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Nitrogen fractions in dehydrated peat soils

Marcin Becher, Emil Żukowski*

Department of Soil Science and Agricultural Chemistry, Faculty of Natural Sciences,
Institute of Agronomy, University of Natural Sciences and Humanities in Siedlce,
14 Prusa Str., 08-110 Siedlce, Poland

*E-mail address: emillo94_15@o2.pl

ABSTRACT

The aim of the study was to determine the nitrogen speciation in organic soils under mulching, made from sedge and peat. For speciation tests, the method based on extraction with neutral reagent and two-stage acid hydrolysis allowing the separation of mineral nitrogen and organic forms: soluble, easily hydrolysable, hardly hydrolyzing and non-hydrolyzing. Characteristic for the degradation of dehydrated peats, the processes of oxygen transformation of organic matter increased the share of the most mobile soluble and easily hydrolysable fractions. The easily soluble and easily hydrolysable fraction was closely correlated with the decomposition processes of the organic matter of the studied soils. It was confirmed that a useful indicator describing the state of transformations of peat soils occurring after their dehydration, may be the ratio of the amount of nitrogen of the easily hydrolysable to the hardly hydrolysable form. The value of this ratio showed a significant linear relationship with soil characteristics characteristically variable in transformation processes of drained peat soils. It grew along with the advancement of decomposition processes of organic matter.

Keywords: peat degradation, muck, peat moss, oles peat soil nitrogen, sequential analysis

1. INTRODUCTION

As a result of lowering the groundwater level of wetlands, about 90% of non-forest peatlands in Poland have been drained. This was done for the purposes of expanding the

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production area of agriculture (drainage drainage) and hydrotechnical and industrial investment. Repealing the conservative action of water, relative to plant debris accumulated in the peat-forming process, results in degradation of peat. The most important effect of peat degradation is the mineralization of organic matter combined with the release of biogenic elements. The effects of these phenomena are the cause of eutrophication of the hydrosphere (mainly through the release of nitrogen and phosphorus compounds) and deepen the greenhouse effect (CO_2 emission) [11, 13].

Nitrogen is a basic biogenic element characterized by very complex and dynamic processes of transformation in the soil environment, which include: mineralization, immobilization, oxidation, reduction, sorption and many others. As a result, this element occurs in the soil in numerous organic combinations (amino acids and protein substances, components of the cell wall of microorganisms, aminosugars, nucleic acids), constituting over 90% of the total nitrogen soil resources [14, 15]. Nitrogen occupies the fourth place (after coal, oxygen and hydrogen) in the mass of soil organic matter. Due to its role for living organisms, it significantly determines the direction and intensity of the transformation of soil organic matter [8].

In the Polish soil system, the process of peat degradation after dehydration is determined by mucking. The progressive oxygenation of the top layers of peat results in an extremely intensive transformation of the soil cover of the drained peatlands areas and the formation of peat-muck soils. The upper muck soils of these soils, in relation to the parent peat, show a different morphology as well as physical, chemical and biological properties [11].

Nitrogen accumulation in organic soils depends, inter alia, on the type of peat and its susceptibility to decomposition processes [8]. Transformation of organic matter in the mucking process refers to the speciation (forms) of nitrogen [2, 15]. Scientific research has shown that the process of mucking transforms peat in the direction of increasing the amount of nitrogen in organic compounds susceptible to mineralization (Becher M. (2013): Organic matter transformation degree in the soils of the upper Liwiec river. Dissertation 125, *University of Natural Science and Humanities in Siedlce*).

Research on the nature of nitrogen transformation in organic soils is important not only in the aspect of the mineralization effects of organic matter and the optimization of their economic use, but also in the aspect of renaturalization of peatlands areas, 9. The aim of the study was to determine the profile variability of the nitrogen fraction in the soils of drained peat and osier bogs.

2. AREA, MATERIALS AND METHODS

Field research was conducted on peatlands located in the upper reaches of the Liwiec River (the Bug river basin). These peatlands constitute the most important wetland area of the Siedlecka High Plain. Peatlands of this area were drained in the 1950s and are intensively used for agriculture (Becher M. (2013): Organic matter transformation degree in the soils of the upper Liwiec river. Dissertation 125, *University of Natural Science and Humanities in Siedlce*).

