The effect of fertilization with brown coal on Haplic Luvisol humic acids

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ABSTRACT

The influence of fertilization with brown coal (used as preparation called Rekulter) on properties of humic acids extracted from haplic luvisol was studied. Characteristics of humic acids were identified by their elemental composition, thermal decomposition, and infrared and UV-VIS spectra. It was found that humic acids formed during decomposition process of Rekulter in soil were characterised by a higher content of carbon (38.6% versus 35.4%), a lower content of nitrogen (1.50% versus 2.73%) and a higher resistance to thermal decomposition, when compared with humic acids extracted from soils without Rekulter application. Due to special properties of Rekulter, additional liming did not affect the properties of humic acids.

Keywords: brown coal; humic acids; liming; soil

Limited usage of farmyard manure leads to progressive deficit of organic matter in soils. Therefore, the requirement for a new source of organic fertilizer grows. A particular growth of interest in fertilizers obtained from brown coal was shown in recent years (Bereśniewicz and Nowosielski 1976, Kalembasa and Tengler 1992, Maciejewska 1993, 1994, Nowosielski 1995). Special properties of brown coal, such as a higher content of total carbon, relatively slow mineralization, lack of pathogenic factor and toxic substances and its porous structure suggest the use of brown coal to enrich soils in humus is particularly attractive (Bereśniewicz and Nowosielski 1976). Brown coal improves physico-chemical properties of soils as well as it influences the properties of soil humic substances. (Gonet et al. 1998). Group composition of humus as well as properties of humic substances are the basic parameters of a given type of soil. There are parameters for estimation of humic acid properties of different origins, generally elemental composition, but also values of aromatic ratios and value of degree of internal oxidising, spectra properties and parameters of thermal decomposition of humic compounds (Chen et al. 1977, Dziadowiec 1979, Gonet 1989, Gonet and Dębska 1993, Gonet and Wegner 1994, Zaujec et al. 1997).

The aim of the present study was to determine properties of humic acids formed as a result of application of the preparation obtained from brown coal – Rekulter into Haplic Luvisols, as well as the influence of additional liming on properties of humic acids.

MATERIAL AND METHODS

Greenhouse experiments were carried out in Wagner type plastic pots filled with soil material taken from the 0–20 cm layer of typical grey-brown podzolic soil (FAO – WRB: Haplic Luvisols) with granulometric composition of loamy sand (16% of fraction < 0.02 mm). The pots were filled with 6.4 kg of the above-mentioned soil material according to the scheme given bellow:

Treatment

1. Acid soil pH_{KCl} = 4.9. The soil was mixed with liquid form of salts: cadmium (1 mg $Cd.kg^{-1}$ d.m. of soil) as $Cd(NO_3)_2$, lead (50 mg $Pb.kg^{-1}$ d.m. of soil) as $Pb(CH_3COO)_2$ and zinc (100 mg $Zn.kg^{-1}$ d.m. of soil) as $ZnSO_4$
2. Acid soil as given above, mixed with Rekulter in dose of 320 g per pot
3. Soil limed twice in 1986 and 1989 with calcium carbonate according to two exchangeable acidities (3.2 $t.ha^{-1}$), not containing heavy metals
4. Soil as given above, mixed with Rekulter in dose of 320 g per pot
5. Soil limed twice in 1986 and 1989 with lime from a lead foundry according to two exchangeable acidities, containing a lot of heavy metals
6. Soil as given above, mixed with Rekulter in dose of 320 g per pot

The Rekulter preparation contained 85% of brown coal, 10% of lowmoor peat, 4% of brown coal ash. In the pot experiment the following plants were cultivated: maize for green forage, rape and lupins, mineral fertilising according to nutrient needs for each plant was applied.

The soil samples were taken after a two year experimental period.

