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BOTTOM ASH FROM MUNICIPAL SOLID WASTE INCINERATION. BASIC PARAMETERS AND ECOTOXICOLOGICAL PROPERTIES

The bottom ash from municipal solid waste incineration is the most important by-product, in terms of energy recovery from municipal solid waste. Safe treatment and reuse of this bottom ash in construction materials is one of the ways of its effective use. The specific use precedes the study of the properties of the bottom ash from municipal solid waste incineration. In this study, samples of bottom ash were examined via sieve analysis, basic chemical parameters, ecotoxicological property analysis and determination of heavy metals (As, Cd, Cr, Cu, Pb, Ni, Zn). The sieve analyses of samples showed different contents of individual fractions. Ecotoxicological tests for acute toxicity on *Daphnia magna* in the raw aqueous extract showed positive results mortality of all individuals after 24 hours. The toxic effect of bottom ash was confirmed by the content of heavy metals.

1. INTRODUCTION

Production of municipal waste is currently increasing worldwide. One sustainability strategy of the European Union (EU) is the introduction of a circular economy. Waste and secondary materials in a circular economy are reused or recycled. Reuse or recycling is ultimately the reduction of primary materials and potentially reduces the amount of material going to landfill. The revised 2008/98/EC on waste (*Waste Framework Directive*) sets the new approach to waste management which is based on three fundamental principles: waste prevention, recycling and reuse, as well as improving final disposal and monitoring [1–3].

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In 2016, the EU produced 243.860 million tons of municipal waste [4]. Energy recovery is an increasingly common solution for the processing of municipal solid waste. Incineration reduces the weight and volume of municipal solid waste by up to 65–90%, provides usable energy and reduces the amount of landfilled waste. An incineration plant is used for the thermal treatment of wastes with or without recovery of the heat of combustion [5–7].

Incineration of municipal solid waste produces two by-products: bottom ash and fly ash. The bottom ash is the most important by-product in terms of the quantity produced during the energy recovery of municipal solid waste. Environmentally friendly treatment and reuse of bottom ash from municipal solid waste incineration depend on its physicochemical composition and ecotoxicological properties.

Keulen et al. [2] examined the use of bottom ash from municipal solid waste incineration as an aggregate in the production of curbs. Their work showed that the release of chloride and antimony is always either just above or below the limit values in their country. The authors suggested improving wet separation or adding specific ingredients to reduce the impact on environmental quality.

Li et al. [6] used bottom ash from municipal solid waste which first passed through magnetic separation and then was ground to a grain size of 45 μm. Its addition to cement compositions increased the demand for water; 10, 20 and 30% cement replacement with bottom ash from municipal solid waste incineration satisfied the strength class 32.5 MP after 28 days. Li et al. found the content of heavy metals in the mixtures to be below the national determined standard and, furthermore, the concentration of heavy metals in the leachate to be lower than the relevant standard.

Sormunen and Rantsi [5] examined harmful substances leached from the bottom ashes produced from municipal solid waste incineration. The bottom ash was produced in a combustion grate at 1000 °C. The fraction >50 mm was removed from the bottom ash and then the material was passed through magnetic separation. Bottom ash was fractionated into 0–2 mm, 2–5 mm, 5–12 mm and 12–50 mm fractions. Leaching tests were performed on samples of each fraction according to EN 12457-3. The contents of hazardous substances (Sb, Cl, Pb, Ni, Zn) in most instances did not exceed the values described in Finnish legislation; however, in some instances, these values for antimony and chlorine were exceeded. Therefore, this material was considered unsuitable for use in residential buildings but could be used in road construction.

Tang et al. [8] dealt with a thermally activated bottom ash from municipal solid waste incineration. After magnetic separation and rotary separation, the bottom ash was crushed in a ball mill and thermally activated in a muffle furnace at 550–750 °C. The thermal activation removed dust that covered the ash which contained more impurities. After thermal activation, the bottom ash was milled and then separated into fractions below and above 63 μm. The prepared ash was then used as a partial replacement of cement. The material obtained was satisfactory in terms of strength and the leaching of heavy metals did not exceed limit values.

