

OPTICAL PROPERTIES OF HUMIC ACIDS IN SELECTED ORGANIC WASTES*

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Abstract. The objective of the study was optical characterisation of humic acids extracted from organic wastes. The study included wastes such as turkey manure, solid sludge from sewage treatment plant Hajdów, substrate after mushrooms growth and materials obtained after the fermentation of residues from a biogasing plant, as well as charcoal derived from biomass biogasing pyrolysis. The tested organic wastes were analysed for the content of organic carbon and for their humification degree. The humification number of investigated wastes was obtained according to the Springer method. Humic acids were extracted from the wastes according to the Schnitzer method. The acids extracted were characterised in terms of their optical properties. A correlation was found between yield of humic acids and organic matter content in organic wastes and their degree of humification. The parameters $q_{4/6}$ for examined organic compounds were characterised to fulvic acids, and the values of $\Delta\log K$ indicated a low or medium humification degree.

Keywords: organic wastes, humic acids, optical properties, organic carbon, humification degree

INTRODUCTION

Agricultural wastes and wastes from the agri-food industry contain all essential components necessary for the growth of microorganisms, such as carbohydrates, proteins, fats, biogenic elements, microelements and vitamins. Leaving the agricultural wastes in their raw state creates a sanitary hazard and environmental

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problems (Baran and Turski 1999). Wastes constitute a vast reservoir of organic matter, including humus substances, which suggests a possibility of their utilisation as an organic fertiliser, improving the physicochemical conditions in soils and in the environment of plant growth.

Among the various fractions of organic matter a special role is played by humus substances which are a polymolecular and polydispersive mixture of polyelectrolytes (Kononova 1966, Orlov 1990, Orlov *et al.* 2005). Humus substances are fairly stable chemically and characterised by specific properties and a high molecular weight (Stevenson 1982). Fulvic acids, both in a free form and as salts, are easily soluble in water and thus highly mobile. Humic acids are a group of specific organic compounds, formed in complex processes of biochemical condensation and polymerisation of products of decomposition of residues of plant and animal origin, as well as of products of microbial metabolism (Kononova 1966, Orlov 1990, Dębska *et al.* 2009). The structure (aromatic links, aliphatic chains, functional groups) and properties of humic acids cause that they participate in all processes taking place in soil (Dębska *et al.* 2009).

Physicochemical methods permit the study of properties resulting from the complex structure of humus substances. Optical methods, e.g. analysis of absorbance in the ultraviolet (UV) and visible light (VIS) bands are most frequently applied for the characterisation of humus substances (Kononova 1966, Orlov *et al.* 1969, Stevenson 1982, Kalbitz *et al.* 1999, Gołębiowska 2004).

The objective of this study was to determine, in organic wastes of various origins, the content of organic carbon and compounds of the type of humic acids and their optical properties, as well as the degree of humification of the wastes under study.

MATERIAL AND METHOD

The experimental material comprised samples of organic wastes such as turkey manure from a poultry farm (sample No. 1), solid post-fermentation residue from a biogasing plant (sample No. 2), charcoal derived from biomass biogasing pyrolysis (Hajnówka, sample No. 3), solid sludge from the sewage treatment plant Hajdów in Lublin (sample No. 4) and substrate after mushroom growing (sample No. 5).

The content of organic carbon in the materials studied was determined using the TOC MULTI N/C 2000, HT 1300 (Analytik Jena) carbon and nitrogen analyser. Calculations were performed with the use of the software supplied with the apparatus.

The extraction and characterisation of organic compounds from the wastes were performed with methods commonly applied for the extraction and optical characteristics of humic acids from soil. Humic acids were extracted from the wastes under study according to the method of Schnitzer (Methodological Guide 1990).

The obtained preparations of humic acids were used to prepare starting suspensions with uniform concentrations. Optical properties of 0.04% clear solutions of sodium humate in 0.1 M NaOH were analysed on a UV/VIS spectrophotometer (Jasco V-500). Values of the optical parameter q were measured at wavelengths of 280 nm, 465 nm and 665 nm, and values of the parameter $\Delta \log K$ as absorbance difference at wavelengths λ of 400 and 600 nm, respectively.

Elemental composition (C, H, N) of the extracted humic acids was assayed on a Perkin Elmer CHN 2400 analyser. The content of oxygen (and strictly speaking the sum of O+S) was calculated from the difference obtained after summing the contents of C, H, N and the value of 100%. On the basis of the elemental composition, the values of the degree of internal oxidation (ω) according to Zhdanov (1965) were calculated from the equation $\omega = [(2O + 3N) - H]/C$.

