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Characteristics of selected fractions of heavy metals in biologically and thermally transformed sewage sludge

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ABSTRACT

Transformation of sewage sludge is basically a multistage and technologically complex process. Obtaining effective and cost-effective results of sewage sludge processing for environmental applications is an important issue in this waste management. Additional enrichment of sewage sludge during its process of treatment into biowaste, such as bark, straw, or sawdust, gives the sludge appropriate physical properties which facilitate their later transformation or immediate application. On the basis of conducted investigations, it was stated that total heavy metal content in the tested mixtures of sewage sludge with straw, sawdust, and bark transformed thermally and biologically does not exclude their use for agricultural or reclamation purposes, whereas a supplement of the above-mentioned components causes an effect of diluting the content of all determined heavy metals. On the other hand, the process of thermal transformation of the mixtures contributed to increasing Cd, Pb, Cu, and Zn content. No such marked content of total forms of investigated elements was registered in the mixtures transformed biologically. Cadmium was an exception. Sequential analysis of the studied heavy metals confirmed changes in mobile forms that share in the content of analyzed elements.

Keywords: Sewage sludge; Mobile forms; Transformation; Heavy metal

1. Introduction

The issue of sewage sludge management currently poses one of the major environmental problems in Poland. The National Waste Management Plan [1] as one of the most important matter considers, among others, a considerable percentage of deposited sewage sludge and lack of overall solutions of its management. The necessity of sewage sludge disposal is determined by legal, practical, and aesthetic reasons [2]. At the same time, these materials are wastes with a considerable content of nutrients for plants and organic matter which argues for their environmental

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use. Sewage sludge, due to its origin, may contain a considerable amount of heavy metals and other toxic substances. Thus, its environmental application besides legal requirements should also consider the principles of good agricultural practice [2,3].

Taking into consideration the criterion of heavy metal content in sewage sludge as one of the elements allowing for environmental application of this material, one should state that a total content of such elements is not an efficient index of their bioavailability assessment. It only reflects the degree to which a material is burdened with the polluting agent. Determining the forms of heavy metals in the sludge by means of sequential chemical extraction seems to be a better solution permitting for risk assessment of the negative impact of its application on living organisms [3–5].

The aim of the presented experiment was to determine chemical forms of cadmium, lead, zinc, and copper in not-transformed sewage sludge, and in the mixtures of sewage sludge with biologically and thermally transformed biodegradable materials taking into account their environmental applications.

2. Materials and methods

The initial material was stabilized, sewage sludge collected from Wastewater Treatment Plant Cracow—"Płaszów", Poland. The plant uses a mechanical–biological sewage treatment system and treats predominantly municipal sewage with some portion of industrial wastes.

Sewage sludge was mixed with biodegradable materials: wheat straw, sawdust, and conifer bark in the gravimetric ratio 1:1 in conversion to material dry matter. The selection of materials used as components was determined by their availability as well as their positive impact on sludge physical properties. Chemical composition of the materials used for the experiment is presented in Table 1.

The experiment comprised the following variants:

Object I—sewage sludge + wheat straw

Object II—sewage sludge + conifer sawdust

Object III—sewage sludge + conifer bark.

The mixtures were transformed in two ways separately: thermally and biologically.

The process of biological transformation of sewage sludge mixtures with various supplements was conducted under laboratory conditions. The mixture of materials was moistened to 55% and then placed in 0.25 m high x 0.45 m long x 0.32 m wide openwork containers enabling its aeration and draining the leachate. Subsequently, the containers were put into a bioreactor isolated from ambient conditions by additional aeration system. During the experiment, the temperature and biomass moisture content were under control in order to maintain optimal conditions for the process. Biomass was aerated in the system of 0.01 $m^3 min^{-1}$ four times per day. Additionally 1–2 times a week, biomass was taken out from the reactor and mixed manually. Two stages were identified during the composting process: the first stage—lasting 30 d, when the temperature of composted mixtures stayed at between 38–58 ˚C, and water loss was supplemented in 2 d cycles and stage II—lasting 87 d during which the temperature of composted mixtures stayed at between 20–30 ˚C, and water loss was supplemented in 4 d cycles.

