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The comparative research of leaching tests and the sequential chemical extraction based on the environmental risk assessment by a selected type of waste

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ABSTRACT

The problem of the circulation of metal ions in the environment remains a current problem in view of the quality of received agricultural crops. The necessity of increasingly frequent use of alternative sources of biogenesis in the form of waste substances poses a threat of bringing significant amounts of heavy metals harmful to human health and life to soils. The article discusses a significant problem, which is the comparison of the results of the environmental impact assessment of waste obtained based on the implementation of legitimate leaching tests (the three-stage leachability test according to PN-Z-15009) with the results obtained from sequential chemical extraction tests according to the Tessier method modified by Kersten and Forstner. The research comprises granulates prepared on the basis of fly-ash from brown coal combustion and the municipal sewage sludge in which Cu, Zn, Cd, Ni, Pb, Cr were determined. The studies confirm that the leaching tests used for application in the natural environment indicate at most such concentrations as those obtained at the first or second stage of sequential chemical extraction. Consequently, it has not been recognized their proper full impact on the environment.

Keywords: Sequential chemical extraction; Heavy metals; Leaching tests; Waste

1. Introduction

Economic development and continuous rise in standards of living cause the emergence of a large amount of waste resulting in negative effects on the human environment. Both international policy and actions taken by countries in their own territories are increasingly focused on the development and implementation of such mechanisms that will help reduce the load of contaminants that degrade the environment [1]. The Polish power industry, based mainly on burning coal, generates millions of tons of ashes every year. Fly ash is a by-product of combustion of coal. It is a ferro-aluminosilicate mineral containing most of the essential elements eg. calcium, potassium and magnesium (but not nitrogen) for the growth of the plant [2]. This phenomenon is present in many countries all over the world. Nowadays, there are many opportunities for their development, and yet innovative solutions are still being sought in order to extend the scope of their environmentally-safe recycling options [3].

Another problem related to the progressive civilization development is sewage sludge produced from the treatment of municipal sewage (as well as industrial), the amount of which increases every year parallel to a number of new plants. Methods of treatment and recovery of municipal sewage sludge must be developed individually for each treatment plant, taking into account the characteristics of

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the generated sludge, but sewage sludge is always a difficulty waste for utilization [4]. Future legislation is expected to limit the methods available to the producers for the disposal of the sludge. The organic load in sewage sludge should make it an excellent growth medium but its wide usage is limited by legislation.

With regard to the above facts, a serious problem is emerging, the solution of which seems to be one of the priorities in the field of environmental policy. The excessive amount of the aforementioned waste motivates to creating legal regulations and solutions in the field of safe waste management, including their treatment [5].

Sewage sludge stabilized with fly ash were used for an artificial soil creation. High water retention and minimization of soil erosion potential were found as positive effects which could introduce this material to farming, but chemical properties were not suitable for plants. As the main problem appears high mobility of heavy metals which are present in sewage sludge and fly ashes (even in presence of lime in artificial soil). The environmental conditions, as well as the physical and chemical properties of the coal fly ash, have a great impact on the mobility of coal fly ash constituents [6]. Usually, exchangeable forms are detected by extraction with DTPA or sequential extraction, but also with using distilled water with pH close to 7.00, high heavy metals amounts were eluted from an artificial soil [2,7]. In the past, a blending of commercial compost with small amounts of fly ash was suggested as a way for the improvement of the availability of essential and nonessential plant food elements (eg. Ca and Mg) with no negative effects on crop quality due to heavy metals content [8]. An artificial fertilizer used for tests in this study was made of sewage sludge and fly ash from brown coal combustion, as an answer for searching possibilities for high amounts of industrial waste re-use.

The article discusses a significant problem, which is the comparison of the results of the environmental impact assessment of waste obtained based on the implementation of legitimate leaching tests (the three-stage leachability test according to PN-Z-15009) with the results obtained from sequential chemical extraction tests according to the Tessier method modified by Kersten and Forstner.