Humic acids (HAs) were extracted and purified according to standard methods by following procedure:

– decalcification with 0.05 M HCl at 1:10 (w/v) ratio, extraction time 24 h; after centrifugation of solution the residue was washed with distilled water until a neutral reaction was reached
– extraction of humus acids: solid remaining after decalcification was subjected to 0.5 M NaOH at 1:10 (w/v) ratio, with 24 h extraction time, occasionally mixed, then centrifuged humus acids extract; in this solution values of absorbance were determined
– precipitation of humic acids: the received alkaline extract was treated with 2 M HCl giving pH = 2, after 24 h precipitate of humic acids was separated by centrifugation
– purification of humic acids, the received residue of humic acids was treated with a mixture of HCl-HF (950 mL H_2O, 5 mL HCl and 5 mL HF) over 24-h period and subsequently separated in a centrifuge; this action was repeated three times; after purification the residue of humic acids was treated with distilled water until a zero reaction to chlorides was achieved, then freeze-dried

The values of absorbance at wave length: 280 nm (A_{280}), 465 nm (A_{465}) and 665 nm (A_{665}) were determined for alkali extracts obtained during fractionating procedure and for 0.02% humic acids solutions in 0.1 M NaOH in Lambda 20 Perkin-Elmer UV-VIS Analyzer. The following factors were calculated:

$A_{2/4}$ – absorbances ratio at 280 and 465 nm
$A_{2/6}$ – absorbances ratio at 280 and 665 nm
$A_{4/6}$ – absorbances ratio at 465 and 665 nm
as well as for humic acids the values of coefficient $\Delta \log K = \log A_{400} - \log A_{600}$ were calculated.

Elemental composition of humic acids was determined by means of CHN 2400 Perkin-Elmer Analyzer. The oxygen content was calculated from the difference [100% – (%C + %H + %N)] in relation to the ashless sample weight. Based on the elemental composition the values of atomic ratio (H:C, O:C, N:C, O:H) as well as the internal oxidation degree (ω) were calculated (Zdanov 1965):

$\omega = (2O + 3N - H):C$

where: C, H, O, N – in atomic percentage, from elemental analysis

Infrared spectra of humic acids were produced in Spectrum BX Spectrometer (Perkin-Elmer) in the range of 400–4000.cm^{-1} for pellets of 3 mg KH in 800 mg KBr.

Thermal analysis was performed in Derivatograph C, MOM Hungary for 40 mg samples of humic acids preparations mixed with Al_2O_3 (1:9) and heated in air atmosphere at the speed of 3.3°C.min^{-1}. Based on the results of thermal analyses the following indecies were calculated:

– weight loss of samples related to the effects registered on DTA curve: endothermic effect (endo), 1st exothermic (exo1), 2nd exothermic (exo2), DTGendo, DTGexo1, DTGexo2, respectively
– maximum temperatures of the effects registered on DTA curve
– parameter *Z* expressing the ratio of weight loss in low temperature range (endo + exo1) to weight loss in high temperature range (exo2). The value Z is proportional to aliphaticity of humic acids (Gonet 1989)
– the ratio of area under the DTA curve to the area under DTG curve corresponding to exothermic effects (DTAexo1/DTGexo1, DTAexo2/DTGexo2) and values of the ratio ΣDTAexo/ΣDTGexo; it is possible to accept with certain approximation that value of this ratio is proportional to calorific value of the sample

