300 and 500 °C.

Chemical analyses of the litters were performed in the certified Ecoanalytic laboratory of the Institute of Biology (Komi Science Center, Urals Branch of the Russian Academy of Sciences) (certificate ROSS RU.0001.511257 from September 2019) and Soil science department. The pH values were determined on an HI2002-02 edge series pH meter with a Hanna digital pH electrode (Hanna Instruments, Romania) at a soil:water ratio of 1 (m):25 (v) for initial and pyrogenic horizons, respectively. Specific electrical conductivity (SEC) aqueous extract was measured in a water-solution, which is prepared from 1 g of soil sieved through a 2-mm mesh mixed with 25 mL of distilled water with a litter:water (1:25) ratio and held for 24 h. Measurement of specific electrical conductivity (SEC) was performed with an FG3 conductometer (Mettler-Toledo, Switzerland). Filtration of litters was done immediately after shaking with hydrophilic regenerated cellulose (pore size of 0.45 μm , Sartorius, Germany). Water-soluble (WS) carbon (CWS), inorganic carbon (IC) and nitrogen (NWS) were assessed using the TOC-VCPN analyzer (Shimadzu, Japan) with TNM-1 module. CWS and IC were measured using the method of thermal catalytic oxidation with non-dispersive IR registration.

2.2.2. Specific Surface Area by N_2 Gas Adsorption

Specific surface area (SSA) of the samples was tested by conduction using a surface-area analyzer (Sorbtometr-M, Katakona, Novosibirsk, Russia) with N_2 gas as an adsorbate at the Faculty of Soil Science, Lomonosov Moscow State University (Moscow, Russia). All

samples were kept in a vacuum chamber at 105 °C for at least 24 h before analysis to remove hygroscopic moisture. Directly before analyses the samples were heated (30 min) in a helium stream at a temperature of 100 °C to remove absorbed gases and vapors from its surface. The SSA analysis operation is based on the thermal removal of nitrogen from the surface of the samples under dynamic conditions. Sorption was carried out at a boiling point of liquid nitrogen of 77 K and a relative pressure $P/P_0 = 0.20$ (single-point method). Surface area was calculated by BET adsorption theory [46].

2.2.3. Isotopic Analyses

The stable isotope ratios $^{13}\text{C}/^{12}\text{C}$ and $^{15}\text{N}/^{14}\text{N}$ were determined by an IsoPrime 100 isotope ratio mass-spectrometer (IsoPrime Corporation, Cheadle, UK) and vario ISO-TOPE cube elemental analyzer (Elementar Analysen systeme GmbH, Hanau, Germany). Stable isotope compositions are reported in delta notation ($\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ ‰) relative to Vienna Pee-Dee Belemnite (VPDB) for C, using the international reference materials IAEA-CH-6 (-10.449 ± 0.033 ‰ VPDB), IAEA-CH-3 (-24.72 ± 0.04 ‰ VPDB) as standards, and relative to atmospheric N_2 for N, using IAEA-N-2 ($+20.3 \pm 0.2$ ‰ air N_2) and USGS-25 (-30.41 ± 0.27 ‰ air N_2) as standards according to Equation (1):

$$\delta_{\text{sample}} = \left(\frac{R_{\text{sample}}}{R_{\text{standard}}} - 1 \right) \cdot 1000, \quad (1)$$

where R represents the ratio of $^{13}\text{C}/^{12}\text{C}$ or $^{15}\text{N}/^{14}\text{N}$, respectively. The measurement error of $\delta^{15}\text{N}$ was approximately 0.2‰ and 0.1‰ for $\delta^{13}\text{C}$.

2.2.4. ^{13}C NMR Spectroscopy

Solid-state ^{13}C cross polarization with magic angle spinning (CP/MAS) nuclear magnetic resonance (NMR) spectroscopy was used to analyze the composition of the organic matter. In the present work, this is used to characterize the pyrolyzed materials, analyse their content in aliphatic and aromatic compounds. Before ^{13}C NMR analysis, combined samples were treated with 10% hydrofluoric acid, as described by Goncalves et al. [47], to remove paramagnetic iron, which strongly reduce the signal-to-noise ratio of the spectra. Solid-state ^{13}C NMR spectra of the organic horizons were recorded on a 100.53 MHz Bruker Avance III 400WB (Bruker, Ettlingen, Germany) with a rotation frequency of 12.5 kHz, a contact time of 5 ms, and a 2-s recycle delay at the resource center of the research park “Magnetic Resonance Research Methods” of Saint-Petersburg State University, Russia. Chemical shifts of fractions were determined in relation to a tetramethylsilane (TMS) shift (0 ppm). The contribution of main carbon forms to the total spectral intensity was determined by integration of the corresponding chemical shift regions according to [48–50]: 0 to 45 ppm (alkyl C), 45 to 110 ppm (O-alkyl C, subdivided in methoxyl/N-alkyl C, 45–60 ppm; O-alkyl C, 60–90 ppm; di-O-alkyl C, 90–110 ppm), 110 to 165 ppm (aryl C, subdivided in aromatic C–H and C–C, 110–140 ppm; aromatic O-substituted C, 140–165 ppm), 165 to 185 ppm (carboxyl C); aldehydes or ketones, 185 to 220 ppm. The total content of aryl C was calculated as the sum of the signals at 110–165 ppm fields. Signals from alkyl C were recorded in the 0–110 ppm range. The degree of aromaticity (fa) was determined as proportion of total content of aryl C components (110–145 and 145–165 ppm) on total C (excluding the contribution in the range of 165–220 ppm).

2.2.5. Statistical Analyses

The effects of fire on the forest litter were analyzed separately for each organic sub-horizon. Statistical processing of the data was carried out using the Excel 2010 (Microsoft, Redmond, WA, USA). Correlation analysis was used to determine the relationship between the temperature effect and the change in the organic horizon properties. Correlation coefficients (r-Pearson) were calculated using the STATISTICA 10.0 (Stat. Soft Inc., Tusla, OK, USA); differences were considered significant at the significance level $p < 0.05$. Here we

used testing correlation for the significance of all analyses but ^{13}C NMR. All data presented here are the mean values of three replications, except for ^{13}C NMR spectra (per sample) because of its high cost and time needed to obtain the spectra.