2. Materials and methods

The research comprises fertilizing granulates of sludge-ash mixtures for the preparation of which were used fermented and dehydrated municipal sewage sludge and fly-ash from brown coal combustion (70% of sewage sludge and 30% of fly-ash). In order to check the movements of contaminants contained in the produced ash-sludge granulates, expressed in their susceptibility to leaching in water, the three-step extraction test was performed according to PN-Z-15009 standard. Its total duration of the process is 78 h, including 24 h of intensive shaking and 54 h of resting. The ratio of ash-sludge mixtures to distilled water was 1:10. For experiment was chosen granules 4–12 mm long. Prior to use, granules were dried in 80°C for 24 h and sieved through 2 mm sieve for dust and small particles removal. Loads of leached contaminants were also calculated from each leaching stage ($q1$, $q2$, $q3$) and their amount (Q), based on the formulas included in PN-Z-15009. The results of the research were referred to the Regulation of the Minister of the Environment of November 18, 2014. Regarding the conditions to be met when introducing sewage into waters or into the ground (Journal of Laws 2014, item 1800). Furthermore, the qualitative combinations of six microelements (Cu, Zn, Cd, Ni, Pb, Cr) were determined, based on the sequential chemical extraction according to the Tessier method [9] modified by Kersten and Forstner separated into six fractions:

- fraction I – exchangeable metals (metals easily accessible for the environment),
- fraction II – metals associated with carbonates,
- fraction III – metals associated with manganese oxides,
- fraction IV – metals related to amorphous iron oxides,
- fraction V – metals associated with organic matter and sulphides,
- fraction VI – metals associated with minerals (residual fraction),
  and the total content was calculated as the number of loads of fractions I–VI.

The procedure for determining individual fractions:

- fraction I – 20 cm^3 of the 1 M CH_3COONH_4 solution was added to the weighted portions and shaken at the room temperature for 2 h, and then the samples were centrifuged,
- fraction II – 20 cm^3 of 1 M sodium acetate are added to the residue from the centrifugation of the fraction I and shaken for 5 h at the room temperature and then centrifuged,
- fraction III – 20 cm^3 of 0.01 M hydroxylamine hydrochloride was added to the residue from the centrifugation of the fraction II and shaken for 12 h at room temperature, then centrifuged,
- fraction IV – 20 cm^3 of 0.1 M oxalate buffer was added to the residue from the centrifugation of the fraction III and shaken for 24 h at room temperature, and then centrifuged,
- fraction V – hydrogen peroxide with nitric acid (up to pH 2) was added to the residue from centrifugation of fraction IV and heated until the end of a turbulent reaction, cooled, 20 cm^3 of 1 M ammonium acetate in 6% HNO_3 was added and the mixture was shaken for 12 h and then centrifuged,
- fraction VI – concentrated acid is added to the residue from the centrifugation of fraction V nitrogenous, heated in a sand bath until the appearance of silica.

The concentration of metals in all the obtained extracts was determined by means of atomic absorption spectrometry on the Thermo-scientific atomic absorption spectrometer (ICE 3500).

3. Results and discussion

3.1. Content of selected trace elements – leaching tests

The results of the physicochemical indices are presented in Table 1 and compared with the limit values, included in the Regulation of the Minister of Environment of November

Table 1
Comparison of analytical research of water extracts in the three-stage leaching test from sludge ash granulates in mg/L with the values included in the Regulation of the Minister of the Environment of November 18, 2014 (Journal of Laws 2014, item 1800)

Parameter	1° (first step leaching)	2° (second step leaching)	3° (third step leaching)	Minister of the environment limit values from 18.11.2014 (JoL 2014, pos. 1800)
pH	6.50	6.87	7.16	6.5–9
EC μS/cm	4,500	1,340	430	2,500
Cu mg/L	0.795	0.490	0.890	0.5
Zn mg/L	10.65	9.85	17.5	2
Cd mg/L	0.010	0.010	0.010	0.05
Ni mg/L	0.465	0.200	0.130	0.5
Pb mg/L	0.250	0.300	0.650	0.5
Cr mg/L	0.145	0.090	0.240	0.1

18, 2014, on the conditions to be met when introducing sewage into the waters or into the ground.

The reaction of the obtained solutions (Table 1) was approx. neutral, and the pH increased with the leaching ratio from 6.50 (eluate from 1° leaching), through 6.87 (eluate from 2° leaching) to 7.16 (eluate from 3° leaching). However, none of the values obtained exceeded the limit value (6.5–9) specified in the Regulation of the Minister of the Environment of November 18, 2014, and was determined by other authors, but reaction strongly depends from granules stability during leaching test (Journal of Laws 2014, item 1800) [5].

The highest electrical conductance was determined in the elution from 1° eluate (4,500 μS/cm), which approx. 2 times exceeded the limit value (2,500 μS/cm), however, in in the eluate with 2° (1,340 μS/cm) and 3° elution (430 μS/cm) was considerably lower. A worrying phenomenon is the strong salinity of the solution (toxic for most plants), particularly pronounced in the case of the 1° leachate. In fact of the good stability of granules so high salinity was an effect of mezopores which were filled with water during leaching test. It is connected with high leaching mobile and semi-mobile fractions of metals even including metals associated with soluble organic matter. Although small particles of granules in 3° of leaching were observed, no increase of salinity was noted. This show that all easy soluble salts were removed in the first two stages of the experiment via mezopores and granules surface [10].