Table 1. The basic properties of analysed soil samples

Sample	Rekulter (g per pot)	pH_{KCl}	TOC ($g.kg^{-1}$)	Zinc ($mg.kg^{-1}$)	Lead ($mg.kg^{-1}$)	Cadmium ($mg.kg^{-1}$)
1	0	4.6	7.8	173.9	70.4	1.03
2	320	6.0	21.4	144.6	65.6	1.14
II. factor (average)		5.30	14.60	159.25	68.00	1.085
3	0	6.4	7.5	23.4	17.8	0.24
4	320	6.7	21.5	23.2	16.5	0.28
II. factor (average)		6.55	14.50	23.30	17.15	0.260
5	0	6.0	7.6	119.9	49.9	1.25
6	320	6.5	21.0	111.3	52.4	1.32
II. factor (average)		6.25	14.45	115.60	51.15	1.285
The average values for I. factor						
0 (1, 3, 5)						
320 (2, 4, 6)		5.67	7.63	105.73	46.03	0.840
		6.40	21.40	93.33	44.83	0.913
LSD						
I. factor		n.s.	0.519	n.s.	n.s.	n.s.
II. factor		n.s.	n.s.	62.15	15.15	0.146

Table 2. Spectral properties of alkali fractions

Sample	A_{280}	A_{465}	A_{665}	$A_{2/4}$	$A_{2/6}$	$A_{4/6}$
1	10.04	1.43	0.230	7.02	43.7	6.22
2	33.54	6.13	1.12	5.47	29.9	5.46
II. factor (average)	27.790	3.780	0.675	6.245	36.80	5.840
3	10.00	1.43	0.232	7.02	43.1	6.15
4	32.24	6.01	1.14	5.37	28.2	5.25
II. factor (average)	21.120	3.720	0.686	6.195	35.65	5.700
5	11.34	1.61	0.260	7.03	43.6	6.21
6	35.57	6.55	1.22	5.43	29.2	5.37
II. factor (average)	23.455	4.080	0.740	6.230	36.40	5.790
The average values for I. factor						
0 (1, 3, 5)	10.460	1.490	0.241	7.023	43.47	6.193
320 (2, 4, 6)	33.783	6.230	1.160	5.423	29.10	5.360
LSD						
I. factor	2.5	0.456	0.090	0.125	1.36	0.175
II. factor	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.

The variance analysis for the two factors experiment by Tukey's test were made (I. factor – dose of Rekulter, II. factor – liming).

RESULTS AND DISCUSSION

The application of Rekulter in the Haplic Luvisols caused an increase of total organic carbon content more than three times.The addition of Rekulter resulted in an increase of soil pH values, which can be comparable with liming (Table 1). The lack of significant differences in pH values is connected with alkali reaction of the Rekulter. The highest contents of zinc and lead were characteristic for soil samples marked 1 and 2, and for cadmium variants 5 and 6.

The consequence of increased TOC content in soil after the application of Rekulter was the increase of absorbance value in UV-VIS range for alkali extracts (Table 2). Alkali extracts are the sum of humic acids (HAs) and fulvic acids (FAs) of fractions.

The values of absorbance ratios $A_{2/6,}$ $A_{2/4}$ and $A_{4/6}$ of extracts from soil samples fertilized with Rekulter were significantly lower than from soil without Rekulter (variants 1, 3 and 5). Exact interpretation is difficult as the analysed extracts were not standardised in respect to carbon concentration.

The elemental composition of humic acids is given in Table 3. Humic acids extracted from the soil with Rekulter fertilization (samples 2, 4 and 6) contained more carbon and less nitrogen in comparison to HAs from soil not treated with this preparation (samples 1, 3 and 5), how-

Table 3. Elemental composition of humic acids

Sample	C	H	N	O	H:C	N:C	O:C	O:H	ω
1	34.85	42.15	2.77	20.22	1.21	0.080	0.58	0.48	0.190
2	38.70	39.43	1.53	20.35	1.02	0.039	0.53	0.52	0.151
II. factor (average)	36.775	40.790	2.150	20.285	1.115	0.060	0.555	0.500	0.170
3	35.83	40.92	2.78	20.47	1.14	0.078	0.57	0.50	0.233
4	38.44	40.78	1.50	19.28	1.06	0.039	0.50	0.47	0.059
II. factor (average)	37.135	40.850	2.140	19.875	1.100	0.058	0.535	0.485	0.146
5	35.58	42.27	2.65	19.50	1.19	0.074	0.55	0.46	0.132
6	38.63	40.13	1.48	19.76	1.04	0.038	0.51	0.49	0.099
II. factor (average)	37.105	41.200	2.065	19.630	1.115	0.056	0.530	0.475	0.116
The average values for I. factor									
0 (1, 3, 5)	35.420	41.780	2.733	20.063	1.180	0.077	0.567	0.480	0.185
320 (2, 4, 6)	38.590	40.113	1.503	19.797	1.040	0.039	0.513	0.493	0.103
LSD									
I. factor	1.562	n.s.	0.138	n.s.	0.138	0.006	0.038	n.s.	n.s.
II. factor	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.