3. Results

3.1. Fractional Composition

The experiment was conducted for all organic subhorizons (O_i , O_e , O_a), but at the same time samples with a different fraction content were used for O_i subhorizon of forest litters (Table 1). In pine forest we had studied two types of O_i subhorizon samples. In the first one, the proportion of lichens was 22% (nature content), in the second, 51% (with the artificial lichen addition). Two types of organic subhorizon O_i in the pine forest were highlighted to distinguish an area where most lichens occur. The spruce forest sample with naturally occurring moss content of 45% was studied. Pine forest litter samples are characterized by a high proportion of lichens, while spruce forest litter samples are characterized by a high proportion of moss remains.

Table 1. Components of O_i subhorizons (% , mas.) ($n = 15$).

Litter Type	Moss	Lichen	Foliage	Needles	Bark	Branch
Pine forest litter O_i -I	31 ± 0.4	22 ± 0.4	0	21 ± 0.1	12 ± 0.1	14 ± 0.3
Pine forest litter O_i -II	19 ± 0.2	51 ± 1.0	0	13 ± 0.1	8 ± 0.1	9 ± 0.2
Spruce forest litter O_i	45 ± 0.4	0	18 ± 0.2	13 ± 0.1	9 ± 0.2	15 ± 0.2

3.2. General Properties and Specific Surface Area

One of the most substantial changes in the organic horizon samples during heating is the loss of mass. In the lichen pine forest, the average weight loss was 10, 40 and 59% at 200, 300 and 500 °C, respectively (Figure 2). In the green moss spruce forest, the average weight loss was 12, 34, and 52% at 200, 300, and 500 °C. Comparing the initial samples, the most substantial decrease in mass was observed at 300 °C, and further heating up to 500 °C led to a relatively smooth decrease in the amount of organic matter °C. The loss of mass of organic matter is obvious and significant ($r = 0.95$, $p < 0.05$). It must be noted that the mass loss is slightly different for the organic horizons of different forest types. The differences are likely to be related to the chemical composition. A minor morphological change occurs until 200 °C. The organic horizons are becoming semi-brown (color). At the temperatures of 300 and 500 °C organic matter is becoming black.

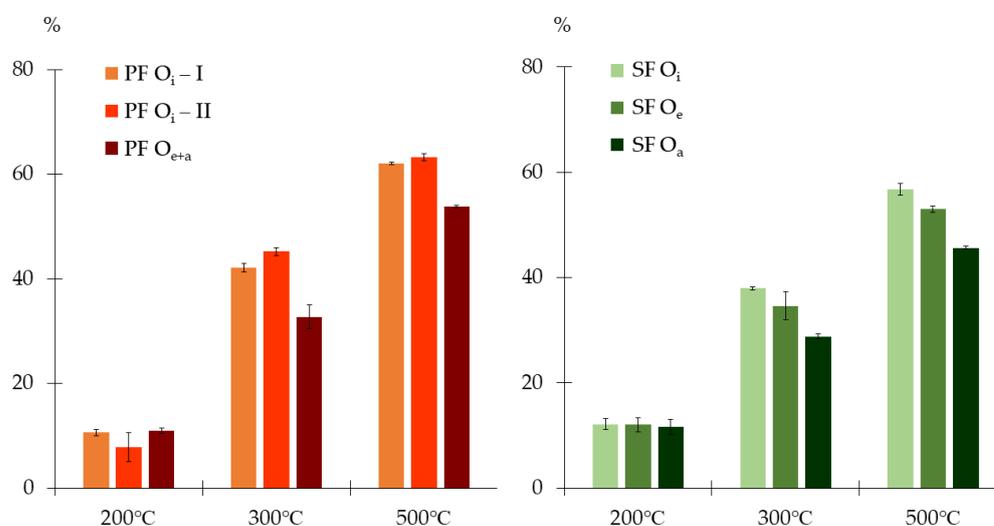


Figure 2. The values of mass loss (%) in organic horizons. Abbreviations: PF—pine forest litter, SF—spruce forest litter, O_i , O_e , O_a —subhorizons of the studied organic horizons ($n = 3$).

It is shown that an increase in temperature leads to an increase in the pH values of the water extracts (Figure 3). The highest increase of pH values is observed in the upper subhorizons (O_i), in contradistinction to the lower subhorizons (O_e , O_a). The pH values increased in pine and spruce forests organic horizons from ~ 5.0 (initial) to ~ 9.1 (500°C) and from ~ 5.0 (initial) to ~ 7.4 (500°C), respectively. The testing for significance revealed strong positive correlation between temperature increasing and pH changing ($r = 0.88$, $p < 0.05$).

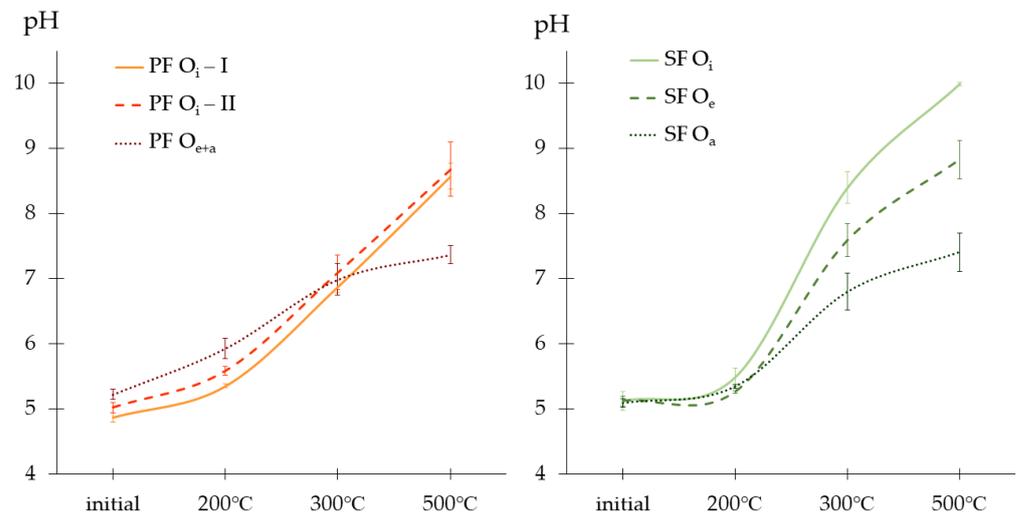


Figure 3. The changes in the pH values (mean \pm standard error) of the water extracts of the litters under heating from 0 to 500°C . Abbreviations: PF—pine forest litter, SF—spruce forest litter, O_i , O_e , O_a —subhorizons of the studied organic horizons ($n = 3$).

