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ELUTION OF HEAVY METALS FROM GRANULATES PRODUCED FROM MUNICIPAL SEWAGE DEPOSITS AND FLY-ASH OF HARD AND BROWN COAL IN THE ASPECT OF RECYCLING FOR FERTILIZATION PURPOSES

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Abstract: Deposits used as fertilizer bring to soil both biogens necessary for plant growth and other ingredients such as metals, including heavy metals. Knowledge of quantities and rate in which heavy metals are to be released to soil from granulates is important because of their toxic influence on plants (in the case of high metals concentration). This paper presents results of investigation of elution of Cu, Zn, Ni, Cd, Pb, and Cr from granulates prepared from municipal sewage sludge, hard coal ash and brown coal ash. Elution to water solution was carried out in static conditions with single-stage and tree-stage extraction. Heavy metal a component of sludge-ash granulates eluted in various quantities, i.e. from trace for cadmium to 9.26–9.53 mg/kg of d.m. for zinc. Among the soluble forms of metals the most mobile are (in decreasing sequence): Cu > Pb > Zn > Ni in granulates containing brown coal ash and Cu > Pb > Ni > Zn in granulates contain hard coal ash.

INTRODUCTION

The easiest and the most rational method of ecologic utilization of sewage deposits is their utilization as fertilizers, not only to supply nutritional elements but also to improve or maintain soil structure. Fertilizer, and soil-improvement properties of municipal sewage deposits result from the presence of organic substances – important biogens, such as nitrogen, phosphorus, magnesium, calcium and microelements, including Cr, Sn, Zn, F, J, Co, Si, Mn, Cu, Mo, V. If, however, specified metals occur in deposits in excessive concentrations, they become undesired or even toxic soil components [1, 2, 14, 19]. Very high toxicity has been revealed for Cd, Hg, Pb, and As [14].

Natural utilization of deposits, included in soil fertilization, requires that deposits meet specific legal requirements concerning the content of heavy metals, pathogens, and

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consistence with conditions ability of correct application [16]. Another, quite different, type of deposits is mineral deposits, which can also be naturally utilized, such as fly-ash from combustion of hard coal and brown coal. Due to their physical and chemical properties they improve productivity of light sandy soils and agricultural reclamation of very heavy soils, by improving their physical and chemical properties, alkalization of acid soils, etc. Multiple research projects were completed on suitability of deposits in cultivation of consumable plants (crops, vegetables, grapevine) and in fertilization of grasslands and forests [10, 14, 20].

Mineral deposits used as fertilizer bring to soil mostly calcium and magnesium, although fly-ash from combustion of hard coal are not as rich in biogens as fly-ash from brown coal [11]. Adding fly-ash to light acid soils de-acids them, improves their sorptional capacity, saturation with alkali cations, improves circulation of water and air in surface soil level by changing its structure, as well as enhances microbiological processes in soil [10]. In addition to macro-components fly-ash contains also micro-components, just as is the case of sewage deposits. Fly-ash micro-component content is very significant from the point of view of their potential effect on living organisms, although their share in total fly-ash weight is as low as 1–3%. As compared with coal in fly-ash those components are more concentrated, with exception of mercury, which undergoes oxygenation in furnace [20]. Trace elements in fly-ash feature relatively low solubility, which is due to their position within hardly eroding halo of aluminosilicate matrix [5]. Solubility of hard coal ash is ca. 2% and of brow coal ash is up to 10%. [14].

Disadvantage of agricultural use is their dusting and tendency to aggregate when improper grainage is adopted [4]. Due to the above physical, chemical and biological properties of sewage deposits and physical and chemical properties of fly-ash, from the point of view of natural utilization, the most beneficial would be to solidify them in non-dusting granular form, suitable for applications in agriculture. Binding properties of fly-ash cause their good solidification with sewage deposits, reducing emission of unpleasant odors, and alkalinization properties reduce threats from pathogens in deposits [13, 21]. With high reaction cations of multiple value elements transform into insoluble or hardly soluble forms, e.g. carbonates or hydroxides, which can limit or inhibit elution of heavy metals from granulates [10]. Jarema-Suchorowska [3] states that extracts from eluted granulates and stabilizates prepared from sewage deposits and hard coal fly-ash feature many times lower contamination indices than extracts from elution of only sewage deposits.

Ash-sludge composites have better physical and chemical properties which enable their transport and disposal, compared to the waste material they originate from. Moreover, those composites contain a significant amount of organic matter and calcium compounds, which regulate soil reaction, and as such may be successfully utilized for land improvement and reclamation of arable as well as degraded soils using conventional agricultural equipment for fertilizer or manure application [12, 21].