The humification degree of the samples was determined with the Springer method (Schlichting *et al.* 1986). The method consists in the measurement of absorbance, A , in visible light (VIS) at wavelength of $\lambda = 530$ nm in an extract obtained after boiling a sample in 0.5% solution of NaOH that was prepared on the base of 0.5% $C_2Na_2O_4$ (sodium oxalate). The absorbance of the solution was measured on the UV/VIS spectrophotometer JASCO V-500.

The humification degree, determined with the Springer method, was expressed as the so-called humification number, H_z . The number defines the percentage of organic substance which migrated to the extract. The humification number H_z was calculated from the relation: $H_z = C_{KH}/M_0 \cdot 100$, where C_{KH} is the concentration of humic acids ($g\ 100\ ml^{-1}$), and M_0 is the amount of organic matter converted to dry matter (g).

RESULTS AND DISCUSSION

Table 1 presents the organic carbon content and the humification number of the wastes under study, and results of alkaline extraction yield, which corresponds to the content of humic acids in the wastes.

As can be seen in Table 1, the wastes were characterised by a varied content of organic carbon, ranging from 250 to 470 $mg\ g^{-1}$ d.m. The highest levels of carbon were in samples No. 2 (solid post-fermentation residue from biogasing plant) and No. 1 (turkey manure), and the lowest in sample No. 4 (solid sludge from sewage treatment plant). Similar results were obtained e.g. by Czekala (2002a) for a number of sewage sludges from the region of Wielkopolska, and by Drozd and Licznar (2002) for composted municipal wastes.

The amount of humic acids migrating to the solution depended on the kind of organic waste. The smallest amount (1-2 g) of the compounds was extracted from the solid sludge from a sewage treatment plant (sample No. 4), mushroom sub-

strate (sample No. 5) and charcoal from biomass biogasing through pyrolysis (sample No. 3). The highest content of humic acids was characteristic of the turkey manure (sample No. 1) and solid post-fermentation residue from biogasing plant (sample No. 2). Generally, alkaline extraction of the wastes yielded small amounts of humic acids, from 0.8 to 9 g from 100 g of dry sample of the wastes. Studies on sewage sludge revealed that it is a slowly-degradable organic material, a considerable part of organic compounds contained in sludge does not undergo extraction, and among the compounds isolated dominant were labile, low-molecular compounds with the properties of fulvic acids (Czekala 2002b). Also a study on the effect of moisture of composted municipal wastes on organic matter transformation demonstrated that although already after a month of composting the organic matter displayed a considerable degree of humification, among its products dominant were low-molecular compounds, represented by fulvic acids and the fulvic fraction 2).

Table 1. Selected parameters characterizing the organic wastes

Parameters	Sample				
	1	2	3	4	5
W (g 100g ⁻¹ d.m.)	4.59	8.90	1.91	0.84	1.66
C _{org} (mg g ⁻¹ d.m.)	413.2	470.5	354.9	252.3	306.7
H _z	4	2	4	3	6

Samples explanations in "Material and method", W – content of humic acids (extraction yield), C_{org} – organic carbon, H_z – humification number.

The values of the humification number, H_z, for the wastes under study fall within the range from 2 to 6. This indicates a rather low degree of humification of

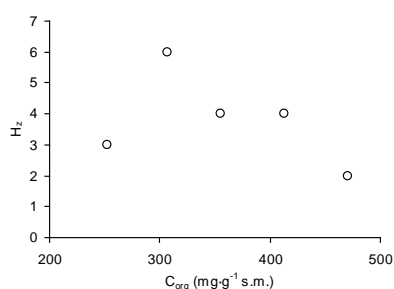


Fig. 1. Relation between organic carbon content (C_{org}) and humification number (Hz) of the investigated organic wastes

the wastes. The highest value of H_z was found for the mushroom substrate (sample No. 5), and the lowest the post-fermentation residue from biogasing plant (sample No. 2) and turkey manure (sample No. 1). For comparison, the humification number values for muck formations fall within the range from 11 to 23 (Matyka-Sarzyńska and Sokółowska 2004), and those of alkaline extracts of samples of low and high peat are 31 and 51, respectively (Bejger 2007). As can be seen from Figure 1, the correlation between the humification number and the content of organic carbon in the wastes is weak – the value of the

number and the content of organic carbon in the wastes is weak – the value of the

coefficient of determination R^2 is only 0.187. This suggests that in the total pool of organic carbon in the wastes the level of carbon compounds accepted by Springer as indicators of the degree of degradation of organic material is low. We could then pose the hypothesis that the wastes under study are weakly humified. However, as follows from a study on peats conducted by Berger (2007), the humification number (H_z) cannot be identified with the degree of humification of peat nor of organic matter, but is rather related with the size of molecules of humic acids.