The SEC of the water extracts of the forest litters increased with a rise in the temperature of burning (Figure 4). In the PF litter at 500°C , its values were maximal. In the spruce forest litters at 200°C , specific electrical conductivity values were maximal. The further heating to 300°C decreased the specific electrical conductivity, however at 500°C its values become high again. The maximum increase in electrical conductivity by about 2 times was revealed in forest litter samples. In accordance with the test for significance, no correlation was revealed between the increased temperature and changed SEC ($r = 0.23$, $p < 0.05$).

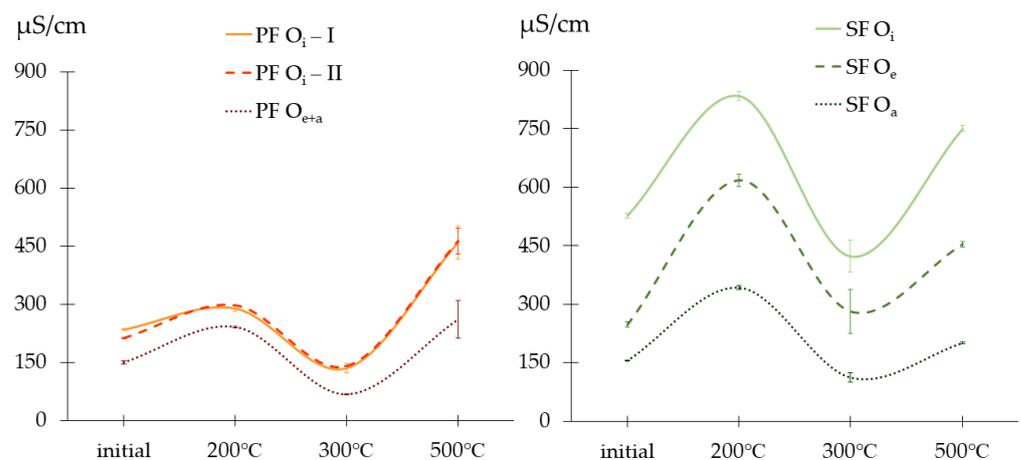


Figure 4. The SEC values (mean \pm standard error) changing during combustion experiment (Abbreviations: PF—pine forest litter, SF—spruce forest litter, O_i , O_e , O_a —subhorizons of the studied organic horizons ($n = 3$)).

The SSA as determined by low-temperature N_2 adsorption of the organic horizons was low due to the dominance of quite intact litter in pine ($\sim 0.32 \text{ m}^2 \text{ g}^{-1}$) and spruce

($\sim 0.51 \text{ m}^2 \text{ g}^{-1}$) forests, and gradually increased with temperature increasing. SSA of organic horizons changed to $\sim 1.8 \text{ m}^2 \text{ g}^{-1}$ in the pine forest and to $1.9 \text{ m}^2 \text{ g}^{-1}$ in the spruce forest (Figure 5). The most substantial changes were observed in O_e and O_a subhorizons. In all studied samples, the influence of high temperatures led to an increase in the values of the specific surface area (SSA), on average, of more than two to five times compared with the initial values (Figure 5). The test for significance revealed that the SSA values correlate (strongly) with temperature increase ($r = 0.73$, $p < 0.05$).

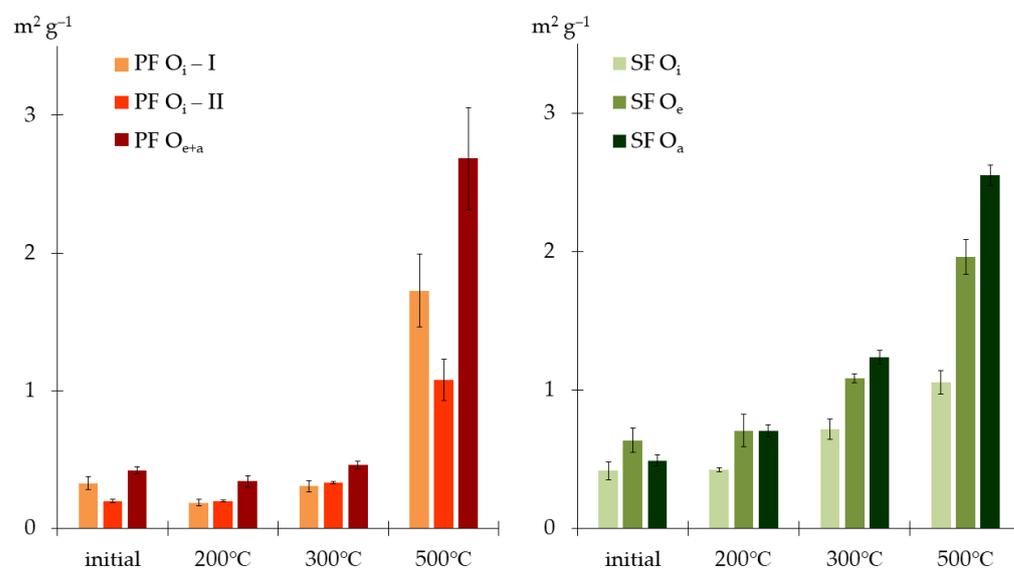


Figure 5. SSA values (mean \pm standard error) changing of litter samples after oven-heating. Abbreviations: PF—pine forest litter, SF—spruce forest litter, O_i , O_e , O_a —subhorizons of the studied organic horizons ($n = 3$).

3.3. Carbon and Nitrogen Content

The content of carbon and nitrogen in the sample remains increases with an increase in the influence of temperatures. But the total content of carbon (C_{tot}) and nitrogen (N_{tot}) decreases considering the initial mass of the sample and its loss during combustion. The highest decrease of C_{tot} and N_{tot} was revealed at a temperature of 500°C (Figure 6). The proportion of C_{tot} content in the lichen pine forest organic horizon samples decreased on average by $\sim 41\%$. The proportion of C_{tot} content in the green moss spruce forest organic horizon samples decreased on average by $\sim 32\%$. The proportion of N_{tot} content in the lichen pine forest organic horizon samples decreased on average by $\sim 0.76\%$. The proportion of N_{tot} content in the green moss spruce forest organic horizon samples decreased on average by $\sim 1.1\%$. The temperature increase correlates strongly with the loss of C_{tot} and N_{tot} content ($r = 0.9$, $p < 0.05$ and $r = 0.89$, $p < 0.05$, respectively).

The organic carbon content of the water-soluble fraction after pyrogenic exposure can substantially increase and reach maximum values at 200°C (up to 25 mg C/g), further decreasing with increasing temperature. The content of N_{WS} in the litter is characterized by a maximum in the initial samples (Figure 7).

