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## ORIGINAL PAPER

## EFFECT OF THERMAL CONVERSION OF MUNICIPAL SEWAGE SLUDGE ON THE CONTENT OF Cu, Cd, Pb AND Zn AND PHYTOTOXICITY OF BIOCHARS\*

Krzysztof Gondek, Monika Mierzwa-Hersztek

Department of Agricultural and Environmental Chemistry  
University of Agriculture in Krakow

## ABSTRACT

Degradation of various compounds with the simultaneous formation of new or activation of initially unavailable chemically bound substances or elements can occur very often during the thermal conversion of organic materials. Thus, the aim of this research was to determine the effect of thermal conversion of municipal sewage sludge on the content of total and water-soluble forms of Cu, Cd, Pb, Zn, and on the phytotoxicity of the solid products generated in the process. The research material consisted of stabilized sewage sludge from municipal sewage treatment plants in Krakow (SS1), Krzeszowice (SS2) and Słomniki (SS3), Malopolska Province. The treatment plants are equipped with systems of mechanical-biological sewage treatment. The content of bioavailable forms was determined after a 24-hour extraction of a sample with redistilled water (material : water = 1 : 5). Phytotoxicity of the extracts (material : water = 1 : 5) prepared from unprocessed sewage sludge and from biochars was examined against *Lepidium sativum*. Thermal conversion of municipal sewage sludge, with reduced air access, caused an increase in the content of total forms of copper, cadmium, lead and zinc in stable products of the process. The process of thermal conversion caused a reduction in the content of water-soluble forms of the elements, which points to their lower bioavailability. Phytotoxicity of sewage sludge was influenced more by its origin, and in consequence by the type of sewage treated, than by thermal conversion.

**Keywords:** sewage sludge, biochar, heavy metals, thermal conversion, phytotoxicity.

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mgr inż. Monika Mierzwa-Hersztek, Department of Agricultural and Environmental Chemistry University of Agriculture in Krakow, Al. Mickiewicza 21, 30-120 Krakow, Poland, monika6\_mierzwa@wp.pl

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## INTRODUCTION

The increasing efficiency of municipal sewage treatment systems leads to generating large amounts of sewage sludge. Rational sewage sludge management is an important issue in every modern municipal sewage treatment plant (SIENKIEWICZ, CZARNECKA 2012). Based on demographic forecasts, it is estimated that the amount of sewage sludge produced in Poland will increase from 612 800 tons in 2010 to 706 600 tons dry matter in 2018 (WERLE, WILK 2010). Such solutions as disposal and temporary storage of sewage sludge at treatment plants or its use in land reclamation (including agricultural purposes) are becoming less popular, alternative methods of sewage sludge conversion are being sought. Methods of sewage sludge thermal conversion are becoming prevalent in Poland and globally (MAGDZIARZ, WERLE 2014)

One of the thermal methods is the conversion (under limited air access) of sewage sludge where this material undergoes various transformations and one of the final products is biochar (IBI 2012). These transformations affect not only physical but also chemical parameters of the material subjected to conversion, which may have an effect on living organisms once biochar has been returned to the environment (CHEN et al. 2014).

For agricultural purposes, the temperature of pyrolysis of the converted material should be about 300°C. Biochar produced at low temperature has lower values of pH, EC and higher concentrations of unstable organic carbon and dissolved organic carbon than biochar produced at high temperature (HALE et al. 2012, SONG, GUO 2012, AL-WABEL et al. 2013). Therefore, in terms of their favourable contribution to soils poor in organic matter content, biochars produced at low pyrolysis temperatures may have a preferred impact on agricultural and environmental systems (GUL et al. 2015). In this aspect, an assessment of the heavy metal content is important. Such an evaluation is also crucial for any environmental use of unprocessed municipal sewage sludge (*Regulation...* 2015). Thermal conversion of sewage sludge creates a risk of an increasing content and mobility of heavy metals. On the one hand, this is due to the loss of organic matter (compaction effect). On the other hand, degradation of various compounds, but at the same time formation of new or activation of initially unavailable chemically bound substances or elements, can occur very often during the thermal conversion of organic materials (HALE et al. 2012, OLESZCZUK et al. 2013). Therefore, this may bring into question the possibility of safe use of biochars from sewage sludge. The problem is particularly serious in the case of soils used for cultivation of plants intended to be used for food or fodder production.