Two soils from the most important peat species of the studied area were selected for the study, characterized by a low level of groundwater (Photos 1 and 2):

– Profile I - Hemi-murshic soil prepared from peat moss.
– Profile II - Sapri-murshic soil prepared from oles peat.

Soil samples taken from the levels of organic matter accumulation were dried at 40 °C; the representative part was triturated in a mortar (up to the particle size ø <0.25). The following analyzes were made (in three replications) (Becher M. (2013): Organic matter transformation degree in the soils of the upper Liwiec river. Dissertation 125, *University of Natural Science and Humanities in Siedlce*): ash content, by weight after mineralization at 600 °C;

- pH, in 1 M KCl, with a ratio of dry soil to solution = 1/5 (v/v);
- Total carbon (TC) and total nitrogen content (TN) determined on a self-analysis analyzer with a TCD detector (Perkin Elmer). Acetanilide was used as the reference material. The weight ratio TC/TN was calculated.

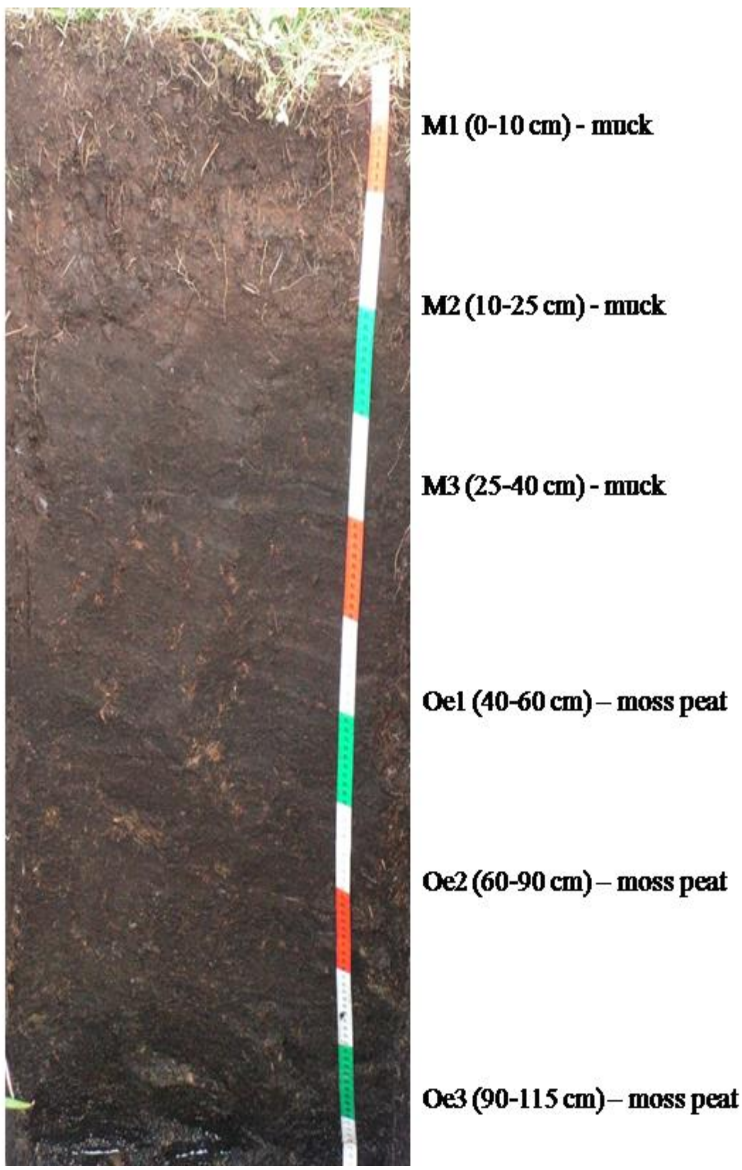

Photo 1. Hemi-murshic soil profile (profile I) (Photo by M. Becher).

Photo 2. Sapri-murshic soil profile (profile II) (Photo by M. Becher).