Figure 2 presents the relation between the content of humic acids in the wastes (extraction efficiency) and the content of organic carbon and humification number of the organic wastes under study.

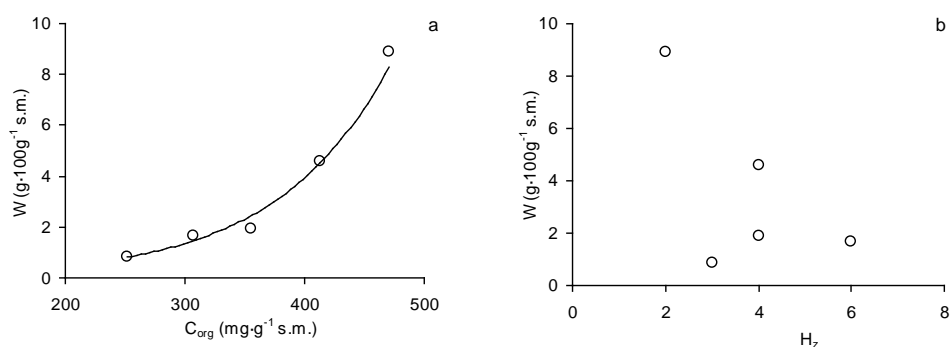


Fig. 2. Efficiency of extraction process of humic acids (W) vs. organic carbon content (C_{org}) and humification number (H_z) of the investigated organic wastes

The efficiency of extraction clearly depends on the content of organic carbon in the wastes (Fig. 2a). Experimental data are well described by an exponential curve, and the value of the coefficient of determination R^2 is 0.976. A similar exponential relation appears in the liberation of soluble organic matter from peats during the process of alkalisation (Matyka-Sarzyńska and Sokołowska 2005). Whereas, no relation was found between extraction efficiency and values of humification number of the wastes ($R^2 = 0.362$). According to Springer (Schlichting *et al.* 1986), humification number H_z is an indicator of the degree of decomposition and chemical transformations in peat. In our opinion, H_z rather is not an indicator of the degree of decomposition and chemical transformations in the wastes under study, which is indicated by the lack of correlation between extraction efficiency and values of humification number of the wastes, and between humification number and organic carbon content (Fig. 1 and Fig. 2b). This observation is in conformance with the conclusion resulting from the study by Berger (2007) on peats that H_z cannot be identified with degree of decomposition (humification).

A frequent method of description and comparison of humic acids from various wastes is the presentation of their elemental composition. Changes in elemental composition are accompanied by changes of atomic ratios (H/C, C/N, O/C, O/H). The values of those ratios permit the approximation of the structure of humic acid molecules thanks to the estimation of the degree of condensation of aromatic rings (H/C ratio) and their degree of maturity (C/N, O/C, O/H ratios, ω).

Table 2 presents the elemental composition, atomic ratios and degrees of internal oxidation (ω) of humic acids extracted from the wastes under study.

In the elemental composition of the extracted humic acids the dominant role is played by carbon (Tab. 2), accounting for 41-52% by weight. Such a content of carbon is comparable to its levels in fulvic acids of soils (Schnitzer and Khan 1978), lake sediments (Ishiwatarii 1985), peats (Bambalow *et al.* 2000, Berger 2007). The content of nitrogen is notably varied, and it is the factor differentiating the isolated humic acids. The highest content of nitrogen is found in acids extracted from turkey manure (sample No. 1), and in the other samples the content of nitrogen is comparable to that of fulvic acids from lake sediments (Ishiwatarii 1985), soil fertilised with vermicompost (Flis-Bujak and Żukowska 2001) or lignite (Kwiatkowska-Malina 2009) or from composts of municipal wastes (Drozd and Licznar 2002). For humic acids, the extent of changes in their elemental composition and their direction depend, among other things, on the composition of organic matter undergoing decomposition. Dziamski (2003) observed the greatest increase in carbon content in humic acids formed after soil fertilisation with vermicompost or FYM, and a considerably weaker increase in the case of straw. Smoliński *et al.* (2005) noted the occurrence of “young humic acids” in a soil with plant residue (fragmented grass).