Research results on properties and chemical composition of biochars of different origins that have been published so far bring to light some shortage of studies concerning the effect of these materials on living organisms. Relatively fast verification of the influence of various materials, including biochars, on living organisms can be achieved by means of tests on plants. Bio-

assays are an important part of bioanalytics and can be used for the environmental risk assessment, including the threat posed by application of various types of substances (BARAN, TARNOWSKI 2013, BEESLEY et al. 2014, GONDEK et al. 2014).

Many studies emphasize the considerable diversity of the chemical composition of municipal sewage sludge, depending on the type of sewage treated. Thus, the aim of our research was to determine the effect of thermal conversion of municipal sewage sludge on the content of total and water-soluble forms of Cu, Cd, Pb, Zn, and on the phytotoxicity of the solid products obtained in the process.

## MATERIAL AND METHODS

### Sewage sludge and the process of its thermal conversion

The research material consisted of stabilized sewage sludge from municipal sewage treatment plants in Krakow (SS1), Krzeszowice (SS2) and Słomniki (SS3), Malopolska Province. The treatment plants are equipped with systems of mechanical-biological sewage treatment.

Thermal conversion of air-dry sewage sludge was conducted under laboratory conditions, on a station designed for thermal processing of biomass at reduced air access (1-2%) (IBI 2012). 100 g samples were placed in a combustion chamber. The temperature inside the combustion chamber was  $300 \pm 10^\circ\text{C}$ . The exposure time was 15 minutes. The rate of heating the combustion chamber was  $10^\circ\text{C min}^{-1}$ . This choice of the duration and temperature of pyrolysis resulted from our preliminary research as well as the investigations conducted by others (AL-WABEL et al. 2013, GONDEK et al. 2014). The mass loss of the material (yield of pyrolysis) was calculated based on the amount of biomass that had been submitted to the process and its residue after thermal conversion (AGRAFIOTI et al. 2013) – Table 1.

Table 1

Values of selected chemical indicators in sewage sludge and biochars

Determination	(SS1)	(SS2)	(SS3)	CV (%)	(BCSS1)	(BCSS2)	(BCSS3)	CV (%)
Dry matter (%)	$30.6 \pm 10$	$25.2 \pm 12$	$19.2 \pm 7$	19	$96.3 \pm 8$	$96.7 \pm 10$	$97.4 \pm 9$	1
pH (H <sub>2</sub> O)	$5.41 \pm 0.01$	$6.99 \pm 0.00$	$6.90 \pm 0.11$	11	$6.85 \pm 0.08$	$7.06 \pm 0.04$	$7.18 \pm 0.04$	2
EC (mScm <sup>-1</sup> )	$59.2 \pm 1.50$	$22.4 \pm 1.2$	$12.2 \pm 0.5$	65	$44.8 \pm 7.3$	$6.7 \pm 0.4$	$5.0 \pm 0.5$	98
Nitrogen (% DM)	$2.93 \pm 0.08$	$3.89 \pm 0.06$	$2.27 \pm 0.13$	22	$2.54 \pm 0.57$	$3.44 \pm 0.18$	$3.09 \pm 0.76$	12
Carbon (% DM)	$18.8 \pm 0.10$	$26.5 \pm 0.2$	$19.4 \pm 0.9$	16	$17.8 \pm 0.4$	$25.2 \pm 0.1$	$18.2 \pm 0.6$	17
Sulphur (% DM)	$1.16 \pm 0.22$	$1.18 \pm 0.10$	$0.52 \pm 0.16$	32	$1.78 \pm 0.43$	$1.02 \pm 0.18$	$0.55 \pm 0.04$	45

Each value represents the mean of three replicates  $\pm$  standard deviation.