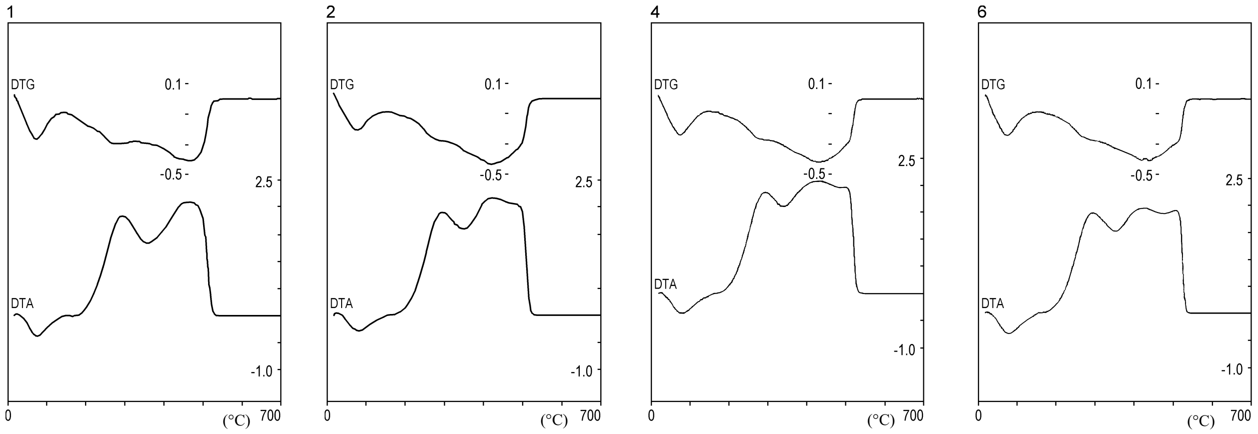

Figure 1. Thermograms of humic acids extracted from soils without Rekulter (1) and after applying of Rekulter (2, 4, 6)

ever contents of oxygen was similar in all the samples. The reflection of elemental composition are the values of atomic ratios (Table 3). Humic acids extracted from the soil samples with Rekulter were characterised by lower values of H:C and N:C ratio in comparison with HAs from non-fertilized soil.

According to Van Krevelen (1950), the values of the H:C ratio of about 0.3 are characteristic for highly condensed aromatic rings, 0.7–1.5 reflect aromatic rings bound with aliphatic chains, whereas the values of 2.0 are characteristic for parafins. Rekulter use resulted in increase of HAs aromaticity (decrease of values H:C ratio). Lime application into soil did not influence the changes in elemental composition of HAs. A particularly low content of nitrogen in HAs can be the indicator of higher needs for nitrogen fertilization of soils improved with Rekulter.