A sequential fractionation method based on extraction with an inert reagent and two-step acid hydrolysis was used for the analysis of nitrogen speciation (Becher M. (2013): Organic matter transformation degree in the soils of the upper Liwiec river. Dissertation 125, *University of Natural Science and Humanities in Siedlce*). The following extraction solutions were used (2 g soil sample, m/v = 1/25):

- 0.5 M K_2SO_4, for the separation of nitrogen in mineral and organic compounds readily soluble. Extraction carried out at room temperature for 24 hours;
- 0.25 M H_2SO_4, for the separation of organically-active compounds called "readily hydrolysable". Hydrolysis was carried out for 4 hours at reflux and under a water reciprocal radiator;

- 3.0 M H_2SO_4, for the separation of organic compounds which are operationally called "sparingly hydrolysable" (in j.);
- H_2SO_4 (96%) for extracting mineralization of the residue.

The extraction solutions were decanted and filtered through a cellulose filter into a flask (v = 100 ml). After each extraction step, soil samples were rinsed with deionized water (3 times, m/v = 1/5) and the main part of the extract was replenished. The soil particles remaining on the filter were rinsed with deionized water into the extraction flasks and evaporated to dryness. The nitrogen content in the solutions was determined by the Kjeldahl' method. The applied sequential fractionation method allows to distinguish nitrogen in the presented forms (operational fractions) in the tables and figures (Table 1 and Figure 1).

The statistical calculations were made using the statistical program STATISTICA 12 PL (StatSoft, Tulsa, USA). The relationships between the studied features are expressed in the form of a straight-line correlation coefficient (r). For selected relationships, the linear regression equation was determined.

Table 1. Nitrogen forms and the method of their receipt.

Form of nitrogen (operational)	**The method of receipt**
$N\text{-}NH_4$–ammonium nitrogen form	distillation from the extract 0,5 M K_2SO_4, after alkalinisation with MgO
$N\text{-}NO_x$–nitrate forms (III and V) of nitrogen	distillation from the extract of 0.5 M K_2SO_4, after distillation of the $N\text{-}NH_4$ form and reduction with the Devarda mixture
N_{MIN} – nitrogen in mineral compounds	$N_{MIN} = N\text{-}NH_4 + N\text{-}NO_x$
N_{ORG}–nitrogen in organic compounds	$N_{ORG} = TN – N_{MIN}$
N_{K2SO4}	determined in the extract of 0.5 M K_2SO_4, after mineralization of the solution
N_{DON} – soluble organic nitrogen	$N_{DON} = N_{K2SO4} – N_{MIN}$
N_{RHON} – easily hydrolyzing organic nitrogen	determined in a 0.25 M H_2SO_4 hydrolyzate after mineralization of the solution
N_{DHON} – hard to hydrolyze organic nitrogen	determined in a 3 M H_2SO_4 hydrolyzate after mineralization of the solution
N_{HON} – hydrolyzing organic nitrogen	$N_{HON} = N_{RHON} + N_{DHON}$
N_{NHON} – non-hydrolyzing organic nitrogen	nitrogen determined after post-extraction mineralization of the residue in concentrated sulfuric acid (VI)