The thermal transformation process of sewage sludge and supplements mixture was also conducted under laboratory conditions. The mixtures were put in porcelain dishes in layer not exceeding 1 cm and exposed to temperatures of 30 ˚C for 5 min → 130 ˚C for 40 min → 200 ˚C for 30 min in a chamber furnace with no air access. When the procedure was completed, the material was left to cool.

The following assessments were made in the ground initial material (sewage sludge, wheat straw, sawdust, and bark), in organic material mixtures

Table 1
Chemical composition of not-transformed sewage sludge and biodegradable materials used for mixture preparation

Determination	Sewage sludge	Wheat straw	Conifer sawdust	Conifer bark
Dry matter ($g\,kg^{-1}$)	236	990	989	989
Organic matter ($g\,kg^{-1}$ dm)	523 ± 1	967 ± 4	996 ± 2	547 ± 17
Total forms ($mg\,kg^{-1}$dm)				
Cd	4.25 ± 1.17	0.38 ± 0.02	0.30 ± 0.01	0.41 ± < 0.01
Pb	61.6 ± 6.1	1.32 ± 0.12	0.23 ± 0.02	12.18 ± 0.88
Cu	351.2 ± 66.2	1.38 ± 0.10	0.84 ± 0.04	5.55 ± 0.13
Zn	1823 ± 74	291 ± 1	133 ± 5	429 ± 5

Note: ± standard deviation (SD), $n = 3$.

Table 2
Conditions of sequential extraction acc. to Tessier et al. [6]

Fraction		Extractant	pH	m/v	Time (h)	Temperature (°C)
F1	Ion exchangeable	1 mol dm^{-3} $MgCl_2$	7.0	1:8	1	Room
F2	Carbonate	1 mol dm^{-3} CH_3COONa	5.0	1:8	5	Room
F3	Bound to iron and manganese oxides	0.04 mol dm^{-3} $NH_2OH{\cdot}HCl$ + 25% CH_3COOH	2.0	1:20	6	96 ± 2°C
F4	Bound to organic matter	(a) 0.02 mol dm^{-3} HNO_3 + 30% H_2O_2	2.0	1:8	2	85 ± 2°C
		(b) 30% H_2O_2	2.0	1:3	3	85 ± 2°C
		(c) 3.2 mol dm^{-3} CH_3COONH_4 + 20% HNO_3	2.0	1:5	0.5	Room
F5	Residual	$HClO_4$ + HNO_3 (3:2, v/v)	–	1:12	–	–

prior to transformation and in the material obtained as a result of biological and thermal transformations:

- dry matter content after sample drying at 105°C for 12 h,
- organic matter content after sample incineration in a chamber furnace at 500°C,
- total contents of trace elements after organic sample incineration in a chamber furnace (450°C for 12 h) and the remains of mineralization in a mixture of concentrated nitric (V) and chlorous (VII) acids (3:2) (v/v) by means of ICP-AES method in JY 238 Ultrace apparatus (Jobin Yvon),
- forms of selected trace elements in sewage sludge and transformed mixtures of sewage sludge with straw, sawdust, and bark were assessed using sequential chemical extraction [6]. The experimental design comprised: not-transformed sewage sludge (SS), mixtures of sewage sludge + straw (BSS + WS), sewage sludge and sawdust (BSS + CS), sewage sludge and bark (BSS + CB) transformed biologically and thermally transformed mixtures of sewage sludge and straw (TSS + WS), sewage sludge and sawdust (TSS + CS), and sewage sludge and bark (TSS + CB). Five fractions of studied trace elements were identified in accordance with the described procedure in Table 2, i.e. an ion exchangeable fraction, a carbonate fraction, a oxide fraction, and a residual fraction. The content of studied heavy metals in the obtained extracts was determined using ICP-AES method on JY 238 Ultrace apparatus (Jobin Yvon).