The technology of sewage sludge and fly ash processing to mineral and organic composites is related to the application of a special technological installation with plate or drum granulators enabling mixing and application of different fly ash types and doses. There is no dust emission from granulates during transportation and disposal, they are

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characterized by a round shape of grains, mechanical resistance, equal distribution of grain composition and good cohesion [3, 11].

GOAL AND OBJECT OF THE RESEARCH

The goal of the research described in this work was to determine the effect of deposit and ash granulates based on hard coal fly-ash, brown coal fly-ash and municipal sewage deposits in selected proportions, on elution of metals, as well as determination if prepared granulates could be safely used in nature.

METHODS

Total content of Cu, Zn, Cd, Ni, Pb, Cr in raw materials and granulates was determined in accordance with [6] upon prior mineralization of sewage sludge and granulate specimens as per [8] (digestion for further marking of elements soluble in *aqua regia*) and fly ashes according to [7] (microwave digestion for further marking of elements soluble in mixture of acids HF, HNO_3 and HCl). Mecury was determined from air-dry sample using Hg analyzer AMA 254.

In the study a single-stage extraction and three-stage extraction were carried out. A single-stage extraction was carried out according to the presently suspended Regulation [18] and a three-stage extraction according to the Polish Norm [9] (preparation of water extract).

Granulate samples were poured with distilled water (1 part of dry matter to 10 parts of water) and extracted as presented in Table 1. Extraction was carried out at room temperature. The obtained extracts in one and three-stage tests of elution were filtered through a filter paper and analyzed. Heavy metals were determined using atomic absorption spectrometry (AAS) on UNICAM-PHILIPS PU 9100 X spectrometer. Loads of eluted contaminants were calculated according to the formula below:

$$A = (C \times L)/M_D$$

A – load of eluted contaminant [mg/kg],

C – concentration of individual component in extract [mg/dm^3],

L – water volume used [dm^3],

M_D – weight of the granulate dry matter [kg].

Table 1. Single-stage and three-stage extraction procedures

Type of extraction	Steps of extraction and duration [h]		Total extraction time [h]
Single-stage extraction	shaking sedimentation shaking sedimentation	4 12 2 6	24
Three-stage extraction	rest each of 3 extraction stages: shaking sedimentation shaking sedimentation	1 4 16 4 2	78

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CHARACTERISTICS OF THE INVESTIGATED MATERIAL

The above granulates were prepared as follows: upon methane fermentation and dehydration sewage deposit was mixed with hard coal fly-ash and brown coal fly-ash, respectively in 7:3 ratio (by weight of dry mass). Auxiliary component used was hydrated lime (3.3% of the quantity of used fly-ash), and fertilization value modifier used was standard potassium fertilizer – KCl (23% of total quantity of all components). Granulation was carried out by using the ring type mould granulator. Granulate prepared basing on brown coal ash (a:d = 7:3) with symbol N41/m featured little higher mechanic strength (compression strength) than granulate (a:d = 7:3) with symbol N42/m, produced using hard coal ash. Brown coal ash contained 9.2% more CaO than hard coal ash. Total content of heavy metals in granulates N41/m and N42/m is presented in Table 2.

Table 2. The total content of heavy metals [mg/kg d.m.] as confronted with allowed content as per Regulation on municipal sewage waste [16] and Regulation on the enforcement of the specific regulations of fertilizers and fertilization act [15]

Metal	Sewage sludge	Brown fly ash	Hard fly ash	N41/m	N42/m	Maximum permissible content for sludge used for agriculture and for agricultural reclamation [16]	Maximum permissible content for organic and mineral fertilizers [15]
Cu	155.7	96	78	92.0	82.0	800	400
Zn	3569	125	208	1740	1748	2500	1500
Cd	3.09	< 2	< 2	2.2	2.2	10	3
Ni	20.7	48	107	20.2	23.1	100	30
Pb	187.1	113	151	138.8	127.6	500	100
Cr	36.9	126	163	42.4	57.2	500	100
Hg	1.99	n.d.	n.d.	< 2	< 2	5	2

n.d. – not determined

RESULTS

Analysis of the findings from marking total content of heavy metals in sludge-ash granulates (Tab. 2) concludes that granulates prepared basing on hard coal ash contain more chromium and little more nickel as compared with granulates prepared basing on brown coal ash, the content of zinc and mercury in both specimens is comparable, and the content of cadmium is at the same level. However the content of copper and lead is higher in granulates prepared basing on brown coal ash.

The content of all metals subjected to analysis does not exceed allowed content of metals for sewage waste utilized in agriculture and land reclamation for agricultural purposes. When, however, the overall contents of metals are referred to quantities of metals allowed for organic and mineral fertilizers it can be observed that contents of zinc and lead are higher in two granulate specimens. Therefore, when considering utilization of granulates in agriculture it proves that it is possible from the viewpoint of Regulation on

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sewage waste [16] and impossible from the viewpoint of Regulation on the enforcement of the specific regulations of fertilizers and fertilization act [15].