## Chemical analyses in sewage sludge and in biochars

The dry matter content in sewage sludge and in biochars was determined at the temp. of 105°C applied for 12 h (JINDO et al. 2012); pH of the material (material : water = 1 : 5) was determined electrochemically using a pH - meter (pH - meter CP - 505); electrolytic conductivity (material : water = 1 : 5) was determined using a conductivity meter (Conductivity/Oxygen meter CCO - 501) (MEIER et al. 2015). The content of total nitrogen, carbon and sulfur was determined in a vario MAX cube CNS analyzer (manufactured by Elementar 2013). The total content of cadmium, copper, lead and zinc was determined after the incineration of a sample in a chamber furnace at 450°C for 12 h and mineralization of the residue in a mixture of concentrated nitric and perchloric acid (3:2) (v/v). The content of bioavailable forms was determined after 24-hour extraction of a sample with redistilled water (material : water = 1 : 5) (GONDEK et al. 2014). Cu, Cd, Pb and Zn content in the solutions and extracts obtained was determined by inductively coupled plasma optical emission spectrometry (ICP-OES, Perkin Elmer Optima 7300 DV). All determinations were made in triplicate (OLESZCZUK 2007)

## Test of phytotoxicity of sewage sludges and biochars

Phytotoxicity of the extracts (material : water = 1 : 5) prepared from unprocessed sewage sludge and from biochars was examined against *Lepidium sativum*. The experimental design consisted of 7 treatments in triplicates: H<sub>2</sub>O – redistilled water, SS1 – sewage sludge from the sewage treatment plant in Krakow, BCSS1 – biochar from sewage sludge SS1, SS2 – sewage sludge from the sewage treatment plant in Krzeszowice, BCSS2 – biochar from sewage sludge SS2, SS3 – sewage sludge from the sewage treatment plant in Słomniki, BCSS3 – biochar from sewage sludge SS3. Seeds of *Lepidium sativum* (50 pieces) were placed in the extracts for 24 hours. Afterwards, they were placed on Petri dishes, padded with absorbent paper and moistened with redistilled water, and finally put into an incubator for 72 h (temp. 25°C ± 0.1). Next, the length of the plant roots was measured. Based on the results, the values of inhibition or stimulation coefficients were calculated, assuming that the treatment carried out using redistilled water was the reference.

## Statistical calculations

The standard deviation (SD) value was calculated for the results and, in order to determine the variation within the analyzed population, the coefficient of variation (CV) was calculated as the share of standard deviation (SD) in the arithmetic mean. The results on phytotoxicity were elaborated using single-factor analysis of variance (ANOVA) Mean values of the analyzed properties were compared using the Duncan multiple range test at the significance level of  $\alpha < 0.05$ . All statistical calculations were done with the use of Statistica 10 (StatSoft Inc.) software package.

## RESULTS AND DISCUSSION

### Selected chemical properties of sewage sludge, biochars and residue after thermal conversion

Municipal sewage sludge samples used in the research varied not only in chemical parameters (Table 1), but also in the source of their origin. The highest diversity among the parameters studied was observed in the value of electrical conductivity (EC) and in the sulfur content. The lowest diversity was found in the total carbon content and pH value.

Thermal conversion of sewage sludge produced different yields of biochar (residue after pyrolysis) – Figure 1. The least residue was recorded after the