The water-soluble C and N content decreases by 10 to 34 times, depending on the type of forest litter and burning time. It is shown that the concentration of nitrogen in the litter of the pine forest at 200°C is much lower than in the litter of the spruce forest. The upper subhorizons (O_i) of the pine forest litter have substantially lower levels of N_{WS} , resulting in an increased ratio of C_{WS} to N_{WS} at 200°C . Here we revealed a strong negative correlation between temperature increase and C_{WS} and N_{WS} content ($r = -0.67$, $p < 0.05$ and $r = -0.76$, $r < 0.05$, respectively).

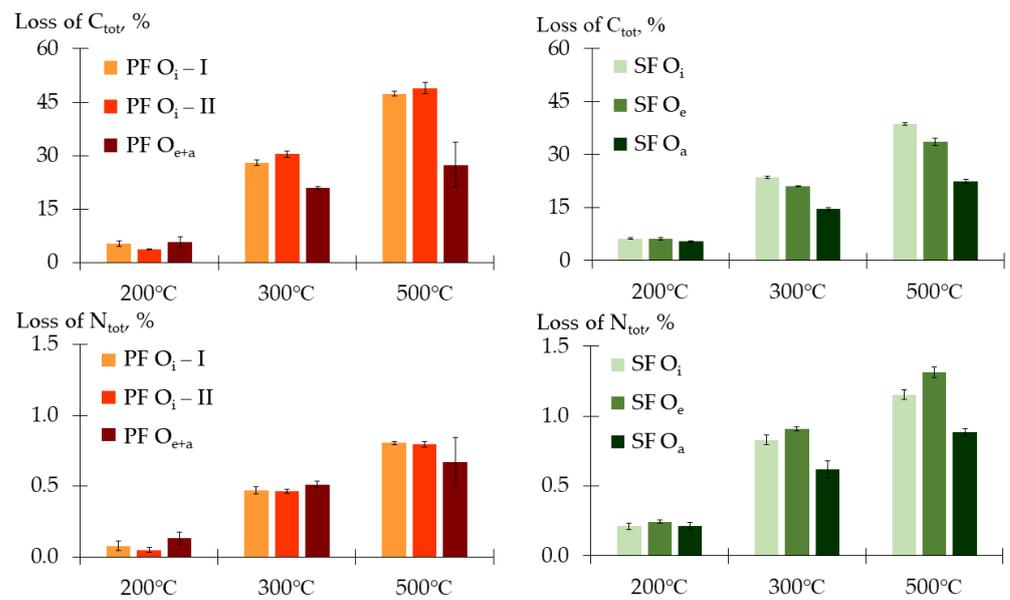


Figure 6. Proportion of total C_{tot} and N_{tot} loss (%) in organic horizons (considering the mass lost). Abbreviations: PF—pine forest litter, SF—spruce forest litter, O_i, O_e, O_a—subhorizons of the studied organic horizons.

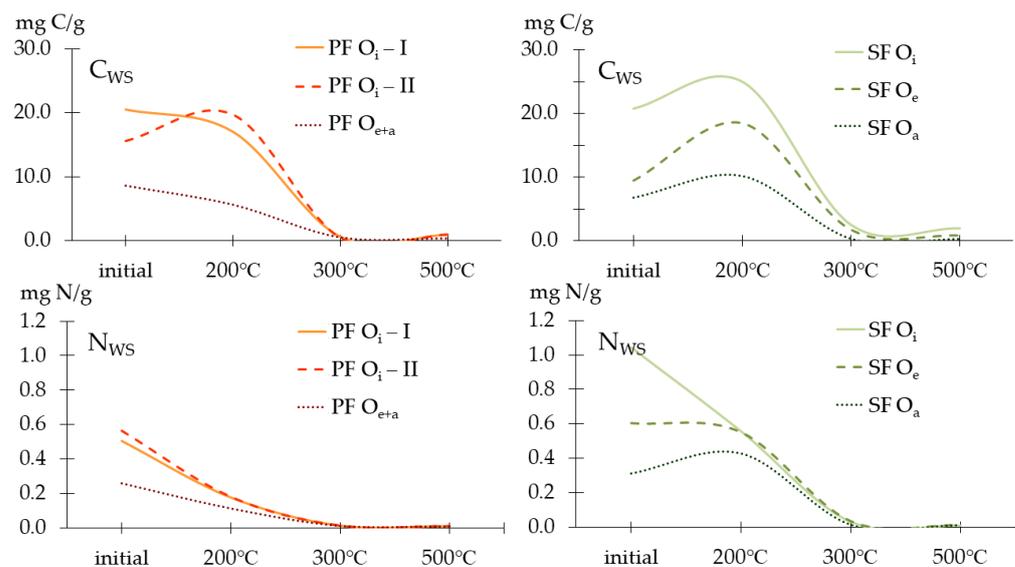


Figure 7. The changes in total water-soluble carbon and nitrogen content. Abbreviations: PF—pine forest litter, SF—spruce forest litter, O_i, O_e, O_a—subhorizons of the studied organic horizons.

The content of inorganic carbon in the form of carbonates and bicarbonates, coal particles formed after fires increases. The heating of the litter led to an increase in the concentration of inorganic water-soluble carbon (IC), especially in the upper subhorizons. IC concentration increased 69 to 715 times. The lower subhorizons of the litters are characterized by a lower content of IC. The test for significance revealed a strong positive correlation between temperature increase and IC content change ($r = 0.65$, $p < 0.05$).

Stable $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ isotope values in organic horizons displayed minor change under the exposure to fire (Figure 8). Initially, the values of $\delta^{13}\text{C}$ isotopes in the soil organic horizons of lichen pine forest were -28.5% in O_i-I, -28.06% in O_i-II, and -28.67% in O_{e+a}. In turn, in the soil organic horizons of green moss spruce forest, the initial values of $\delta^{13}\text{C}$ isotopes were -30.34% in O_i, -29.59% in O_e, and -28.59% in O_a.