Römbke et al. [9] examined the bottom ashes from 12 various incineration MSW in terms of ecotoxicity. The purpose of the research was the evaluating of the hazard property H14 – ecotoxicity. The ecotoxicological potential of 12 bottom ashes was assessed by using biological test systems. The testing included aquatic tests with eluates (algae, daphnia and luminescent bacteria) and terrestic tests with solid waste (plants, earthworms and bacteria). The test results revealed that fresh ashes were several times more toxic than aged ashes.

In the research of Stiernström et al. [10] the ecotoxicological properties of a five-year-old bottom ash from the municipal solid waste incineration for potential utilization as filling material or for construction purposes was examined. The ecotoxicity of leachates from the bottom ash was evaluated in an environmental relevant way using a sequential batch leaching method at the liquid/solid (L/S) ratio interval 1–3, and to test the leachates in a (sub)chronic ecotoxicity test. Larval development test with *Nitocra spinipes* was used. The studied material had limited leakage of hazardous heavy metals and fast decaying leaching of soluble metals (K and Ca) at low L/S ratios. The observed toxicity on *Nitocra spinipes* in the (sub)chronic test was low and completely absent already at L/S 2. The findings of Stiernström et al. [10] indicate that aged bottom ash from municipal solid waste incineration may be used in geotechnical constructions (e.g., embankments and roads) without posing increased environmental risks.

In this study, we analyzed bottom ash samples from an experimental municipal solid waste incineration plant. Currently, the bottom ash from the municipal solid waste incineration is landfilled. Some authors [2, 5, 6, 8] demonstrated its potential use in construction, thereby reducing the number of primary resources used (aggregates, sand, cement). We focused on ecotoxicological properties and determination of heavy metals, which is essential information for utilization of this type of waste.

2. MATERIALS AND METHODS

The samples of bottom ashes used for the analyses originated from the test equipment in municipal solid waste incineration. It consisted of a grid mechanism and combustion with partial air access. The gasification reactor had a capacity of 500–1200 $kg \cdot h^{-1}$ of separated waste. Separated municipal waste was processed into refuse derived fuel (RDF). The fuel was fed in the gasification reactor. The bottom ash was transported into silo storage tanks by a grating device. The estimated bottom ash mass flow was 120 $kg \cdot h^{-1}$. The gasification zone consisted of a drying zone (temperature ca. 300 °C) and an oxidation zone (temperature ca. 800 °C). The ignition temperature was 300 °C. During the development of the gasification reactor, several test runs were carried out. In the test runs, the gasification process in the reactor occurred at temperatures above 1000 °C.

In samples of bottom ash, the fractions by size, basic chemical parameters (pH, loss of ignition (LOI), solid content), ecotoxicological properties and content of heavy metals (As, Cd, Cr, Cu, Pb, Ni, Zn) were determined. The results of measurements are the average of 6 repetitions with repeatability *r* lower than 5%.

Sampling. From each test run of the gasification reactor (two test runs) aggregated samples were sampling. Each aggregated sample (100 kg) consisted of 10 incremental samples taken from different places of bottom ash storage tanks. From the aggregated samples, 6 laboratory samples were taken by quartering, which was used to determine the selected indicators.

Bottom ash samples for the determination of basic chemical parameters, sieve analysis, ecotoxicological tests (in water leached) and heavy metal determination were adjusted by quartation and then homogenized by grinding (Mixer Mill MM 301, Retsch, Germany). The analysis process of the bottom ash samples from the test equipment in municipal solid waste incineration is shown in Fig. 1.