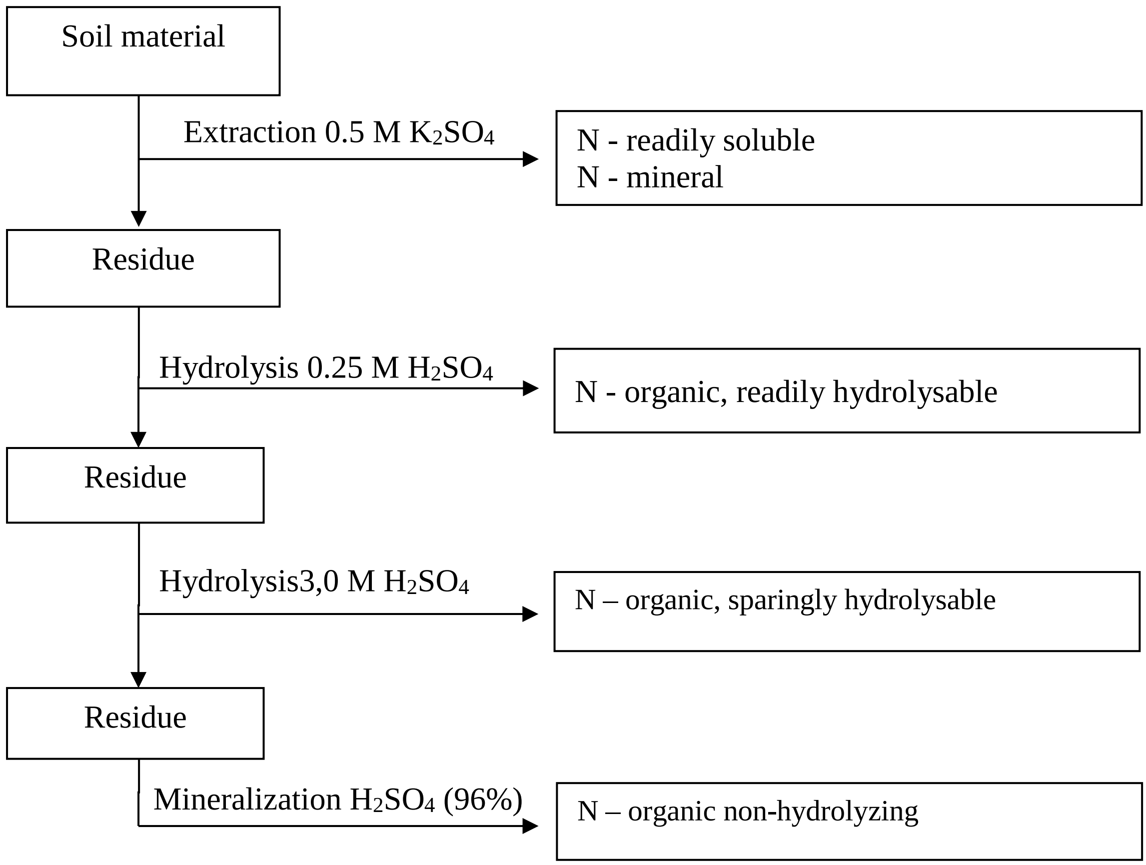

Figure 1. Diagram of sequential coal and nitrogen fractionation.

3. RESULTS AND DISCUSSION

Selected for detailed laboratory tests, Muck soils represent mucking process on various peat species (moss and oless). In both soils, the ground water level was below the peat deposit. A characteristic feature of the structure of the studied soils was the very clear formation of muck (morphing - M1 and substitute M2) levels with a permanent crumb structure and transitional level (M3) built of cracked organic mass and intermediate features between upper muck levels and deeper overhanging peat. Peat forming parental deposits of the studied soils showed morphological features characteristic of moss peat (profile I) and oless peat (profile II) [11, 16]. The distinct morphological difference of muckishlevels in relation to peat, has been confirmed by the results of laboratory tests.

An assessment of the degree of mineral content in organic soils is the ash content obtained after burning the material at high temperature [11]. Muck level of the studied soils, adequate to the advancement of mucking, showed a tendency to increase the ashiness. In contrast, peat levels showed clear associations with individual peat species (moss < oless) and the degree of degradation (*hemic* < *sapric*).

Measurement of the pH value of soil suspensions in 1 M KCl solution showed little variation of this parameter, both between the studied soils and in soil profiles. Studies have shown that muck levels relative to peat are characterized by a lower content of carbon (as a consequence of organic matter) and lower values of the TC/TN ratio. In contrast, the nitrogen content was characterized by low profile variability (Table 2 and 3).

One of the most characteristic phenomena in the mowing process is the decrease in the carbon content due to mineralization of organic compounds in the oxygenated top part of the soil profile. Then, the migration of carbon to the atmosphere in oxidized (CO_2) forms [3, 4, 10]. The nitrogen content stays at a level close to the "initial" (i.e. before the abolition of the protective action of water), or it may occur its secondary accumulation (compaction) in the mass of the created muck. As a consequence of the transformation of carbon and nitrogen compounds in the process of mucking, the ratio of carbon to nitrogen is narrowed. The quantitative relation of these elements is an important indicator of the biochemical direction of organic matter - at low values (below 20) it accelerates the rate of mineralization of organic matter.