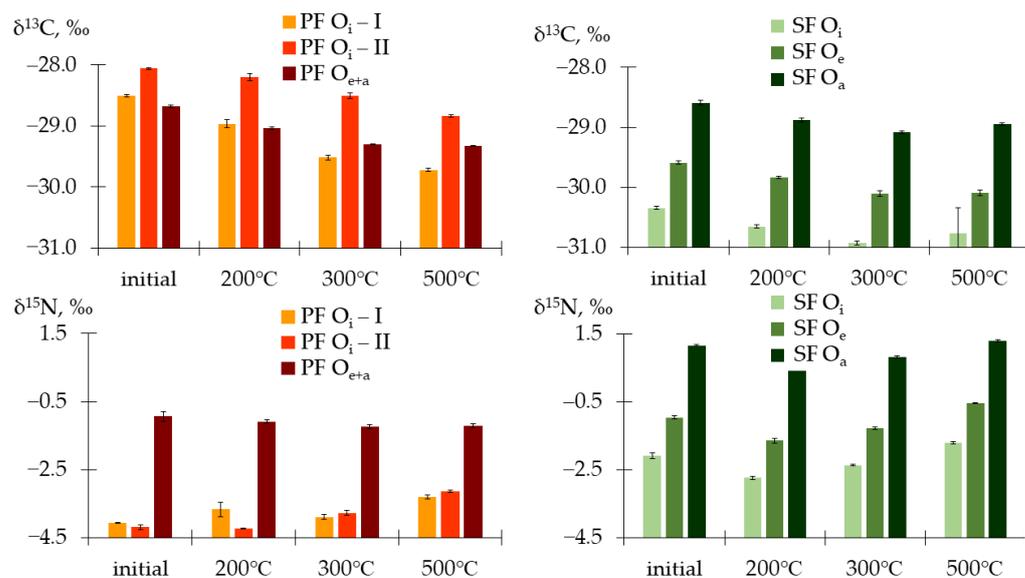


Figure 8. Stable-carbon ($\delta^{13}\text{C}$) and stable-nitrogen ($\delta^{15}\text{N}$) isotope values of study litter samples (sampled in pine and spruce forests). Abbreviations: PF—pine forest litter, SF—spruce forest litter, O_i , O_e , O_a —subhorizons of the studied organic horizons ($n = 3$), (mean \pm standard error).

The values of $\delta^{15}\text{N}$ isotopes in the initial samples of the lichen pine forest were -4.07% , -4.19% , and -0.94% in $\text{O}_i\text{-I}$, $\text{O}_i\text{-II}$, and O_{e+a} , respectively. The values of $\delta^{15}\text{N}$ isotopes in the initial samples of green moss spruce forest were -2.08% , -0.96% , and 1.16% in O_i , O_e , and O_a . With the temperature increasing, we observed $\delta^{13}\text{C}$ isotope values show minor gradual decreasing by an average of 0.8, and of 0.4 in the pine and spruce forest litters, respectively. Only for spruce forest O_i and O_a subhorizons, minimal $\delta^{13}\text{C}$ content was at 300 degrees. Combustion of spruce forest organic horizons leads to fire-induced changes in the $\delta^{15}\text{N}$ isotopic composition, namely, an increase in the content of $\delta^{15}\text{N}$ in forest litter samples at 500 degrees, but its decrease at 200 and 300. The content of $\delta^{15}\text{N}$ in the pine forest varies according to subhorizons. We identified that the increasing of temperature lead to changed $\delta^{15}\text{N}$ values: in $\text{O}_i\text{-I}$ and $\text{O}_i\text{-II}$ gradually increasing, but decreasing in O_{e+a} . The correlation between the increased temperature and changed $\delta^{13}\text{C}$ values is negative and quite moderate ($r = -0.31$, $p < 0.05$). The correlation between the increased temperature and changed $\delta^{15}\text{N}$ values is positive and weak ($r = 0.09$, $r < 0.05$).

3.4. CP/MAS ^{13}C NMR Data

The content of dominant molecular fragments in the organic matter of bedding differs between initial and subsequent heating in all types of forest litters. These results are provided in Table 2 and Figure A1.

As a result of ^{13}C NMR analysis, it was revealed that the initial soil organic horizons samples are characterized by a high proportion of aliphatic compounds. As a result of the experiment, it was revealed that the effect of temperature in the litter samples of the lichen pine forest and green moss spruce forest led to the transformation of litter organic matter (OM). Regardless of the fact that individual subhorizons have a different fractional composition with different mass fractions of individual fractions, the results of changes in OM are similar. A direct relationship between an increase in the combustion temperature and an increase in the proportion of degree of aromaticity (fa) with significant positive correlation ($r = 0.9$, $p < 0.05$) was revealed. The maximum content of aromatic compounds was found at a temperature of 500 °C.

Table 2. Percentage distribution (%) of signal intensity between selected chemical shift regions (ppm) of CP/MAS ¹³C NMR spectra.

Sample	Alkyl C	O-Alkyl C			Aryl C		Carboxyl C/ Amide/Ester		Alkyl/ O,N-Alkyl	fa	AR/AL
		Methoxy/ N-Alkyl	O-Alkyl	di-O-Alkyl	Aromatic	Phenolic	Carboxyl/ Amide	Aldehyde/ Ketone			
Pine forest organic horizons (O _i -I)											
initial	15.9	4.1	45.9	11.2	10.3	5.4	4.3	2.9	0.3	15.7	0.2
200 °C	21.0	6.1	43.6	10.3	11.1	5.2	2.7	–	0.4	16.3	0.2
300 °C	31.7	1.2	–	4.3	48.0	14.4	0.4	–	5.8	62.4	1.7
500 °C	6.5	1.3	4.6	2.5	78.2	5.9	1.0	–	0.8	84.1	5.6
Pine forest organic horizons (O _i -II)											
initial	17.1	3.9	56.2	12.3	5.2	1.9	3.3	–	0.2	7.2	0.1
200 °C	17.2	4.72	49.8	11.6	9.3	4.0	3.2	0.2	0.3	13.3	0.2
300 °C	25.2	3.8	0.4	3.4	44.5	15.7	4.1	3.0	3.3	60.2	1.8
500 °C	4.1	–	–	2.0	81.1	9.2	1.7	2.0	2.1	90.3	14.7
Pine forest organic horizons (O _{e+α})											
initial	25.1	6.0	30.3	8.1	14.0	7.4	6.1	3.1	0.6	21.3	0.3
200 °C	26.3	4.5	1.7	19.1	23.5	21.5	3.4	–	1.0	45.0	0.9
300 °C	31.9	2.8	1.9	6.5	44.2	11.9	0.9	–	2.8	56.0	1.3
500 °C	10.6	0.3	0.9	2.5	78.5	7.2	–	–	2.9	85.7	6.0
Spruce forest organic horizons (O _i)											
initial	23.5	7.6	36.9	9.9	11.4	5.2	4.9	0.6	0.3	16.7	0.2
200 °C	23.2	6.4	26.5	8.1	19.3	8.9	5.8	1.7	0.6	28.2	0.4
300 °C	26.3	3.4	1.5	5.3	45.5	14.2	3.1	0.8	2.6	59.7	1.6
500 °C	10.4	1.2	2.7	2.9	76.1	6.7	–	–	1.5	82.8	4.8
Spruce forest organic horizons (O _e)											
initial	22.4	7.3	33.1	9.3	11.7	6.6	7.2	2.3	0.4	18.3	0.3
200 °C	23.7	6.5	21.8	7.0	21.5	10.3	6.7	2.3	0.7	31.9	0.5
300 °C	26.0	3.1	2.7	5.6	44.5	14.9	2.4	0.7	2.3	59.5	1.6
500 °C	5.7	0.5	–	2.1	81.7	8.4	0.8	0.7	2.2	90.1	10.8
Spruce forest organic horizons (O _α)											
initial	25.0	5.4	30.0	9.2	16.0	7.2	7.0	0.1	0.6	23.2	0.3
200 °C	29.5	6.1	17.2	6.3	21.8	9.8	7.0	2.2	1.0	31.6	0.5
300 °C	34.3	2.4	–	3.2	37.5	16.1	3.0	3.6	6.2	53.5	1.3
500 °C	10.4	–	2.5	1.7	72.1	11.0	1.9	0.5	2.5	83.1	5.7