Comparing metal concentrations in eluates (Table 1) with the limit values of the above regulation it can be stated that only Cd and Ni meet its requirements. Especially Pb elution increased in subsequent stages of leaching (from 0.250 mg/dm^3, through 0.300 mg/dm^3 to 0.650 mg/dm^3), thus reaching in 3° a value higher than the admissible (0.5 mg/dm^3) by the regulation. Due to low leachability of lead from fly ash (silicates-bound complexes) and increasing concentration show its sewage sludge origin [11]. Leaching of other metals (Cu, Zn, Cr) was irregular. Copper and zinc in eluates from each level of leaching exceeded the admissible values, including the highest – 8 times exceedance in 3° elution for Zn (17.50 mg/dm^3) and about 2 times for Cu (0.890 mg/dm^3), a Cr in the eluate with 1° (0.145 mg/dm^3) and 3° (0.240 mg/dm^3) elution. In this stage, bioleaching processes could be present, due to good EC and pH values. Comparable results due to water elution of Cu and Cr were obtained in a pot experiment with artificial soil made of sewage sludge and fly ash [2]. Column leaching tests made on fly ash only, confirm Zn removal but also Ni, but not Cu. It shows a great role of sewage sludge presence as in Cu donor [12]. A long time of the procedure (72 h) is a good possibility for fungi and bacterial colonies growth [13]. It is very important to take into account that the main source of heavy metals for sewage sludge is wastewater and its origin [14].

The concentrations of the studied elements were used to calculate the leached loads from each leaching stage ($q1$, $q2$, $q3$) and their sum (Q) based on the formulas given in PN-Z-15009 norm, which are presented in Table 2. Due to very low numerical values they were presented in mg/kg DM, not in g/kg DM as recommended by PN.

During the three-stage leachability test for ash-sludge mixtures in the case of Ni, these loads definitely decrease in subsequent steps of leaching, Pb increase, and Cd are the same. In other metals such as Cu, Zn and Cr, the variability of leaching loads was observed in the subsequent stages of leaching. The largest total load was calculated for zinc (305.8 mg/kg DM), more than seventeen times smaller (compared to Zn) for copper (17.50 mg/kg DM), and total loads of other microelements do not exceed 10 mg/kg DM (in the descending sequence: Pb – 9.64 mg/kg DM, Ni – 6.42 mg/kg DM, Cr – 3.82 mg/kg DM., Cd – 0.25 mg/kg DM). Obtained data was confirmed by other authors, but in some cases amount of zinc was lower. It is strongly connected to ash origin [15]. Except heavy metals concentrations, high

Table 2
Loads of determining indicators (mg/kg DM) in eluates from ash-sludge granulates

Indicator	$q1$	$q2$	$q3$	Q
	mg/kg DM			
Cu	6.42	3.96	7.12	17.50
Zn	86.1	79.7	140	305.8
Cd	0.081	0.081	0.081	0.25
Ni	3.76	1.62	1.04	6.42
Pb	2.02	2.42	5.20	9.64
Cr	1.17	0.73	1.92	3.82

salinity of obtained leachate suggests that open dumping should be restricted to reduce the potential for groundwater contamination [12].

3.2. Qualitative content of trace elements, based on sequential chemical extraction according to the Tessier methodology modified by Kersten and Forstner