Table 2. Some properties of studied soil.

Profile	Genetic horison	pH_{KCL}	Ash content	TC	TN	TC/TN
			g kg^{-1}			
I	M1	5,33	290,0	391,7	28,9	13,5
	M2	5,77	277,1	388,7	30,0	12,9
	M3	5,80	171,3	462,6	30,0	15,4
	Oe1	5,87	100,9	510,1	30,1	16,9
	Oe2	5,65	100,1	513,1	29,4	17,5
	Oe3	5,59	105,8	512,1	30,2	17,0
II	M1	5,81	376,2	330,3	26,7	12,4
	M2	5,89	410,9	307,0	26,9	11,4
	M3	6,27	327,1	329,9	24,9	13,2
	Oa1	5,65	223,7	431,2	27,7	15,6
	Oa2	5,75	194,0	445,4	28,0	15,9
SD		-	110,9	76,5	1,74	2,08
CV (%)		-	47,3	18,2	6,12	14,2
mean–*murshic*layers		-	308,8	368,4	27,9	13,2
mean – *histic*layers		-	144,9	482,4	29,1	16,6

Table 3. The amount and proportion of mineral and organic nitrogen forms.

Profile	Genetic horison	N_{MIN}		N_{ORG}	
		g kg^{-1}	%TN	g kg^{-1}	%TN
I	M1	0,323	1,12	28,6	98,9
	M2	0,298	0,99	29,7	99,0
	M3	0,286	0,95	29,7	99,0
	Oe1	0,249	0,83	29,9	99,2
	Oe2	0,193	0,66	29,2	99,3
	Oe3	0,181	0,60	30,0	99,4
II	M1	0,341	1,28	26,3	98,7
	M2	0,283	1,05	26,6	98,9
	M3	0,200	0,80	24,7	99,2
	Oa1	0,150	0,54	27,6	99,5
	Oa2	0,185	0,66	27,8	99,3
SD		0,065	0,236	1,74	0,236
CV (%)		26,7	27,4	6,17	0,238
mean – *murshic*layer		0,288	1,03	27,6	99,0
mean – *histic*layer		0,192	0,657	28,9	99,3

In the studied soils, the TC/TN value is reduced according to the degree of organic matter processing - from the highest values in peat levels to significantly lower levels in muck levels. The values of this parameter in the muck soil levels of the studied soils are similar to those observed in humus horizons of intensively used arable soils, characterized by organic matter with a high degree of processing and good conditions for its transformation [1]. The TC/TN values (11.4 - 17.5) obtained in the study, as well as slight acidification (pH_{KCl} 5.33 - 6.27) indicate the eutrophication of the studied soil environment, high biological activity and a significant degree of organic matter processing as a result of mineralization processes and humification (Becher M. (2013): Organic matter transformation degree in the soils of the upper Liwiec river. Dissertation 125, *University of Natural Science and Humanities in Siedlce*, 11).

Tables presented (Table 4 and 5) results of fractionation indicate a high dynamics of various forms of nitrogen and their close relationship to soil properties. The largest share of mineral nitrogen forms was characterized by turf muck levels (M1), which is probably due to the inflow and intensive mineralization of fresh organic matter and other sources such as biological reduction of N_2 and fertilization. It is also known that this form of nitrogen is subject to significant seasonal dynamics [5]. In the studied soils 98.7-99.3% TN was organic nitrogen. Results of fractionation of organic nitrogen forms were presented as their share in the total organic nitrogen content (% TON).

Application in sequential extraction of reagents with increasing "extraction power" (0.5M K_2SO_4<0.25M H_2SO_4<3.0M H_2SO_4), allowed to separate nitrogen resources in various forms (contractual operating fractions): readily soluble (N_{DON}), readily hydrolysable (N_{RHON}), sparingly hydrolysable (N_{DHON}) and non-hydrolyzing (N_{NHON}). The separated fractions of this element probably represent organic compounds with different potential durability in the soil environment (i.e. resistance to degradation processes) [Becher M. (2013): Organic matter transformation degree in the soils of the upper Liwiec river. Dissertation 125, *University of Natural Science and Humanities in Siedlce*]. They are arranged in the following sequence: readily soluble<readily hydrolysable < sparingly hydrolysable <non-hydrolyzing.