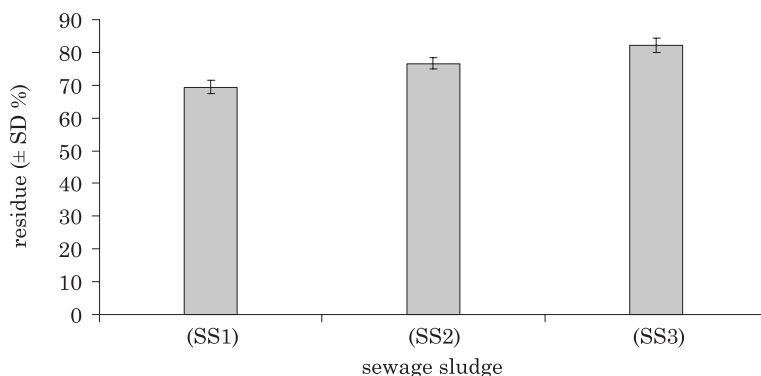


Fig. 1. The yields of sewage sludge pyrolysis

thermal conversion of sewage sludge from the sewage treatment plant in Krakow (SS1). This was due to the higher losses of substances undergoing oxidation in this sewage sludge. According to KARAYILDIRIM et al. (2006), organic matter in sewage sludge shows diverse susceptibility to oxidation. THIPK-HUNTHOD et al. (2006) also obtained varied results concerning the residue after thermal conversion of different kinds of sewage sludge, suggesting that differences in the mass loss of pyrolysed sewage sludge can result from quantitative and qualitative differences between components, including the ash content and the degree of fineness. This is an important conclusion, especially as sewage sludge in our study had different origin, which undoubtedly had some impact on the chemical composition of particular samples, including the stability of organic compounds. HOSSAIN et al. (2011) showed that the mass loss of materials subjected to thermal conversion, apart from their quantitative and qualitative composition, is affected by the temperature at which the process is conducted.

## Content of total forms and of Cu, Cd, Pb and Zn extracted with water in sewage sludge and biochars

As a result of the thermal conversion of municipal sewage sludge, there was an increase in the total content of all the elements studied (Table 2). In

Table 2

The total content of Cd, Cu, Pb and Zn in sewage sludge and biochars

Determination	(SS1)	(SS2)	(SS3)	CV (%)	(BCSS1)	(BCSS2)	(BCSS3)	CV (%)
Cd (mg kg <sup>-1</sup> DM)	1.97 ± 0.03	2.94 ± 0.00	0.86 ± 0.00	44	2.79 ± 0.09	3.87 ± 0.05	1.12 ± 0.04	44
Cu (mg kg <sup>-1</sup> DM)	338 ± 7	137 ± 2	81 ± 1	60	467 ± 14	182 ± 6	100 ± 6	63
Pb (mg kg <sup>-1</sup> DM)	60 ± 0	54 ± 0	18 ± 0	42	81 ± 2	70 ± 1	22 ± 1	44
Zn (mg kg <sup>-1</sup> DM)	1680 ± 26	1069 ± 4	589 ± 14	40	2321 ± 30	1436 ± 26	723 ± 30	44

Each value represents the mean of three replicates ± standard deviation.

their research on the effect of temperature on the content of macronutrients in biochar from sewage sludges, HOSSAIN et al. (2011) stated that thermal conversion of such material increased the concentration of trace elements. According to GASKIN et al. (2008), mineralogical composition of biochars is conditioned by the kind of substrate used in the process. These authors confirm an increase in the content of ash components, at the same time indicating that a higher temperature may result in an increase in the content of mineral components in biochar. The reason is the depletion of substances of organic character.

The results of our research indicate that the thermal conversion of sewage sludge effected a decrease in the mobility of the analyzed trace elements in comparison to the original, unprocessed sewage sludge (Table 3). Also, LIU

Table 3

The content of water-soluble forms of Cu, Cd, Pb, Zn extracted from sewage sludge and biochars

Determination	(SS1)	(SS2)	(SS3)	CV (%)	(BCSS1)	(BCSS2)	(BCSS3)	CV (%)
Cd (mg kg <sup>-1</sup> DM)	0.30 ± 0.00	0.20 ± 0.01	0.10 ± 0.02	41	0.10 ± 0.00	0.05 ± 0.01	0.10 ± 0.00	28
Cu (mg kg <sup>-1</sup> DM)	1.70 ± 0.10	0.20 ± 0.02	0.05 ± 0.00	115	0.00	0.00	0.00	-
Pb (mg kg <sup>-1</sup> DM)	1.65 ± 0.05	1.05 ± 0.05	0.85 ± 0.05	29	1.00 ± 0.00	0.45 ± 0.05	0.40 ± 0.00	44
Zn (mg kg <sup>-1</sup> DM)	8.16 ± 0.6	9.00 ± 0.00	6.30 ± 0.60	108	3.75 ± 0.65	2.35 ± 0.25	0.65 ± 0.10	56