– not found; fa—degree of aromaticity.

The content of dominant molecular fragments in the organic matter differs between initial and after-combustion in the litter samples from both types of forests. All initial samples are characterized by three major peak values of ¹³C NMR data in the ranges of 0–45, 60–95 and 95–110 ppm. Peak values in the range of 0–45 ppm are represented by a high content of alkyl. High content of C related to aliphatic fragments double-replaced by heteroatoms (including carbohydrate) and methylene carbon of ethers and esters was demonstrated by 60–95 and 95–110 ppm ranges. A small pick of carboxyl groups, amides, and their derivatives was demonstrated by 165–185 range. After-combustion samples in turn demonstrated one wide peak in the 110–165 range characterized by aryl fragments. A similar shift is reliably observed in all the samples studied.

4. Discussion

4.1. Direct Effects of Fire on General Properties

The weight loss at 200 °C is associated with dehydration, then a significant weight loss at 300 and 500 °C is explained by the destruction of organic matter [51]. It is known that a large part of carbon can be lost at lower temperatures and organic matter can commonly volatilize at temperatures between 200 and 315 °C [52]. It was revealed that the greater weight loss is typical for the upper subhorizons of the litter. Organic carbon is known to burn out most severely at a temperature of about 500 °C [53].

The experiment was conducted under certain conditions such as synchronous occurrence of fire, identical temperature conditions (at room temperature, 200, 300 and 500 °C) and time duration (3 h) for all the studied organic horizons. This made it possible to

correctly compare different analysis results. It is widely known that upon heating, physico-chemical changes occur, the organic material is exposed to distillation, destruction, charring (formation of “black carbon”), and complete oxidation up to CO₂ and H₂O according to the temperature and concentration of oxygen in the environment [54–56]. Organic matter decomposes incompletely as a result of less severe fires [57]. First months after the fire, the products of partial pyrogenic decomposition of organic horizons can be transferred into mineral horizons [22,56]. Thus, it is essential to understand how changes in organic horizon properties caused by combustion will affect the properties of the underlying mineral soils.

The main changes at lower temperatures (below 200 °C) affect mostly biological properties [58], physical properties such as soil water repellency and aggregate stability can also be altered [59]. Changes in organic horizons from low-intensity fires are known to stimulate further microbial growth [60]. At higher temperatures (above 200 °C), properties are affected through combustion of SOM and production of pyrogenic organic compounds and increases in soil pH. Already at 300 °C and even more so at 500 °C the samples turn black, there is a strong transformation and destruction of the organic material of forest litters.

With an increase of temperature, a transition of pH values to the alkaline region is observed, which matches with Certini’s review [15]. Most likely, this is due to the destruction of the organic material of the organic horizon and their ashing [61]. As a result of fires in forests an increase in pH values occurs due to an increase of carbonates proportion [62] and it is likely because of volatilization of low-molecular organic compounds, particularly acids. Probably, a strong transition to the alkaline direction in the temperature range of 300–500 °C is due to the denaturation of organic acids and the release of bases. The results obtained are comparable with the results of a similar experiment [63]. If the burnt matter of organic horizons will be transferred to the mineral horizons, it will probably result in substantial changes, at least temporarily. Increase in alkalinity values is likely to improve the quality of the studied soils [64], given that the underlying soils under study are mostly acidic [2].

The correlation of the specific electrical conductivity (SEC) of soils and organic horizons with their properties affecting the crop productivity is widely recognized [65,66]. The measurement of SEC provides information about soil properties such as texture, drainage conditions, cation exchange capacity and others [65,67]. The SEC of soils determine the salinity of soils and thus their ability to transmit electricity in an aqueous solution. An increase in SEC values, together with the rising of pH from acidic to alkaline as a result of fire, will lead to increased decomposition and a more saturated metabolism in soils in the future [68]. The results of our experiment led to similar changes in organic horizons as a result of fires. These results are corresponding to literature data [51,63,69]. The results obtained suggest an increase of the specific electrical conductivity role in the water-soluble organic matter of the organic horizons when high temperatures occur. It is possible that burnt material of organic horizons transferring down will change the properties of mineral horizons. It is shown that in samples of spruce forest organic horizons, the SEC is higher than in the pine forest organic horizon. The differences between SEC in the studied organic horizons of forests are likely to be caused by the predominance of moss and foliage amount in the fractional composition of the spruce forest organic horizons.

Specific surface area (SSA) plays an important role in processes of soil organic horizons, including substances sorption, water-retention capacity and chemical reaction processes. The soil sorption capacity can change depending on organic matter structure and content in soils. It is likely this is due to SSA and general soil physical property relationships. The total SSA is a factor that can relate a texture of medium with physical and chemical properties. The result of our studies was a significant increase in SSA values as a result of fire-induced transformation of organic horizon materials. The sorption and respiration capacity of mineral horizons are likely to increase due to the mixing with SSA rich organic horizons. During fire events, organic matter undergoes a series of changes which include free vaporization of moisture at 100 °C, degradation and decomposition of lignin and

hemicellulose between 130 °C and 200 °C, chemical dehydration of cellulose at 280 °C, burning at 500 °C [70,71]. Most likely, this is associated with the formation of nanosized pores with a large surface area in the content of charred organic horizons.