Table 4. Separated organic nitrogen fractions of the studied soils

Profile	Genetic horison	N_{DON}	N_{RHON}	N_{DHON}	N_{HON}	N_{NHON}
		g kg^{-1}				
I	M1	0,916	9,46	10,5	20,0	7,74
	M2	0,985	9,71	10,4	20,1	8,68
	M3	0,704	6,87	10,9	17,8	11,2
	Oe1	0,509	5,68	11,1	16,8	12,5
	Oe2	0,392	5,68	11,0	16,7	12,1
	Oe3	0,347	6,14	11,1	17,3	12,4
II	M1	0,802	9,79	9,49	19,3	6,24
	M2	0,996	9,93	9,43	19,4	6,27
	M3	0,754	7,54	8,94	16,9	7,09
	Oa1	0,496	6,93	9,49	16,4	10,6
	Oa2	0,432	7,07	9,64	16,7	10,7
SD		0,242	1,70	0,807	1,44	2,46
CV (%)		36,3	21,9	7,92	8,0	25,6
mean–*murshic*layer		0,859	8,95	9,95	18,9	7,87
mean –*histic*layer		0,435	6,30	10,5	16,8	11,7

Table 5. Separated nitrogen fractions of studied soil

Profile	Genetic horison	N_{DON}	N_{RHON}	N_{DHON}	N_{HON}	N_{NHON}	N_{RHON}/N_{DHON}
		% N N_{ORG}					
I	M1	3,20	33,0	36,7	69,7	27,1	0,900
	M2	3,31	32,6	34,9	67,5	29,2	0,935
	M3	2,37	23,1	36,8	59,9	37,7	0,629
	Oe1	1,71	19,0	37,3	56,3	42,0	0,510
	Oe2	1,34	19,5	37,7	57,2	41,5	0,517
	Oe3	1,16	20,5	37,1	57,5	41,3	0,553
II	M1	3,05	37,2	36,1	73,2	23,7	1,03
	M2	3,74	37,3	35,4	72,7	23,5	1,05
	M3	3,05	30,5	36,1	68,3	28,7	0,890
	Oa1	1,80	25,2	34,4	59,6	38,6	0,731
	Oa2	1,55	25,4	34,6	60,0	38,4	0,733
SD		0,913	6,95	1,12	6,51	7,37	0,202
CV (%)		38,2	25,1	3,11	10,2	21,8	26,2
mean–*murshic*layer		3,12	32,6	36,0	68,6	28,3	0,906
mean –*histic*layer		1,51	21,9	36,2	58,1	40,4	0,609

The share of soluble forms of nitrogen (N_{DON}), in relation to other organic forms, was characterized by the lowest share and high variability. Muck peat levels compared to peat plants showed a ca. 2-times greater share of readily soluble forms. With the depth, a decrease in the proportion of nitrogen in mineral compounds was noted. As reported by the correlation coefficients (Table 6). The share of this nitrogen fraction was significantly related to the processes of organic matter decomposition, including mucking (positive correlation with ash content and negative with TC/TN value). Because the organic compounds separated with a neutral salt solution represent the so-called readily soluble part of soil organic matter (DOM), it is probably the mobility of organic compounds extracted using 0.5M K_2SO_4, additionally profiled the share of these fractions [6, 7]. The DOM can include such organic compounds as amino acids, aminosugars, proteins, carbohydrates, low molecular weight fractions of humic acids and many other labile organic compounds [12].

Among the hydrolyzing forms, the fractional fraction with 0.25M H_2SO_4 characterized by readily hydrolyzing (N_{RHON}) was characterized by a significantly higher variability of the share. In all soils, the highest fraction of this fraction was found in muck turf levels, i.e. to the largest extent fed with "fresh" organic matter from the current plant formation. Along with the depth, the growing tendency of this fraction was recorded. Values of correlation coefficients unambiguously indicate that the presence of elements of this fraction is favored by the oxygen transformation of organic matter in muck layers and peat-forming processes. It was found that significant positive correlations with characteristic variables for the mulching process and the distribution of organic matter with the properties of organic soils.