Each value represents the mean of three replicates ± standard deviation.

et al. (2014) point out a reduction in the content of bioavailable forms of trace elements. However, HE et al. (2010) showed that pyrolysis temperature above 350°C leads to a higher stability of forms of such trace elements as Zn and Cu. According to KLOSS et al. (2012), an increase or decrease in the content of mobile forms of heavy metals in sewage sludge under thermal conversion is conditioned by the presence of aliphatic bonds and formation of more

stable aromatic bonds. GONDEK et al. (2014) confirmed this theory experimentally.

### Phytotoxicity of the extracts from sewage sludge and biochars

Results of the tests on the phytotoxicity of the extracts prepared from unprocessed and thermally converted sewage sludge are shown in Table 4.

Table 4

The toxicity test on *Lepidium sativum* L. root growth

Determination	H <sub>2</sub> O	(SS1)	(SS2)	(SS3)	CV (%)	(BCSS1)	(BCSS2)	(BCSS3)	CV (%)
The length of the roots (mm)	50.1b ± 2.1	33.7a ± 1.9	32.9a ± 2.0	37.2a ± 0.3	5	53.2b ± 1.6	53.2b ± 1.5	37.5a ± 3.2	15
Inhibition / stimulation (%)	100	67.2a ± 1.0	65.8a ± 5.4	74.4a ± 3.5	-	106.5b ± 6.5	106.5b ± 5.2	75.0a ± 6.2	-

Each value represents the mean of three replicates ± standard deviation. The different letters within a column indicate a significant difference at  $p \leq 0.05$  according to the Duncan's multiple range tests.

The tests revealed a significantly adverse effect of the extracts from raw sewage sludge (SS1, SS2, SS3) and from thermally converted sewage sludge from the sewage treatment plant in Słomniki (BCSS3) on the root growth of *Lepidium sativum*. Conversely, the thermal conversion of sewage sludge from the sewage treatment plants in Krakow (BCSS1) and Krzeszowice (BCSS2) resulted in the extracts having a considerably more beneficial effect on *Lepidium sativum* root growth than the raw sewage sludge from these plants. The thermally converted sewage sludge extracts did not exert any significant effect on *Lepidium sativum* root growth, except BCSS3. Significant stimulation of root growth relative to the control was observed after an application of BCSS1 and BCSS2 extracts. Also Fuentes et al. (2004) reported that sludge extracts did not have significantly negative effects on relative seed germination of barley (*Hordeum vulgare* L.) or cress (*Lepidium sativum* L.), although a certain reduction in the germination index indicates some adverse effect on root growth. Another study carried out by GONDEK et al. (2014) showed low toxicity of extracts prepared from mixtures of thermally converted sewage sludge with plant materials against *Vibrio fischeri* and *Lepidium sativum*. *Lepidium sativum* showed higher sensitivity to substances occurring in the said extracts than *Vibrio fischeri*. OLESZCZUK et al. (2013) showed somewhat different dependences, namely the biochars they examined were more toxic to *Vibrio fischeri* than to *Lepidium sativum*.

## CONCLUSIONS

1. Thermal conversion of municipal sewage sludge, with reduced air access, caused an increase in the content of total forms of copper, cadmium, lead and zinc in the biochars obtained.

2. Thermal conversion of municipal sewage sludge caused a decrease in the content of water-soluble forms of copper, cadmium, lead and zinc in stable products of the process, pointing to their lower bioavailability.

3. Phytotoxicity of biochars from sewage sludge was influenced more by the origin of sludge than by its thermal conversion.

4. Extracts from thermally converted sewage sludge did not exert any significant effect on *Lepidium sativum* root growth, except BCSS3. Significant stimulation of root growth relative to the control was observed only after BCSS1 and BCSS2 extracts had been used.

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