Table 2. Elemental composition of the humic acids, values of the atomic ratio (C/N, H/C, O/C) and internal oxidation degree (ω)

Sample	C	H	N	O	C/N	H/C	O/C	ω
1	41.27**	4.33**	19.99**	34.40**	2.41	1.26	0.63	1.24
	30.32*	38.15*	12.58*	18.95*				
2	52.50**	5.80**	4.01**	37.69**	15.27	1.33	0.54	0.10
	34.13*	45.24*	2.23*	18.34*				
3	48.74**	4.41**	5.09**	41.76**	11.17	1.09	0.63	0.47
	35.47*	38.52*	3.18*	22.79*				
4	45.33**	5.49**	4.56**	44.62**	11.37	1.45	0.74	0.28
	30.51*	44.35*	2.68*	22.48*				
5	50.55**	3.98**	5.12**	40.35**	11.52	0.94	0.60	0.51
	38.02*	35.92*	3.30*	22.76*				

Samples explanations in “Material and method”, ** weight percentage, * atomic percentage.

On the basis of H/C ratio one can make an approximate estimation of the degree of aromatisation of humus substances. The value of H/C atomic ratio is inversely proportional to the aromaticity of humus compounds (Kononova 1966, Gonet and Zawalska 1993). According to van Krevelen (1950), H/C values of ca. 0.3 are characteristic of compounds with highly condensed aromatic rings, values of about 0.7 characterise monocyclic aromatic hydrocarbons, values in the range of 0.7-1.5 correspond to aromatic systems related with aliphatic chains composed of up to 10 carbon atoms, values from 1.5 to 1.7 correspond to alicyclic hydrocarbons, and values of about 2 are characteristic for paraffins. Values of H/C atomic ratio characterising humic acids extracted from the wastes under study fell within the range from 0.94 to 1.45 (Tab. 2). The variation in the values of H/C ratio permits arraying the humic acids extracted from the wastes under study in a series with regard to the share of aliphatic structures and the degree of condensation of the nucleus. Taking the value of H/C ratio as the basis, we can arrange the acids extracted from the wastes in accordance with increasing share of aliphatic structures (acids from sample No. 5 < from sample No. 3 < from sample No. 1 < from sample No. 2 < from sample No. 4). As follows from the series, the highest share of aliphatic structures is characteristic of humic acids extracted from the solid sludge from sewage treatment plant (sample No. 4), and the lowest – from the mushroom substrate (sample No. 5).

The O/C ratio and the degree of internal oxidation (ω) are good indicators of quality of humus compounds. The value of ω of the humic acids extracted from the wastes under study was within the range from 0.1 to 1.24 (Tab. 2), which indicates a variation of the degree of internal oxidation. The lowest value of ω was found for the humic acids extracted from the solid waste from biogasing plant (sample No. 2), and the highest for humic acids from turkey manure (sample No. 1). Studies by Kwiatkowska-Malina (2009) indicate that the introduction of lignite (as a source of organic matter) into soil caused structural changes of humic acids extracted from the soil. The acids were characterised by a higher degree of aromaticity and greater susceptibility to oxidation as compared to the control soil. The values of ω of humic acids extracted from soils fertilised with the lignite preparation were 0.058-0.212.

The C/N ratio was more diversified. The values of that ratio fell within the range from about 2 to 15 (Tab. 2). The highest C/N values were found in humic acids extracted from the post-fermentation residue for biogasing plant (sample No. 2), and the lowest – in those from turkey manure (sample No. 1). The lowest value of C/N ratio in humic acids from turkey manure indicates that they were the “least mature” compared to the other humic acids. Humic acids extracted from residue after pyrolysis of charcoal from biomass biogasing (sample No. 3), solid sludge from sewage treatment plant (sample No. 4) and from mushroom substrate (sample No. 5) had similar values of C/N ratio. Studies by Drozd and Licznar

(2002) showed that the C/N value of humic acids in composts of municipal wastes was within the range from about 9.6 to 12.4 and varied in relation to moisture and to the time of composting.