In order to determine the proper environmental impact of ash-sludge granulates, they were also subjected to the analysis of sequential chemical extraction according to the Tessier method is modified by Kersten and Forstner (Table 3). The highest number of exchangeable connections (fraction I) was associated with Zn (42.02 mg/kg DM), considerably lower Cu (3.55 mg/kg DM), Ni (2.03 mg/kg DM), Cr (0.62 mg/kg DM), and Cd and Pb below the limit of quantification of the method. In combinations with carbonates (fraction II) Zn (5.96 mg/kg DM) was also the most determined, slightly less Cu (5.05 mg/kg DM) and the least Ni (1.12 mg/kg DM). In combinations with manganese oxides (fraction III), Zn (1,738.1 mg/kg DM) was re-determined the most, considerably less Cu (30.62 mg/kg DM), Pb (24.76 mg/kg DM), Cr (15), 29 mg/kg DM, the least Ni (9.96 mg/kg DM) and Cd (1.29 mg/kg DM). In the connections with amorphous iron oxides (fraction IV) Zn (238.6 mg/kg DM) was also the most determined, significantly less Pb (79.53 mg/kg DM), Cu (30.62 mg/kg DM), Cr (21.08 mg/kg DM), and the least Ni (4.12 mg/kg DM). In the connections with organic matter and sulphides (fraction V) Zn (25.79 mg/kg DM) were the most determined, slightly less Pb (22.11 mg/kg DM), Cu (17.96 mg/kg DM), Cr (11.38 mg/kg DM), and the least Ni (4.88 mg/kg DM), as in earlier fractions. In the connections with silicates (fraction VI) the highest was determined Pb (202.3 m/kg DM), much less Cr (62.91 mg/kg DM) and consecutively: Ni (44.63 mg/kg DM), Zn (28.93 mg/kg DM), Cd (12.95 mg/kg DM) and Cu (9.43 mg/kg DM). In sewage, sludge metals are present in different forms, in fly ash – mostly in oxides. The total concentration of toxic metals can be high and exceed 37 mg/kg Cd and 150 mg/kg Pb. [16].

Summarizing the results obtained after the sequential extraction, it can be concluded that in all joints determined as I to V fractions Zn predominates (in the descending sequence: III – 1,738.1 mg/kg DM, IV – 238.6 mg/kg DM, I – 42.02 mg/kg DM, V – 25.79 mg/kg DM, II – 5.96 mg/kg DM), and only in fraction VI, the highest Pb (202.3 mg/kg DM) concentration was determined.

The total content (Table 3) of trace elements (as the sum of loads of fractions I–VI) obtained as a result of the analysis of sequential chemical extraction (using solutions of chemical compounds with varying leaching strength) was significantly higher compared to the sum of leached loads obtained by water extraction (during the three-stage leaching test) [15].

The highest total content (as the sum of loads of fractions I–VI – Table 3) was obtained for Zn (2,079.4 mg/kg DM), more than six-fold lower for Pb (328.7 mg/kg DM), significantly lower for Cr (111.3 mg/kg DM), Cu (97.23 mg/kg DM), Ni (66.74 mg/kg DM), and Cd (14.24 mg/kg DM). Especially poor leaching of Cd was reported even in the use of strongly acidic solutions [11]. As reported other authors leachability similar to zinc and copper has also manganese [17]. The problem of long-term leaching (eg. after granulated waste as "fertilizer" were used) was described. Total eluted by rainwater cadmium exceed 20% (1.76% in this study) of total content but in the case of zinc – only 0.2% (14.7% in this study). Differences in case of Zn can be explained by a high content of sewage sludge in granules were not only oxides but also soluble zinc salts – due to a high share of fractions I–III are present [16].

A high share of unavailable fractions can be an effect of CaO (brown coal ash) addition, what change metals contain in sewage sludge solubility [18]. Analyzing the percentage of determined metals in compounds according to the Tessier method in the modification of Kersten and Forstner (Table 4), it is stated that:

- in the interchangeable compounds (fraction I) Cu (3.65%) was predominantly associated, followed by Ni (3.04%), Zn (2.02%), Cr (0.56%),
- with carbonates (fraction II), also the most predominantly associated was Cu (5.19%), successively: Ni (1.68%), Zn (0.29%),
- with manganese oxides (fraction III), mainly associated was Zn (83.59%), followed by Cu (31.49%), Ni (14.92%), Cr (13.74%), Cd (9.06%)) and Pb (7.53%),

Table 3
Comparison of the results of sequential chemical extraction according to the Tessier method modified by Kersten and Forstner and the sum of loads of trace metals from the three-stage leaching test of the ash sludge granulates

Sequential chemical extraction according to the Tessier method modified by Kersten and Forstner								Elution test
Metal	Fraction I	Fraction II	Fraction III	Fraction IV	Fraction V	Fraction VI	Sum of loads fractions I–VI	Sum of eluted loads Q
	mg/kg DM	mg/kg DM	mg/kg DM	mg/kg DM	mg/kg DM	mg/kg DM	mg/kg DM	mg/kg DM
Cu	3.55	5.05	30.62	30.62	17.96	9.43	97.23	17.50
Zn	42.02	5.96	1,738.1	238.6	25.79	28.93	2,079.4	305.8
Cd	0	0	1.29	0	0	12.95	14.24	0.25
Ni	2.03	1.12	9.96	4.12	4.88	44.63	66.74	6.42
Pb	0	0	24.76	79.53	22.11	202.3	328.7	9.64
Cr	0.62	0	15.29	21.08	11.38	62.91	111.28	3.82