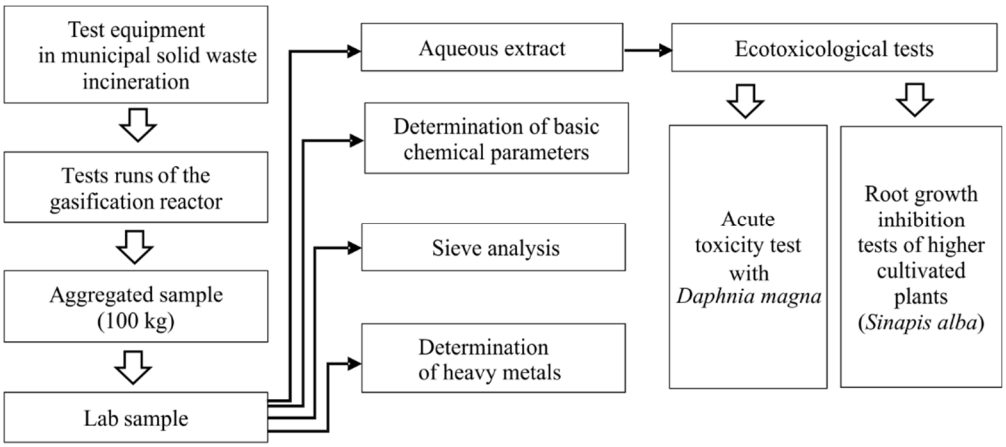

Fig. 1. The analysis process of the bottom ash samples from the test equipment in municipal solid waste incineration

Determination of basic chemical parameters. pH was determined by using a pH meter with a glass electrode. 20 g of solid waste was weighed and transferred into 100 cm^3 beaker. 40 cm^3 of distilled water was added and stirred well with a glass rod. This was allowed to stand for half an hour with intermittent stirring. The mixture was allowed to settle and pH was measured [11].

Determination of loss on ignition (LOI). Determination of loss on ignition (LOI) was performed by EN 15169 [12]. 5 g of dried sample in a crucible was heated in the furnace at 550±25 °C for at least 60 min. The hot crucible containing the residue on

ignition was placed in the desiccator and left to cool. The weighing was carried out immediately after removal of the crucible from the desiccator and the weighing operation was completed as quickly as possible. The mass of the residue on ignition and therefore the loss on ignition may be regarded constant if the mass obtained after a further half-hour period of ignition at 550 °C in the pre-heated furnace does not differ by more than 0.5% from the previous value or 2 mg, whichever is the greater.

The loss on ignition (W_V) of the dry mass of a solid sample as a percentage was calculated from the equation:

$$W_V = \frac{m_b - m_c}{m_b - m_a} \times 100\% \tag{1}$$

where m_a is the mass of the empty crucible, g, m_b is the mass of the crucible containing dry mass, g, m_c is the mass of the crucible containing the ignited dry mass, g.

The residue on ignition (W_R) of the dry mass of a solid sample was calculated from the equation

$$W_R = 100 - W_V \tag{2}$$

Determination of dry matter. Determination of solid content was performed according to EN 14346 [13]. Samples were dried to constant mass in an oven at 105±5 °C. The evaporating dish was placed in the drying oven set at 105±5 °C for a minimum of 30 min and then cooled to ambient temperature in a desiccator with the lid closed. After cooling, the dish was weighed to the nearest 1 mg.

The dry matter (W_{dm}) as a percentage difference in mass before and after the drying process was calculated according to the:

$$W_{dm} = \frac{m_e - m_d}{m_f - m_d} \times 100\% \tag{3}$$

where m_d is the mass of the empty dish, g, m_e is the mass of the dish containing the sample, g, m_f is the mass of the dish containing dry matter, g.