Figure 3 presents absorption spectra of humic acids extracted from the wastes under study. Within the analysed range of λ , the spectra had a monotonic character and are similar to each other. The exception to that general tendency is the spectrum of humic acids extracted from turkey manure (curve 1). Based on the spectra obtained we can state that the humic acids extracted from the wastes under study are not homogeneous. The maximum absorbance (at $\lambda = 280$ nm) varied within the range from ca. 0.3 to ca. 1.6. The highest absorbance (Fig. 3) was characteristic of humic acids extracted from sample No. 3 (charcoal derived from biomass biogasing through pyrolysis), and the lowest of those from sample No. 4 (solid sludge from sewage treatment plant). As can be seen in Figure 3, the spectra for sample No. 1 and 4 (if we skip the initial section of the spectrum for sample No. 1) and of samples No. 2 and 5 are very similar and show similar values of absorbance, which can suggest similar character of the humic acids. Analysis of data in Table 2 permits the conclusion that humic acids of samples No. 1 and 4 have similar carbon content, but the humic acid of sample No. 1 has a notably higher content of nitrogen and a greater value of the degree of internal oxidation compared to the humic acid of sample No. 4. Humic acids of samples No. 2 and 5 have similar content of nitrogen, differ slightly in their carbon content, and significantly in the degree of internal oxidation and in H/C ratio values.

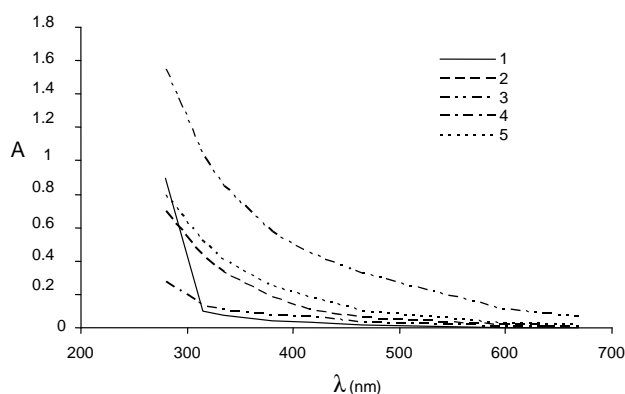


Fig. 3. UV-VIS absorption spectra of humic acids extracted from the investigated wastes.

Explanations:

A – absorbance; λ – wavelength; Samples explanations No. 1-5 in Table 1

The absorption spectra of humic acids extracted from the wastes under study were used to calculate the values of parameters $q_{2/4}$ and $q_{4/6}$ ($q_{280/465}$ and $q_{465/665}$) and the value of parameter $\Delta\log K = \log K_{400\text{nm}} - \log K_{600\text{nm}}$. The results obtained are presented in Table 3.

Parameters q ($q_{280/465}$ and $q_{465/665}$) characterising the humic acids extracted from the organic wastes under study displayed considerable variation (Tab. 3).

Parameter $q_{465/665}$ ($q_{4/6}$) had the highest value for humic acids from turkey manure (sample No. 1), and for the other acids its values varied within the range from about 5 to 9. The values are comparable to those for fulvic acids, for which the value of $q_{4/6}$ is 6.0 – 18.5 (Kononova 1966). Only in the case of humic acids from sample No. 3 (residue after charcoal pyrolysis) parameter $q_{4/6}$ assumes values in the range characteristic of humic acids (< 6) (Kononova 1966). As mentioned above, the spectra for samples No. 1 and 4 (if we skip the initial section of the spectrum for sample No. 1) and for samples No. 2 and 5 are very similar and show similar values of absorbance, which can suggest similar properties of the humic acids. Humic acids of samples No. 1 and 4 have higher values of parameters $q_{280/465}$ and $q_{465/665}$ compared to the humic acid of sample No. 4. Whereas, humic acids of samples No. 2 and 5 have similar values of parameters $q_{280/465}$ and $q_{465/665}$.

Table 3. Optical characteristics of humic acids obtained from the investigated organic wastes

Parameter \ Sample	1	2	3	4	5
$q_{2/4}$	58.7	16.1	4.8	7.2	8.3
$q_{4/6}$	13.0	7.0	4.9	8.6	7.5
$\Delta\log K$	1.05	1.1	0.7	0.9	0.7

Explanations in "Material and Method".