3. Results and discussion

Subjecting by-product or waste material to biological and thermal treatment affects not only the change of total heavy metal content but also influences some mutual changes between forms in which that element occurs. As a result of transformations conducted under aerobic conditions using a biological agent and irrespective of the technology, constant changes in the trace element bond forms occur through such complex reactions as precipitation and sorption. Under such conditions bonds with iron and manganese oxides, organic compounds, carbonates and sulfides are of major importance [7]. There is some data given in various literary sources concerning transformations of heavy metal forms, among others, in thermally transformed sewage sludge. The conducted research is currently focused mainly on the identification of the total content of trace elements in ashes of sewage sludge transformed at high temperatures [8]. On the other hand, there is no data concerning the impact of low-temperature process of the material transformation on the contents of various heavy metal forms.

The share of individual heavy metal fractions in the total content was mainly determined by properties of the element itself and the method of mixture transformation, whereas the kind of added plant waste did not modify this property to any marked degree.

The content of total cadmium forms in the analyzed mixtures of sewage sludge with biodegradable materials, irrespective of the method of transformation, was lower than Cd content assessed in not-transformed sewage sludge (Table 3).

Irrespective of the method of transformation, the highest amount of total cadmium forms was assessed in the mixture of sewage sludge and straw (BSS + WS, TSS + WS) (Table 3). The highest content of cadmium accumulated in potentially bio-available fractions (F1, F2, and F3) was assessed in biologically transformed mixtures irrespective of the biodegradable material added to sewage sludge (Table 4).

Table 3
Content of total forms of analyzed heavy metals in sewage sludge and in biologically and thermally transformed mixtures

Material*	Cd mg kg^{-1}dm	Pb	Cu	Zn
SS	4.25 ± 1.17	61.6 ± 6.1	351 ± 66	1823 ± 74
BSS + WS	2.42 ± 0.32	46.6 ± 0.8	228 ± 1	1403 ± 2
BSS + CS	1.68 ± 0.14	42.4 ± 0.7	196 ± 7	1269 ± 21
BSS + CB	1.55 ± 0.07	41.9 ± 2.5	175 ± 10	1133 ± 77
TSS + WS	2.20 ± 0.11	56.0 ± 3.6	299 ± 24	1812 ± 140
TSS + CS	1.80 ± 0.07	51.8 ± 2.4	265 ± 24	1662 ± 90
TSS + CB	1.30 ± 0.02	45.6 ± 6,5	202 ± 28	1322 ± 184

*Objects as in materials and methods; ± standard deviation (SD), $n = 3$.

The use of a thermal method in order to transform prepared mixtures caused a decrease in cadmium accumulation in its most mobile fractions. Irrespective of the biodegradable material added to the sewage sludge, the element was to a slight degree (on average 1.38% of total Cd content) bound with the organic matter (F4) of transformed mixtures. Cadmium share in this fraction in thermally transformed mixtures ranged from 7 to 12% of the total content. The highest quantity of cadmium, irrespective of the biodegradable material added to sewage sludge or the transformation method used, was accumulated in the residual fraction (F5) (Table 4). However, it should be emphasized that the contents were lower than assessed in sewage sludge used for preparing the mixtures. In research of Jakubus and Czekała [9] and Latosińska and Gawdzik [3], approximate cadmium contents were accumulated in the most mobile fractions. Both in the studies conducted by Ayari et al. [10] and Castaldi et al. [11] on composts from municipal wastes, much higher cadmium concentrations were registered in exchangeable and carbonate fractions. However, a considerably higher total concentration of this element assessed in the analyzed organic material was confirmed by above-mentioned authors. On the other hand, Castaldi et al. [11] did not find any increased cadmium concentration in water-soluble form, which they explained by immobilization of the metal in a residual fraction.