The typical thermograms of the humic acids from soil non-fertilized and fertilized with Rekulter are given on Figure 1. The HAs thermograms contained one endothermic effect and two exothermic effects. On the basis of thermal decomposition studies (Dziadowiec 1979, Gonet 1989) it was stated that humic substances definitely consist of two parts. The part with a lower decomposition temperature reacts in temperature range of up to 350°C (so-called low-temperature) and the part with a higher resistance to decomposition reacts in the high temperature range (so-called high-temperature). In the low-temperature range the endothermic effect appeared in generally (endo, about 100°C) it is related with evaporation of adsorbed water and destruction of functional groups as well as exothermic effect (exo1) which is caused by further oxidising of functional groups and destruction of peripheric aliphatic chain. In the high-temperature

Table 4. Parameters of humic acids thermal decomposition

Sample	Areas under DTA curves (%)			DTAexo1/ DTGexo1	DTAexo2/ DTGexo2	ΣDTAexo/ ΣDTGexo	*Z*
	DTAendo	DTAexo1	DTAexo2				
1	4.8	39.1	56.1	4.37	5.06	4.75	1.07
2	3.6	36.0	60.4	5.07	5.28	5.20	0.821
II. factor (average)	4.20	37.55	58.25	4.720	5.170	4.975	0.946
3	4.6	41.4	54.0	4.33	5.00	4.69	1.12
4	4.4	32.2	63.4	5.20	5.31	5.27	0.743
II. factor (average)	4.50	36.80	58.70	4.765	5.155	4.980	0.931
5	4.7	42.5	52.8	4.46	4.98	4.73	1.15
6	4.3	36.2	59.5	4.99	5.10	5.06	0.855
II. factor (average)	4.50	39.35	56.15	4.725	5.040	4.895	1.003
The average values for I. factor							
0 (1, 3, 5)	4.70	41.00	54.30	4.387	5.013	4.723	1.113
320 (2, 4, 6)	4.10	34.80	61.10	5.087	5.230	5.177	0.806
LSD							
I. factor	n.d.	n.d.	n.d.	0.423	n.s.	0.311	0.161
II. factor	n.d.	n.d.	n.d.	n.s.	n.s.	n.s.	n.s.

Table 5. Parameters of thermal decomposition of humic acids

Sample	Temperature of maximum			Weight losses		
	endo	exo1	exo2	DTGendo	DTGexo1	DTGexo2
1	67.1	291	476	12.5	39.1	48.4
2	73.7	294	428	11.0	34.1	54.9
3	73.0	289	459	10.9	41.8	47.3
4	68.7	291	436	12.9	29.7	57.4
5	70.4	289	459	11.6	41.8	46.6
6	68.7	290	428	12.6	33.5	53.9

range (exo2) decomposition of aromatic part of humic acids proceeds. Humic acids extracted from soil, where Rekulter was applied, have got a higher area under DTA curve corresponding to second exothermic effect compared to HAs extracted from soil without this preparation (Table 4). Distinct broadening of the DTA curve in the second exothermic effect (Figure 1) suggests that in this region exist many effects difficult to isolate.

The highest weight losses in low-temperature range and the lowest in high-temperature range were found for HAs extracted from soil without Rekulter. Application of Rekulter increases weight losses in the second exothermic effect (Table 5). It was reflected in the values of *Z* parameter, which has the highest values for HAs from soil where Rekulter has not been applied (Table 4 – variants 1, 3 and 5). The values of *Z* parameter can be used as a measure of aliphaticity of humic acids. The highest values of this parameter caused the highest participation of aliphatic structure in HAs. The humic acids extracted from soils fertilized with Rekulter were characterised by lower values of *Z* parameter than HAs from soil without Rekulter, therefore their particles display higher participation of aromatic structures.

The values of DTAexo1/DTGexo1, DTAexo2/DTGexo2 and ΣDTAexo/ΣDTGexo ratios are the basic parameter characterising the calorific values of samples. We can conclude, that the calorific values of HAs (for the 1st exotermic reaction and for the total exotermic effect) extracted from soils fertilized with Rekulter are higher than for HAs extracted from samples without Rekulter (No. 1, 3 and 5 – Table 4.).