In soils, the value of parameter $q_{4/6}$ depends on the soil type and for humic acids it forms the range from 3 to 5.8 (Schnitzer and Khan 1978, Stevenson 1982). It is accepted that the value of absorbance at wavelength of 400-464 nm defines the content of substances in the initial stage of humification, and at λ 600-664 nm, in the final stage of humification. That relation, according to Schnitzer and Khan (1978), does not depend on the concentration of the extracted humus substance, but on its character. With increase in the degree of condensation of the aromatic nucleus of molecules and with increase of their molecular weight the optical density increases, but there is a drop in the value of parameter $q_{4/6}$ (Gonet 1989).

Parameter $q_{280/465}$ ($q_{2/4}$) of humic acids of the wastes under study varied within a broad range of values (Tab. 3) and it was the highest for humic acids from turkey manure (sample No. 1), which suggests a content of compounds not easily susceptible to humification. Parameter $q_{2/4}$ describes the ratio of the content of compounds resistant to humification, of the type of lignins, to strongly humified substances, and an increase in its value indicates that it is related with enrichment of alkaline extracts in compounds not easily undergoing humification, of the type of lignins (Greenland *et al.* 1978, Sapek and Sapek 1986). Parameter $q_{2/4}$ characterises the stage of the process of humification, and its value decreases with the progress of the process (Gonet and Dębska 1993). Comparison of the values of the humification number (H_z) of the wastes, determined earlier (Tab. 1) with the values of parameter $q_{2/4}$ (Tab. 3) suggests that the humification number cannot be identified with the degree of decomposition of organic material.

The values of parameter $\Delta\log K$ varied only slightly (from 0.7 to 1.2). The lowest value was obtained for humic acids extracted from sample No. 5 (residue after charcoal pyrolysis) and sample No. 5 (mushroom substrate). Kumada (1975) relates the stage of the process of humification with the value of the parameter $\Delta\log K$, which allows humic acids to be arranged in a series in accordance with their degree of transformation (humification) – the higher the degree of humification the lower the value of $\Delta\log K$. And thus $\Delta\log K$ values up to 0.6 mean a high, from 0.6 to 0.8 – medium, and from 0.8 to 1.1 – a low degree of humification. Analysing the humic acid extracted from the wastes under study in accordance with the division proposed by Kumada (1975) it can be assumed that compounds from samples No. 3 and 5 are humified in medium degree, and those from the other samples – in low degree.

As follows from the discussion, the wastes under study contain small amounts of humic acids. The acids are characterised by low and medium degree of humification, predominance of aliphatic structures, and values of optical parameters similar to those of fulvic acids.

CONCLUSIONS

1. Spectra of humic acids from the wastes under study are monotonic in character and similar to each other. The exception is the spectrum for the humic acids isolated from turkey manure.
2. Values of $q_{4/6}$ characterising the humic acids studied are comparable to those for fulvic acids. Only in the case of humic acids isolated from the residue after charcoal pyrolysis the values of parameter $q_{4/6}$ fall within the range of values characteristic of humic acids.

3. Values of parameter $\Delta\log K$ suggest that humic acids extracted from the wastes under study are humified to a low and medium degree.

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CHARAKTERYSTYKA OPTYCZNA KWASÓW HUMINOWYCH W WYBRANYCH ODPADACH ORGANICZNYCH*

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Streszczenie. Do badań wykorzystano naturalną pozostałość pofermentacyjną stałą z biogazowni, węgiel drzewny pochodzący ze zbiogazowania biomasy w wyniku pirolizy, pomiot indy czy, osad stały z oczyszczalni ścieków oraz podłoże po pieczarkach. W odpadach organicznych określono zawartość węgla organicznego oraz kwasów huminowych. Z odpadów organicznych wyekstrahowano kwasy huminowe zmodyfikowaną metodą Schnitzera. Stopień humifikacji odpa-

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dów oznaczono metodą Springera. Wyekstrahowane kwasy scharakteryzowano pod kątem ich właściwości optycznych. Określono także zależność pomiędzy wydajnością ekstrakcji tych związków od zawartości węgla organicznego i stopnia humifikacji odpadów. Stwierdzono, że większość z badanych kwasów huminowych miała cechy zbliżone do cech kwasów fulwowych. Badane odpady organiczne wykazywały mały lub średni stopień humifikacji.

Słowa kluczowe: odpady organiczne, kwasy huminowe, parametry optyczne, węgiel organiczny, stopień humifikacji