The contents of total lead forms in the analyzed mixtures of sewage sludge and biodegradable materials, similar to cadmium and irrespective of the transformation method, was lower than Pb concentration assessed in not-transformed sewage sludge. A decreased content of total lead forms in the biologically transformed mixtures, as compared with the content in not-transformed sewage sludge, was on average about 30% whereas in transformed mixtures it was on average 17%. Irrespective of the transformation method, the highest content of total lead forms was assessed in the mixture of sewage sludge and straw (Table 3). Lead in the sewage sludge used for preparation of mixtures and in the biologically and thermally transformed mixtures occurred almost exclusively in a residual fraction (F5) (Table 5).

The share of the sum of other element forms, regardless of the supplement and transformation method, did not exceed 2% of the total content. The results obtained by Gawdzik and Latosińska [3] point to a slight (not exceeding 16% of total content) mobility of this element in sewage sludge. Chen et al. [4] explain a relatively low Pb mobility in these materials by occurrence of lead in the form of insoluble salts. Lead reveals very high affinity to humic acids with which it forms durable complexes through oxide ligands [7]. The authors' own investigations did not determine any significant content of this element in fraction bound to organic matter. Sidełko [7] and Amir et al. [6] found a decrease in lead mobile forms in composted sewage sludge in comparison with the

Table 4
Fractions of cadmium bonds in sewage sludge and mixtures of sewage sludge with biodegradable material after biological and thermal transformation

Material*	F1 mg Cd kg^{-1}dm	F2	F3	F4	F5
SS	0.41 ± 0.03	0.25 ± 0.01	0.37 ± 0.03	0.03 ± 0.01	3.19 ± 1.10
BSS + WS	0.33 ± 0.02	0.16 ± 0.03	0.32 ± 0.02	0.02 ± 0.03	1.59 ± 0.28
BSS + CS	0.20 ± 0.02	0.11 ± 0.04	0.25 ± 0.01	0.01 ± 0.02	1.11 ± 0.07
BSS + CB	0.21 ± < 0.01	0.10 ± < 0.01	0.18 ± < 0.01	0.05 ± 0.07	1.01 ± 0.21
TSS + WS	0.01 ± < 0.01	0.04 ± < 0.01	0.40 ± 0.02	0.19 ± 0.11	1.56 ± 0.07
TSS + CS	Traces	0.03 ± 0.03	0.25 ± 0.07	0.12 ± 0.01	1.40 ± 0.01
TSS + CB	Traces	0.01 ± 0.02	0.27 ± 0.07	0.16 ± 0.01	0.86 ± 0.19

*Objects as in materials and methods; ± standard deviation (SD), $n = 2$.

Table 5
Fractions of lead bonds in sewage sludge and mixtures of sewage sludge with biodegradable materials after biological and thermal transformation

Material*	F1 mg Pb kg^{-1} dm	F2	F3	F4	F5
SS	Traces	0.09 ± < 0.01	Traces	Traces	61.51 ± 6.08
BSS + WS	0.09 ± 0.03	0.34 ± 0.08	Traces	Traces	46.15 ± 0.68
BSS + CS	0.08 ± 0.01	0.31 ± 0.07	Traces	Traces	42.00 ± 3.75
BSS + CB	0.11 ± 0.05	0.35 ± 0.01	Traces	0.16 ± 0.03	41.23 ± 0.91
TSS + WS	Traces	0.41 ± 0.08	Traces	0.13 ± 0.23	55.46 ± 2.29
TSS + CS	Traces	0.31 ± 0.07	Traces	Traces	51.43 ± 2.47
TSS + CB	0.02 ± 0.03	0.27 ± 0.01	Traces	Traces	45.26 ± 6.53

*Objects as in materials and methods; ± standard deviation (SD), $n = 2$.

quantities assessed in raw sewage sludge, which the authors explain as mineralization of organic matter. It should be noted that the application of higher temperature during thermal transformation of the mixtures did not cause any changes in mobile Pb forms of the material in comparison with biologically transformed mixtures. According to many authors [12–14], trace element concentrations differ depending on the kind of biomass subjected to thermal transformation, still the problem of a considerable content does not concern lead.