The absorbency values of humic acids are presented in Table 6. Humic acids extracted from soils fertilized with brown coal preparation, when compared with non-fertilized soils, had higher values of absorbency in UV-VIS region (No. 1, 3 and 5). For soil humic acids, it is possible to interpret absorbency values according to following principles:

A_{280} – express participation of lignin structures
A_{465} – express participation of young humic acids in initial humification stage
A_{665} – express participation of mature humic acids characteristic for well humificated organic matter

Kumada (1975) wrote about the relationship between the values of Δ log *K* coefficient and the degree of the

Table 6. Spectral properties of humic acids

Sample	A_{280}	A_{465}	A_{600}	A_{665}	$A_{2/4}$	$A_{2/6}$	$A_{4/6}$	Δ log *K*
1	4.09	0.898	0.311	0.169	4.55	24.2	5.31	0.714
2	4.64	1.07	0.398	0.198	4.32	23.4	5.43	0.740
II. factor (average)	4.365	0.984	0.354	0.183	4.435	23.800	5.370	0.727
3	3.34	0.717	0.253	0.141	4.65	23.6	5.08	0.706
4	5.44	1.25	0.426	0.240	4.35	22.7	5.21	0.728
II. factor (average)	4.390	0.984	0.340	0.190	4.500	23.150	5.145	0.717
5	5.08	0.848	0.292	0.161	5.99	31.5	5.26	0.724
6	6.88	1.41	0.471	0.262	4.87	26.3	5.40	0.741
II. factor (average)	5.980	1.032	0,382	0.211	5.430	28.900	5.330	0.732
The average values for I. factor								
0 (1, 3, 5)	4.170	0.821	0.285	0.157	5.063	26.433	5.217	0.715
320 (2, 4, 6)	5.653	1.243	0.432	0.233	4.513	24.133	5.347	0.736
LSD								
I. factor	1.100	0.440	0.128	0.066	n.s.	n.s.	0.029	0.011
II. factor	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	0.049	n.s.

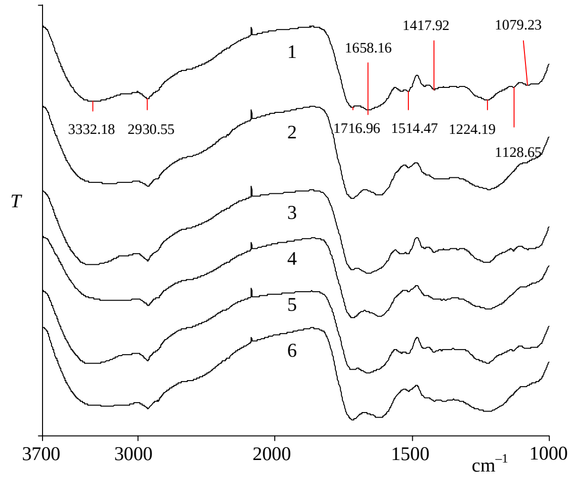

Figure 2. IR-spectra of humic acids extracted from soils without Rekulter (1, 3, 5) and after applying of Rekulter (2, 4, 6)

humification of humic acids. The author, on the basis of Δ log *K* coefficient, divided humic acids into three basic types: type A – humic acids of high degree of humification for which values of Δ log *K* coefficient is up to 0.6, type B – those of corresponding values between 0.6 and 0.8, and type R_p – Δ log *K* values within the range of 0.8–1.1. Analysed HAs were, according to Kumada's classification, the B-type humic acids. Fertilization with Reculter regardless of liming has modified the type of HAs. Application of brown coal preparation into soil resulted in an increase of Δ log *K* values of HAs as compared to those from soil without Rekulter.

The values of $A_{2/4}$ and $A_{2/6}$ ratios for humic acids extracted from soils have not varied significantly. Humic acids extracted from soil where brown coal preparation was used, in comparison to humic acids from soil non-fertilized with Rekulter, were characterised by higher values $A_{4/6}$ and Δ log *K*. It was the consequence of an increase in participation of humic acids in the initial decomposition stage. The application of fresh (but specific) organic matter together with brown coal in soil caused formation of young humic acids. The values of $A_{4/6}$ are inversely proportional to the molecular weight of humic substances (Chen et al. 1977, Kumada 1987, Gonet and Wegner 1994). The analyses of results revealed that Rekulter fertilization was responsible for obtaining of humic acids with the highest participation of smaller molecular weight molecules during extraction procedure.