It is shown that as a result of the influence of temperature, the changes in the studied parameters discussed above have similar regularities. Supposedly, all of the above discussed changes in soil properties will lead to a partial increase of soil quality, which may favorably affect the restoration of above-soil vegetation and the restoration of the organic horizons.

4.2. The Impact of Fire on the Soil Organic Matter

The fire to varying degrees affects all examined parameters of soil organic horizons in lichen pine and green moss spruce boreal coniferous forests. Investigated parameters were found to be substantially dependent on the temperature of heating of forest soil organic horizons. It is widely known that the remains of burnt organic material transfer down into the subsoil layers [14]. Thus, changes in organic horizon properties resulting from combustion to a high probability will affect the properties of the underlying mineral horizons of soils.

Organic content of C and N are often featured as one of the central focuses in the study of soils because of the important role in SOM [13,15,63]. Organic horizon mineralization determines the balance of carbon and nitrogen in soils [72,73]. The highest concentrations of organic carbon and nitrogen involved in the cycle are concentrated precisely in the forest soil organic horizons. Fires can greatly affect this balance [2,74]. It is evident that the carbon proportion loss is one of the most considerable changes in the soil organic horizons upon heating. Thus, the conducted studies have shown that, against the background of the pyrogenic factor, there are changes in the properties of soil organic horizons, such as the loss of the carbon content. The degree of transformation depends on the intensity of the fire: the greatest changes can be traced at 500 °C. It was found that the loss of nitrogen was less intense than that of carbon during the combustion. Apparently, this is due to differences in the destruction of organic substances at different temperatures. The C/N ratio practically does not change with an increase in the influence of temperature, which indicates a uniform destruction of the organic material of forest litters. The only significant difference in the C/N ratio was found between the organic horizons of soils of different forest types. The C/N ratio in the lichen pine forest is on average twice as high as in the green moss spruce forest. It is likely to be due to low nitrogen content in pine forest samples. It is known that the pine forests of the study region grow on Podzol soils poor in ash elements and nitrogen [75,76].

The content of C_{WS} and N_{WS} during the heating experiment change. Following combustion, the water-soluble forms of carbon and nitrogen content decreased. These changes were caused by the fact that a substantial part of the organic matter was lost during combustion. Similar results were obtained previously for soils of pine and spruce forests of the Komi Republic [77] and larch forests of Central Siberia [63]. The decrease of water-soluble element's content is likely to be affected by the formation of charcoal that is capable of absorbing a substantial part of organic compounds [14,78,79].

According to the data of C_{WS} , the proportion of IC in the samples increases with the influence of temperature. Ash produced at high temperatures is likely to increase the soil's inorganic compounds [80]. IC formed by after-fires can increase in the solid phase to carbonate forms [77] and carbonate and bicarbonate particles interacting with moisture [80]. IC is important for many properties and processes of the carbon component of the soil [81]. The increase of IC as a result of the organic horizons combustion can lead to a rise of IC content in the mineral horizons.

Natural abundance of stable $\delta^{13}C$ and $\delta^{15}N$ isotopes is widely accepted as fingerprints to identify ecosystem processes [38,82,83]. Therefore, stable $\delta^{13}C$ and $\delta^{15}N$ isotope analysis has been used to analyze after-combustion changes in organic horizons. Variation in stable isotope values within organic horizons is associated with chemical and physical

environmental conditions and can change after wildfires [84]. As a result, the experiment clearly indicates a decrease (facilitation) of the $\delta^{13}\text{C}$ and an increase (weighting) of the $\delta^{15}\text{N}$ values. Previously, $\delta^{13}\text{C}$ stable isotopes were found to have more weight after fire in soils [77,85], which is consistent with the logic of isotope behavior [86]. Cellulose is enriched with $\delta^{13}\text{C}$ relative to lignin and lipids in organic horizons [87,88]. It is probable that the differential loss of compounds during heating could therefore explain the lower $\delta^{13}\text{C}$ value of thermally altered organic horizons. Similar results of $\delta^{13}\text{C}$ facilitation and $\delta^{15}\text{N}$ weighting in organic horizons after fire were received in the papers of Alexis et al. [89] and Wang et al. [90]. Changes of the organic matter caused by fires are likely not to lead to major changes of the stable $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ isotope [23,91,92]. The differences between types of forest soil organic horizons are visible. Organic horizons with partial content of foliage seem to have $\delta^{15}\text{N}$ depleted [93,94]. Further investigation is needed to determine changes in $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ values which may occur at different temperatures and durations that were not explored in this paper.

The relationship between thermal properties, elemental composition and chemical-shift regions in the ^{13}C NMR is a useful tool for characterizing organic matter properties [29]. In similar papers, solid-state ^{13}C nuclear magnetic resonance spectroscopy with cross polarisation-magic angle spinning (CP/MAS ^{13}C NMR) was used to obtain changes of fire-induced C structures regions in organic matter [39,89,95]. The result of our research was a substantial transformation of organic matter in organic horizons after fire. The minor differences of results in different type of forests are received. It is possible because of the differences in the composition of the organic horizons of each type of forest and their chemical degradation during burning.

A recent research suggests that low-intensity surface periodic wildfires decrease the danger of catastrophic wildfires [96], and lead to create polycyclic aromatic hydrocarbons (PAH) largely resistant to decomposition [28,97]. Where burning occurs under aerobic conditions, organic matter is completely oxidized to ash. A limited supply of oxygen in the soil during the passage of fire may be common, as indicated by the presence of pyrolysis products of organic matter in soils following burning [98]. As the heat increases during a wildfire, plant biomass is subject to chemical transformation involving the formation of aromatic ring structures and the gradual condensation of smaller aromatic units [14,99]. According to Knicker et al. [100] as a result of combustion the solid-state ^{13}C NMR spectra of the carbohydrate fraction is converted into condensed dehydrated material producing intense signals in the aromatic region. This clearly happens due to the input of charred material, whose signal is centered at ~ 130 ppm [101].

Fires with temperatures of between 250 and 460 °C could cause volatilization, charring or complete oxidation of SOM. At these temperatures, González-Pérez et al. [14] and Santín and Doerr [58] indicated that aromatic compounds accumulate at the expense of aliphatic ones. It may turn out to be the progressive transformation of the original biopolymers into condensed macromolecular aromatic substances of disordered structure [100]. Aromatic compounds are mainly formed of such compounds as lignin, cellulose and hemicellulose. Condensation of complex aromatic compounds as a result of fires can sequester the carbon compounds from the carbon cycle for a long timescale. This occurs because these organic compounds are difficult to decompose. In turn with the formation of complex structures, PAHs are formed. The toxicity of PAHs to biotic communities, including humans, is well established [102–105]. PAHs are affected by the pore size distribution in soils and the amount and nature of organic matter [106]. Hence the bioavailability and environmental persistence of PAHs are closely related by organic matter [107–109].