Table 4
Percentage of metals in fractions according to Tessier sequential chemical extraction in Kersten and Forstner modification in ash sludge granulates

Sequential chemical extraction according to the Tessier method modified by Kersten and Forstner							Sum of fractions	
Metal	Fraction I	Fraction II	Fraction III	Fraction IV	Fraction V	Fraction VI	I–III	IV–V
	%	%	%	%	%	%	%	%
Cu	3.65	5.19	31.49	31.49	18.47	9.70	40.33	49.96
Zn	2.02	0.29	83.59	11.47	1.24	1.39	85.90	12.71
Cd	0.00	0.00	9.06	0.00	0.00	90.94	9.06	0
Ni	3.04	1.68	14.92	6.17	7.31	66.87	19.64	13.48
Pb	0.00	0.00	7.53	24.20	6.73	61.55	7.53	30.93
Cr	0.56	0.00	13.74	18.94	10.23	56.53	14.30	29.17

- with amorphous iron oxides (fraction IV), Cu (31.49%) was mainly associated, and in a lesser degree: Pb (24.20%), Cr (18.94%), Zn (11.47%), Ni (6%), 17%),
- with organic matter and sulfides (fraction V) also mainly associated was Cu (18.47%) is, to a much lesser extent Cr (10.23%), Ni (7.31%), Pb (6.73%), Zn (1.24%),
- with a mineral residue (fraction VI) mainly associated was Cd (90.95%), respectively: Ni (66.87%), Pb (61.55%), Cr (56.53%), Cu (9.70%), Zn (1.39%).

Availability of Cu and Zn was also observed during fly ash Tessier sequential extraction, what should be the first step in tests of groundwater and soil protection [19]. Summarizing the results of the ash-sludge granulate assays, it can be noted that: Zn and Cu are mainly associated with manganese oxides (in fraction III), while Pb, Cr, Ni, Cd and silicates (fraction VI – unavailable for ecosystems), which definitely limits their negative impact on the environment [3].

Investigated material can be used in agriculture, but in first days a lot of easy soluble compounds are released, however, a deacidification effect was relatively low. Comparable results (high EC in first days), were obtained in long time experiment with ash-coffee waste fertilizer [13]. This phenomenon can be toxic for young plants and especially be as a source of groundwater pollution with macro and micronutrients [12,16].

Mass of produced sewage sludge in 2018 in Poland was high: 1,046.5 thow. tons of dry mass, but the stored mass is much higher – over 6,220 tons of dry mass. Mass of produced lignite ashes is also very high – about 8 mln tons per year. Use of lime fertilizers in agriculture was 0.77 mln tons per year, so ashes with calcium content up to 20% can be a real alternative of typical lime fertilizers (stat.gov.pl).

4. Conclusions

Comparing the results obtained from the study of ash sludge granulates performed as a leachability test and sequential chemical extraction (according to the Tessier method in the modification of Kersten and Forstner), in terms of environmental impact, it can be concluded that:

- a/According to the leachability tests a/the reaction in the eluates obtained from the 3° leachability test meets the requirements of the regulation quoted above, while the value of the specific conductance from just 1° of leaching does not allow for its introduction into the groundwater environment,
- b/only concentrations of Cd and Ni (from the following three stages of leaching) are lower than those specified in the regulation, while the concentration of Pb (in the eluate from the 3° leachability), Cr (in the eluate from 1° and 3°), Cu and Zn (in eluates from each stage) are higher than the limited value.

According to the analysis of sequential chemical extraction, which allows for the assessment of the proper impact of waste on the environment, it has been demonstrated that Cu associated in the compounds determined as fractions I–III – easily accessible to the environment constitutes about 40% of the total amount of fractions I–VI, whereas in compounds determined as fractions IV and V (only under specific conditions of the soil environment can be released to the environment) it constitutes about 50%. Toxic compounds (Cd, Ni, Pb and Cr) are metals mainly associated to compounds inaccessible to the environment (fraction VI), and they constitute from 56.53% (Cr) to 90.94% (Cd) from the amounts of loads of fractions I–VI. The amounts of loads (mg/kg DM) of elements determined in the eluates from the leachability test is often lower than the amount leached in fractions I to III (sequential extraction) referred to as the easiest available to the environment.

The research confirms that the leaching tests, required by the regulation, are used with waste before their application to the environment, indicate only the content which we obtain in the first three stages of sequential chemical extraction.

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