Table 6. Correlation of the share of nitrogen fraction with soil properties

Parameter	Ash content	TC	TN	TC/TN
N_{MIN}	0,681	-0,641	-0,176	-0,755
N_{DON}	0,898	-0,893	-0,450	-0,961
N_{RHON}	0,982	-0,966	-0,623	-0,981
N_{DHON}	-0,485	0,484	0,380	0,439
N_{NHON}	-0,963	0,948	0,586	0,977
N_{RHON} / N_{DHON}	**0,981**	**-0,966**	**-0,627**	**-0,977**

Critical values (n = 11): 0,602 (at α = 0,05); 0,735 (at α = 0,01)

The fraction of the hardly hydrolyzing fraction was characterized by lower variability and poor dependence on soil properties. The amount of elements remaining in the soil samples after extraction with a solution of neutral salt and two-stage acid hydrolysis was determined as a non-hydrolyzing form (N_{NHON}). Conversely to the efficiency of extraction, the higher proportion of this form of elements was characterized by peat levels than muck.

The obtained results for the genetic levels of the studied soils confirm the significant dynamics of the variability of the nitrogen form share during the areobic decomposition of peat organic matter [2, 15]. The transformation proceeds towards increasing the proportion of readily soluble and hydrolysable forms, especially readily hydrolysable ones. This is in line with the literature in which the view prevails that the mucking process transforms peat in the direction of increasing the potential availability of nitrogen, i.e. increasing the amount of organic compounds susceptible to mineralization. But as this process continues (with stable dehydration conditions), the proportion of these nitrogen forms is successively reduced, leading to the depletion of nitrogen available to plants [11].

Becher (Becher M. (2013): Organic matter transformation degree in the soils of the upper Liwiec river. Dissertation 125, *University of Natural Science and Humanities in Siedlce*) in the study of organic soils transformed under the influence of dehydration proposed an indicator to assess the degree of organic matter transformation. He defined it as a ratio of the amount of readily hydrolysable to hardly hydrolyzing forms (N_{RHON}/N_{DHON}). Therefore, it expresses the ratio of quantifiable fractions of these elements - variable (readily hydrolysable) to relatively stable (sparingly hydrolysable).

In the studied soils the value of this quotient was from 0.510-1.05. Based on the value of this indicator, the genetic levels studied can be ranked: moss peat<oles peat<muck. The studies showed a significant correlation of this parameter with properties subject to the characteristic variability in dehydrated peats: positive with ash content and negative with TC content and TC/TN ratio. Selected relationships are presented graphically in the form ofregression equations (Figure 2 and 3) which indicate a linear relationship between the value of this index and the properties of the studied soils.