The content of copper mobile forms in the analyzed mixtures of sewage sludge and biodegradable materials, regardless of the transformation method, was lower than Cu concentrations assessed in not-transformed sewage sludge (Table 3). It was caused by an addition to the sewage sludge of biodegradable materials characterized by relatively low concentration of this element. Obviously, more copper total forms were registered in thermally transformed mixtures in comparison with biologically transformed, which resulted from a bigger weight loss. Irrespective of the transformation method, the highest amount of total copper forms, similar to cadmium and lead, was determined in the mixture of sewage sludge and straw (BSS + WS, TSS + WS) (Table 3). The share of copper accumulated in ion exchangeable fraction (F1), carbonate (F2), and in the fraction bound to iron and manganese oxides (F3) in a total content of this element in biologically transformed materials was the highest in the sewage sludge mixture with straw (BSS + WS), i.e. 7%, similar to the one in the sewage sludge used for preparing the mixture (SS) (Table 6).

In transformed mixtures, the share of most mobile copper fractions (F1, F2, and F3) in a total Cu content did not exceed 2%. Copper was strongly bound with organic matter (F4) and a residual fraction (F5) of the transformed mixtures as corroborated by the studies of other authors [3,4]. A small content of copper in best bio-available forms may increase formation of high-molecular weight humus bonds determining copper compound solubility. They indicate considerable Cu contents assessed in fraction bound to organic matter. Castaldi et al. [11] indicated similar relationships. They found a fast increase in copper concentrations in water extract during the initial period of biological transformation of municipal wastes and then, its decrease after 100 d. According to Agrafioti et al. [12], the copper content in materials obtained as a result of biomass thermal transformation may prove to be an indicator of the environmental application of these wastes.

The content of total zinc forms in analyzed mixtures of sewage sludge with biodegradable materials was lower than Zn concentration assessed in not-transformed sewage sludge, regardless of the transformation method (Table 3). A smaller amount of zinc in the prepared mixtures, irrespective of the transformation method, resulted from diluting Zn content. No matter what transformation method was used, the greatest amount of total zinc forms was determined, as it was in the case of previously discussed elements in the sewage sludge mixture with straw (BSS + WS, TSS + WS) (Table 3). It should be noted that these mixtures were characterized by the greatest weight loss during their transformation. The greatest quantities of zinc were accumulated in the fraction bound to manganese and iron oxides (F3) (Table 7).

The contents ranged from 287 mg Zn kg^{-1}dm in biologically transformed sewage sludge mixture with bark (BSS + CB) to 490 mg Zn kg^{-1}dm in biologically transformed mixture of sewage sludge and straw (BSS + WS). Moreover, a residual fraction (F5) played a key role in zinc binding, particularly in thermally transformed mixtures. It should be emphasized that

Table 6
Fractions of copper bonds in sewage sludge and mixtures of sewage sludge with biodegradable materials after biological and thermal transformation

Material*	F1 mg Cu kg^{-1} dm	F2	F3	F4	F5
SS	6.16 ± 0.25	9.86 ± 0.07	8.79 ± 1.11	214.07 ± 6.47	111.91 ± 66.94
BSS + WS	2.09 ± 0.44	3.01 ± 0.23	11.28 ± 0.44	67.75 ± 16.19	144.30 ± 7.13
BSS + CS	0.65 ± 0.03	1.31 ± 0.26	3.99 ± 1.20	94.12 ± 8.37	95.60 ± 22.46
BSS + CB	0.54 ± 0.03	0.57 ± 0.04	1.84 ± 0.02	96.57 ± 0.56	75.35 ± 16.61
TSS + WS	0.02 ± < 0.01	0.12 ± 0.04	0.92 ± 0.08	20.83 ± 2.92	276.74 ± 30.00
TSS + CS	0.01 ± 0.02	0.15 ± 0.02	0.75 ± 0.10	17.80 ± 2.66	246.71 ± 8.38
TSS + CB	0.58 ± 0.67	0.35 ± 0.35	1.04 ± 0.10	20.44 ± 12.64	179.69 ± 25.16

*Objects as in materials and methods; ± standard deviation (SD), $n = 2$.