In addition to the content of heavy metals which decide on their natural utilization it is also important in what quantities and in what rate they are to be released to soil. The most "mobile", i.e. those which fastest get to soil solution, and then get adsorbed by plants, are water-soluble forms of heavy metals, so-called "interchangeable" forms [22]. Table 3 contains the findings of research on elution of metals from granulates in one-stage test and in subsequent stages of three-stage test, as well as allowed contents of metals which can be released in the sewage stream to soil and water, as per Regulation [17].

Table 3. Concentration [mg/dm^3] of heavy metals eluted from granulates from the single-stage and three-stage elution test as confronted with allowed content as per Regulation [17]

Metal	N41/m				N42/m				Maximum permissible for treated wastes [17]		
	1-st	1(3)	2(3)	3(3)	1-st	1(3)	2(3)	3(3)	municipal	industrial	
										biodegradable	others
pH	8.25	8.3	8.04	8.02	8.46	8.49	8.4	8.1	6.5–9	6.5–9	6.5–9
Cu	0.08	0.115	0.035	0.02	0.09	0.125	0.04	0.02	n.c.	0.5	0.5
Zn	0.23	0.455	0.305	0.16	0.455	0.34	0.45	0.17	n.c.	2	2
Cd	0.005	0.005	0.005	0.005	0.005	0.005	0.005	0.005	n.c.	0.2	0.2
Ni	0.035	0.05	0.03	0.035	0.055	0.07	0.02	0.04	n.c.	n.c.	0.5
Pb	0.045	0.145	0.035	0.075	0.055	0.14	0.02	0.045	n.c.	n.c.	0.5
Cr	0.045	0.06	0.02	0.02	0.04	0.05	0.02	0.02	n.c.	n.c.	1

n.c. – not concern

Comparison of heavy metals concentrations prepared during tests of elution and allowed concentrations as per regulation [17] proves that concentrations of heavy metals eluted from sludge-ash granulates are lower than concentrations of metals provided for municipal sewage and the most restrictive concentrations for industrial sewage other than biodegradable. The lowest concentrations of metals compared with the said allowed concentrations were noted for cadmium (trace) and chromium (16 times slower as compared to the highest marked content). Concentrations of copper, zinc and lead were 4 times lower (as compared to the highest marked concentration) than allowed.

Degree of threat to environment due to elution of heavy metals from granulates is determined by quantity of metals (presented as loads) released during elution process. Quantities of individual metals in subsequent water extracts from granulates shall depend on mobility of their water-soluble forms as well as on the contact time and contact area of granulate and water. Contact area increases in subsequent stages of elution due to friction and soaking of granulate.

Loads of eluted heavy metals are very low compared to their content in granulate dry mass (Tab. 4). Sequence of metals elution in a three-stage test, as the sum of loads, is

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as follows: Zn > Pb > Cu > Ni > Cr > Cd. Calculated loads of individual eluted metals, with exception of chromium, correlate with their content in dry mass of granulate. In dry mass of granulate chromium content is higher than nickel content, and the sum of eluted charges of chromium is lower than of nickel. It means that in test conditions soluble forms of nickel outnumber soluble forms of chromium.

Table 4. Loads of heavy metals eluted from granulates from the single-stage and three-stage elution tests [mg/kg]

Metal	N41/m					N42/m				
	1-st	1(3)	2(3)	3(3)	1(3)–3(3)	1-st	1(3)	2(3)	3(3)	1(3)–3(3)
Cu	0.8	1.15	0.35	0.2	1.7	0.9	1.25	0.39	0.2	1.84
Zn	2.3	4.6	3.06	1.6	9.26	4.6	3.4	4.43	1.7	9.53
Cd	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
Ni	0.4	0.50	0.30	0.35	1.15	0.6	0.7	0.2	0.4	1.3
Pb	0.5	1.45	0.35	0.75	2.55	0.6	1.4	0.2	0.45	2.05
Cr	0.5	0.60	< 0.2	< 0.2	< 1.0	0.4	0.5	< 0.2	0.2	< 0.9

Granulate specimens in one-stage test were shaken 2 hours shorter than in the case of individual stages of three-stage test, and therefore eluted charges of heavy metals are little lower than those eluted during first stage of three-stage test. In one-stage test charges of metals eluted from granulates containing hard coal ash are little higher (or the same for chromium) as compared with charges of metals eluted from granulates contain brown coal ash.