Infrared spectra of humic acids varied significantly (Figure 2). Fundamental differences between the HAs spectra originating from the fertilized and non-fertilized soils were found in the ranges: 2860–2960.cm^{-1} ($-CH_3$ and $=CH_2$ in aliphatic chains), 1530–1670.cm^{-1} (complex band: C=O of various origin, NH in amids, C=C of aromatic rings) and 1000–1200.cm^{-1} (complex band: $-CH_3$, –OH, polysaccharides) (Stevenson and Goh 1971). The shape of spectral band with maximum at 1720.cm^{-1} (carboxyl functional groups) was characteristic for HAs isolated from soils fertilized with brown coal. The band at the wave range 1530–1670.cm^{-1} with maximum in 1640.cm^{-1} was observed for humic acids separated from soil without any additives. In spectra of humic acids separated from soil fertilized with brown coal at the wave range 1530–1670.cm^{-1} the presence of wide band with maximum shifted to 1620.cm^{-1} was observed. It indicates higher aromaticity of these HAs in comparison to HAs from the control. It corresponds with the analysis of spectra in the 2900.cm^{-1} region. The higher intensity of this band for HAs extracted from non-fertilized soil was recorded. Absorption band at 1640.cm^{-1} resulted from the occurrence of nitrogen in HAs structures. According to elemental composition (Table 3), humic acids from the soil fertilized with brown coal had a lower content of nitrogen. This was confirmed by lower absorption in this IR range.

CONCLUSIONS

The obtained results indicate that the application of brown coal preparation in Haplic Luvisols created humic acids with higher aromaticity and higher resistance to thermal destruction as compared to humic acids extracted from soil without the preparation. The higher content of carboxyl functional groups in humic acids extracted from soil containing Rekulter increases the cation exchange capacity in these soils (Maciejewska 1993), which is particularly important in processes of heavy metal bonding. After the application of Rekulter the nitrogen content in HAs has decreased, while the content of oxygen was not significantly changed. It can be stated that the application of brown coal in soil results in the significant increase of C:N ratio and this indicates the necessity for fertilization of these soils with nitrogen.

The Rekulter preparation has similar effect on soil reaction as soil liming. There were no significant differences between properties of humic acids extracted from limed and non-limed soils. The influence of heavy metal contents in soil on properties of humic acids was not noted.

In general, it can be concluded that the application of brown coal as a means of soil improvement resulted in an increase of organic carbon content in soil and introduced organic matter, especially humic acids, more resistant to decomposition than other organic substances.

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ABSTRAKT

Vliv aplikace hnědého uhlí na obsah huminových kyselin v luvizemi

Byl sledován vliv aplikace hnědého uhlí (v přípravku Rekulter) na vlastnosti huminových kyselin extrahovaných z luvizemě. Vlastnosti huminových kyselin byly stanoveny na základě jejich prvkového složení, tepelného rozkladu, infračerveného spektra a spektra UV-VIS. Ve srovnání s huminovými kyselinami extrahovanými z půdy bez aplikace Rekulteru huminové kyseliny, které vznikají při rozkladném procesu přípravku Rekulter, měly vyšší obsah uhlíku (38,6 % oproti 35,4 %), nižší obsah dusíku (1,50 % oproti 2,73 %) a vyšší odolnost vůči tepelnému rozkladu. Doplňkové vápnění neovlivnilo vlastnosti huminových kyselin v důsledku zvláštních vlastností přípravku Rekulter.

Klíčová slova: hnědé uhlí; huminové kyseliny; vápnění; půda

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