Table 7
Fractions of zinc bonds in sewage sludge and sewage sludge mixtures with biodegradable materials after biological and thermal transformation

Material*	F1 mg Zn kg^{-1} dm	F2	F3	F4	F5
SS	188.9 ± 2.6	371.2 ± 3.7	725.1 ± 29.5	475.3 ± 5.0	63.5 ± 43.5
BSS + WS	278.4 ± 32.1	190.0 ± 12.6	490.4 ± 12.1	256.1 ± 11.1	188.3 ± 76.3
BSS + CS	239.1 ± 7.0	151.7 ± 26.1	364.3 ± 18.9	233.4 ± 5.1	280.2 ± 97.3
BSS + CB	247.5 ± 6.2	107.6 ± 5.4	287.5 ± 0.1	231.5 ± 19.2	259.2 ± 41.0
TSS + WS	33.7 ± 1.5	54.7 ± 4.2	461.3 ± 19.7	386.9 ± 89.5	875.5 ± 16.7
TSS + CS	27.0 ± 1.3	38.4 ± 0.5	360.8 ± 80.6	315.0 ± 21.8	921.0 ± 19.4
TSS + CB	14.1 ± 0.9	38.4 ± 1.8	425.3 ± 3.5	347.0 ± 35.2	497.2 ± 155.2

*Objects as in materials and methods, ± standard deviation (SD), $n = 2$.

regardless of applied supplement of biodegradable material, both biological and thermal method of mixtures transformation contributed to a considerable increase in Zn concentrations in this fraction in comparison with not-transformed sewage sludge (SS). The analysis of zinc content in ion exchangeable (F1) and carbonate (F2) fractions demonstrated an evident decrease in the share of a total content which resulted from the impact of a thermal agent used to transform prepared mixtures, both in comparison with biologically transformed mixtures and not-transformed sewage sludge. Strong zinc binding by iron and manganese oxides was also confirmed by research of Gawdzik and Gawdzik [15]. This relation may be explained by a considerable content of iron in sewage sludge resulting from adding iron sulfate as PIX in order to precipitate phosphorus compounds on which zinc sorption may take place [16]. Results of dynamics of mobile zinc forms changes during municipal wastes composting obtained by Castaldi et al. [11] indicate a fast decrease in zinc forms transferring to water extract, which was not confirmed by authors' research findings. According to the quoted authors, it may have been due to an increase in high-molecular weight humus compounds which form during transformation of composted biomass. Research conducted by Ciesielczuk et al. [14] showed that the process of thermal biomass transformation causes a considerable zinc accumulation in transformed materials, which may reduce their application. However, as confirmed by research findings, the process of thermal transformation clearly contributes to increasing the availability of zinc fractions, including the residual fraction, which will significantly determine this element passing into the soil solution.

4. Conclusions

- Contents of selected trace elements in biologically and thermally transformed sewage sludge mixtures with biodegradable materials do not exceed limit value determined for environmental application of sewage sludge.
- The contents of analyzed heavy metals in ion exchangeable, carbonate fractions, and fraction

bound with iron and manganese oxides were more affected by biological transformation, whereas thermal transformation more influenced metal concentrations in fractions bound with organic matter and in residual fraction.

- Applied methods of prepared mixture's transformation affected an increased, in comparison with not-transformed sewage sludge, content of less available forms of heavy metals, which diminishes the risk of environmental applications of these materials.

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