Differences in summaric quantity of eluted charges (three-stage test) of individual metals from granulates with both types of ash are marginal. Summaric elution of copper, zinc and nickel from granulates containing hard coal ash is higher and of lead lower as compared with granulates containing brown coal ash. Elution of cadmium and chromium from both granulates, except first stage, is in trace quantities. Release rate for individual metals can be traced basing on the charges eluted in subsequent stages of elution (in per cent) as compared with their total content in granulates.

In Table 5 percentage elution of copper, zinc, nickel and lead charges in one-stage test and individual stages of three-stage test from granulates N41/m and N42/m (a:d = 7:3) is presented. Copper is eluted faster from granulate N42/m (after stage one 1.52%, after stage two 2.0%, after stage three 2.24%) as compared with granulate N41/m (after stage one 1.25%, after stage two 1.63%, after stage three 1.85%). Zinc is eluted faster from granulate N41/m after stage one (0.26%) and in the same rate from both granulates after stage two (0.44%) and three (0.53%). Nickel is eluted faster from granulate N42/m after stage one (3.03%) and in the similar rate from both granulates after stage two (c.a. 3.90%) and three (c.a. 5.70%). Initially lead is eluted faster from granulates N42/m (1.10%) as compared with granulates N41/m (1.04%), but as early as after stage two the same percent loads is eluted from both granulates and after stage three higher from granulate N41/m (1.83%). Mobility of water-soluble forms of metals after all stages of elution can be positioned in decreasing sequence: Ni > Cu > Pb > Zn for granulates (Tab. 5).

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Table 5. Percentage of eluted loads of Cu, Zn, Ni and Pb from granulates N41/m and N42/m from one-stage and three-stage elution tests

Metal	N41/m				N42/m			
	1-st	1(3)	1(3)–2(3)	1(3)–3(3)	1-st	1(3)	1(3)–2(3)	1(3)–3(3)
Cu	0.87	1.25	1.63	1.85	1.09	1.52	2.00	2.24
Zn	0.13	0.26	0.44	0.53	0.26	0.19	0.44	0.54
Ni	2.00	2.48	3.97	5.70	2.60	3.03	3.90	5.63
Pb	0.36	1.04	1.29	1.83	0.47	1.10	1.26	1.61

CONCLUSIONS

The conducted investigation provided the basis for formulating the following conclusions:

1. Heavy metal components of sludge-ash granulates elute in various quantities, i.e. from trace for cadmium to 9.26–9.53 mg/kg of dry mass for zinc, and charges of metals eluted from granulates containing brown coal ash are little lower than charges of metals eluted from granulates containing hard coal ash, with exception of lead. Concentration of all heavy metals eluted from sludge-ash granulates are lower than concentrations of metals which can be released in the sewage stream to soil and water provided for sewage.
2. From among the soluble forms of metals the most mobile are (in decreasing sequence): Ni > Cu > Pb > Zn in both granulates.
3. From granulate N42/m more Cu, Ni, Pb is eluted after stage 1, more Cu after stage 2 and less Ni, Pb after stage 3 in comparison to granulate N41/m.
4. Sewage-ash granulates with analyzed fly-ash added to sewage sludge weight ratio 7:3 can be used in environment as fertilizers, but to meet requirements for organic and mineral fertilizers sewage sludge used in their production is to contain smaller quantities of zinc and lead.

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WYMYWANIE METALI CIĘŻKICH Z GRANULATÓW WYTWORZONYCH NA BAZIE KOMUNALNYCH OSADÓW ŚCIEKOWYCH ORAZ POPIOŁÓW LOTNYCH Z WĘGLA KAMIENNEGO I BRUNATNEGO W ASPEKCIE ICH NAWOZOWEGO ODZYSKU

Wykorzystywanie odpadów w celach nawozowych wiąże się z wprowadzaniem do środowiska glebowego poza niezbędnymi do wzrostu i rozwoju roślin biogenami, również innych zawartych w nich składników m.in. metali (w tym ciężkich). Z uwagi na toksyczne oddziaływanie metali ciężkich na rośliny (w przypadku nadmiernego stężenia) istotna jest wiedza na temat ilości i tempa uwalniania poszczególnych metali z takich odpadów. W pracy przedstawiono wyniki badań wymywania Cu, Zn, Ni, Cd, Pb i Cr z granulatów przygotowanych w oparciu o komunalne osady ściekowe i popioły lotne ze spalania węgla kamiennego i brunatnego. Wymywanie do roztworu wodnego prowadzono w układzie statycznym: jednoetapowo i trójetapowo. Metale ciężkie były wymywane w zróżnicowanych ilościach tj. od śladowych dla kadmu do 9,5 mg/kg s.m dla cynku. Spośród rozpuszczalnych form metali najbardziej mobilnymi były, w sekwencji malejącej, Ni > Cu > Pb > Zn zarówno dla granulatów zawierających popiół z węgla brunatnego jak i kamiennego.