Boreal forest fires are known to be the main drivers of changes in soil organic compounds [110,111]. The high severity and widespread fires in the boreal region affect the global scale through the release of char and its removal [112,113]. By contrast surface and low-intensity fires create conditions where coal particles are transported over short distances and accumulate in soils [15,64,114]. Similar data on the increase in the proportion of pyrogenic materials under the combustion of peat are described earlier [115]. It

is considered that biochar formed from combustion is a suitable material for carbon storage in soils [116]. Formation of resistant organic materials is likely to be associated with cyclization of biogenic organic-N such as peptides and amino sugars [117].

5. Conclusions

(1) The experiment was conducted to determine fire effect on forest soil organic horizons of lichen pine and green moss spruce forests properties. We clearly observe fire-induced changes in forest soil organic horizons. With an increase in temperature of fire (up to 500 °C), a strong transformation of organic substances was revealed. Remains of burnt material cause concentration of charcoal particles. With an increase in the influence of temperature, the pH, the specific surface area and the inorganic carbon content increase. The aromaticity of organic matter in the organic horizons increases after the fire as well.

(2) Post-pyrogenic soil organic horizons material will penetrate the underlying soil mineral horizons. It is likely to improve soil quality. By improvement humification processes and, in addition, concentration of charcoal removed from carbon cycle are meant. In turn, burnt organic horizons are likely to lead to negative changes in soils, namely to an aromatic compound increase. The fires strongly influence soil organic horizons and therefore play an important role in soil processes. In view of the amount of carbon stored in boreal region soils and the lack of knowledge on how fire alters the soil organic horizon, further research is required.

The study of the fire influence on coniferous boreal forest soil organic horizons currently requires additional experimental and theoretical studies due to knowledge gaps. It is necessary to develop an understanding of the fire-induced processes in the organic horizons, since organic horizons, reflect the existing trends in humus formation. Boreal forests are the world's largest forests; therefore, this paper could produce important information that can be applied globally.

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Appendix A

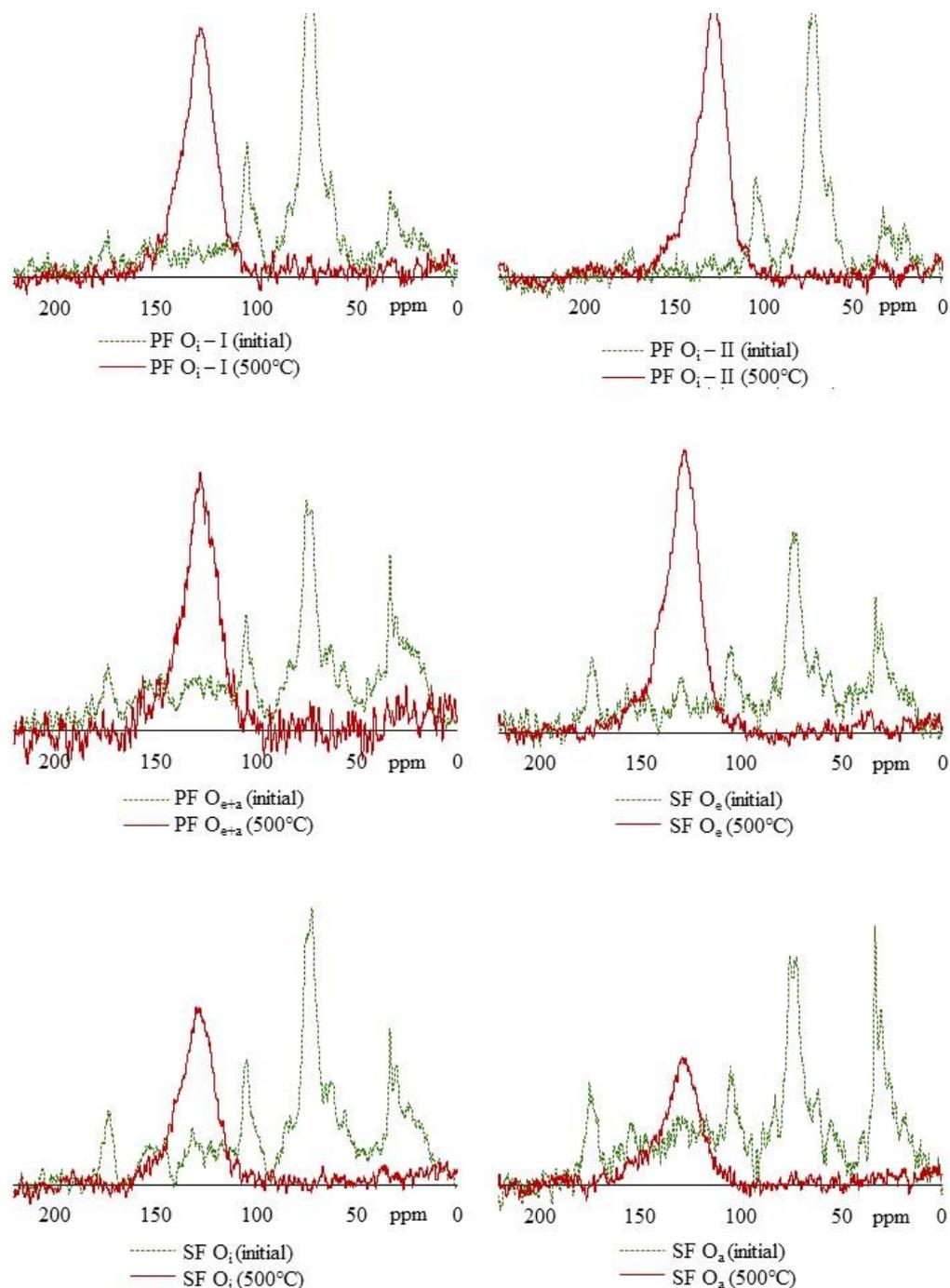


Figure A1. Changing of CP/MAS ^{13}C NMR spectra before and after combustion of studied forest litters organic matter (differences between initial and burned samples). Abbreviations: PF—pine forest litter, SF—spruce forest litter, O_i , O_e , O_a —subhorizons of the studied forest litters.

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