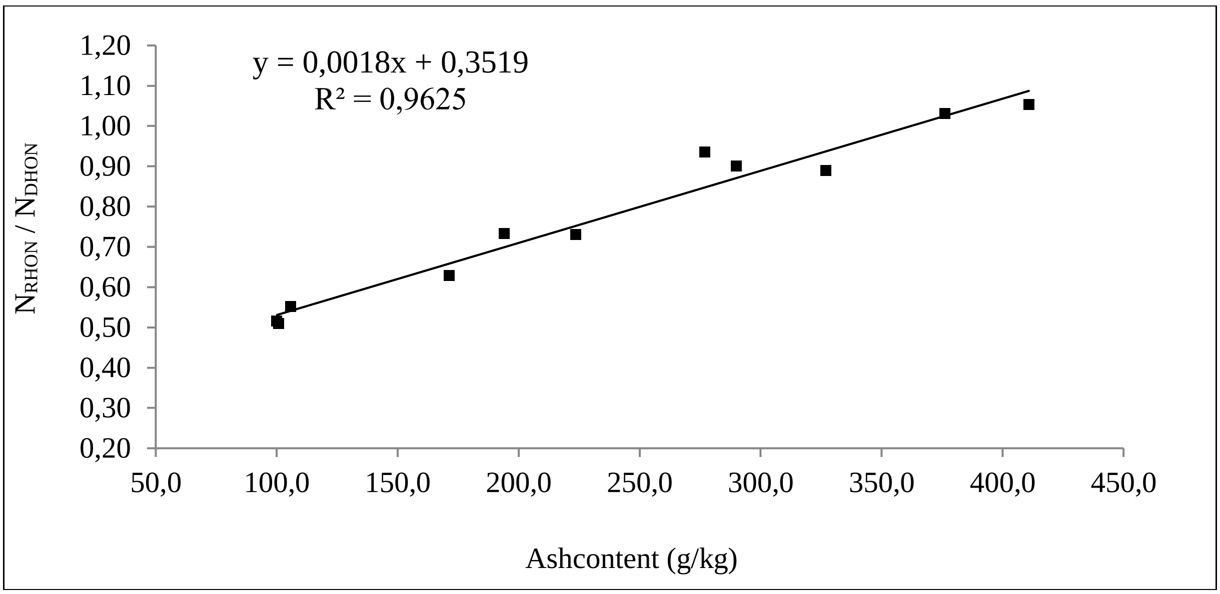

Figure 2. Relationship between ratio of N_{RHON} / N_{DHON} and ash content.

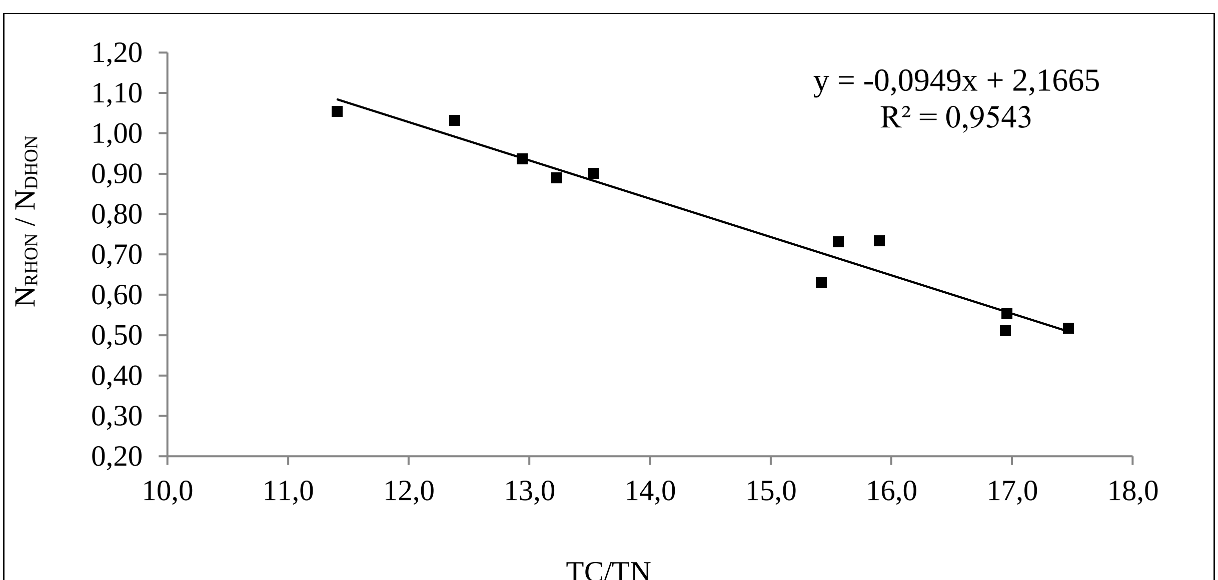

Figure 3. Relationship between ratio of N_{RHON} / N_{DHON} and ratio of TC/TN.

4. CONCLUSIONS

Fractionation of nitrogen compounds consisting of extraction with an inert reagent and two-stage acid hydrolysis provided a lot of information on the transformation of organic matter occurring in dehydrated peats. The process of oxygen transformation of organic matter favors increasing the share of the most mobile readily soluble and readily hydrolysable fraction. It was confirmed that the index describing the state of transformation of organic soils in the decomposition processes may be the values of the ratio of the amount of readily hydrolysable nitrogen forms (fora subject to high dynamics of variation) to the sparingly hydrolysable form (relatively stable form). The value of this indicator showed a significant linear relationship with soil properties (increased along with the advancement of decay processes).

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