Determination of particle size distribution: sieving method. Determination of particle size was performed in line with EN 933-1 (72 1186) *Tests for geometrical properties of aggregates. Part 1. Determination of particle size distribution via the sieving method* [14]. The test consists of dividing and separating material into several particle size classifications of decreasing sizes by means of a series of sieves. Sieve analysis was performed on the vibratory sieve shaker AS 450 basic, Retsch, Germany. The test portion was dried at 110±5 °C to constant mass. After cooling, the sample was weighed and its

mass was labeled as M_1. The test sample was placed in a container during washing and a sufficient amount of water was added to cover the test sample. Subsequently, both sides of a 0.063 mm sieve reserved for use in this test only were wet and a guard sieve (e.g., 1 or 2 mm) was fit on top. Sample washing was continued until the water passing through the 0.063 mm test sieve was clear. The residue retained on the 0.063 mm sieve was dried at 110±5 °C to constant mass. The sample was cooled, weighed and the mass was labeled as M_2.

The washed and dried material (or the dry sample) was poured into the sieving column. The column comprised a number of sieves fitted together and arranged, from top to bottom, in order of decreasing aperture sizes with the pan and lid. Washed, dried and weighed samples were poured into the sieve column and the sieving operation was conducted for 10 min. The retained material was weighed for the consecutive sieves starting from that with largest aperture size, and their masses were labeled as R_1, R_2, ..., R_i, R_n. If there was any screened material remaining in the pan, it was weighed and its mass was labeled as P.

Then for the mass retained on each sieve, a percentage of the original dry mass M_1 was calculated. The percentage of the original dry material passing each sieve down to the 0.063 mm sieve was calculated according to the equation:

$$f = \frac{(M_1 - M_2) + P}{M_1} \times 100\% \tag{4}$$

where: M_1 is the dried mass of the test portion, kg, M_2 is the dried mass of the residue retained on the 0.063 mm sieve, kg, P is the mass of the screened material remaining in the pan, kg [14].

Total metals content. Determination of heavy metals content (As, Cd, Cr, Cu, Pb, Ni, Zn) was performed by CEN EN 16192 *Characterization of waste. Analysis of eluates* [15]. Mineralization of samples was performed by aqua regia and subsequently, the method of atomic absorption spectrometry AAS AVANTA PM PLUS SYSTEM 3000 (GBC) was applied. The mercury content was analysed by the direct determination in a solid sample by AMA 254.

Aqueous extract preparation. The volume of extraction solution was calculated according to STN EN 14735 [16]:

$$L = \frac{10 - M_C}{100} \times M_D \tag{5}$$

where L is the volume of the used extraction solution, dm^3, M_D is the weight of dried (determined by gravimetric drying to a constant weight at 105 °C) tested part, kg, and M_C is the moisture content, %.

Determination of ecotoxicity. Ecotoxicology tests were performed according to STN 83 8303 *Testing of dangerous properties of wastes. Ecotoxicity*. These include acute toxicity tests on aquatic organisms and growth inhibition tests of algae and higher cultivated plants [17].

Acute toxicity test Daphnia magna Straus. For the standard test, *Daphnia magna* was used, at least the third generation, obtained by acyclic parthenogenesis under specific reproduction conditions. An individual (*Daphnia magna Straus*) used in the test should be younger than 24 h. Pregnant females are transferred to containers with fresh water for 24 h and new born individuals are caught. In the preliminary test, twenty *Daphnia magna* were placed in undiluted aqueous extract. Concurrently, the same numbers of *Daphnia magna* were put in reconstituted water (Table 1) as a control sample. The reconstituted water was prepared from the solutions, by pipetting 10 cm^3 of each of solutions 1–4 up to the volume of 1 dm^3, and was used as a control.

Table 1

Stock solutions for the preparation of reconstituted water [17]

Stock solution	Chemical substance	Concentration [$g \cdot dm^{-3}$]
1	$CaCl_2 \cdot 2H_2O$	117.6
2	$MgSO_4 \cdot 7H_2O$	49.3
3	$NaHCO_3$	25.9
4	KCl	2.3

Immobilization and mortality were monitored at 24 and 48 h, respectively. The result of the preliminary test was considered negative if during the test death/immobilization of the *Daphnia magna* was <50% in that of the control sample. The result of the preliminary test was considered positive, if during the test death/immobilization of the *Daphnia magna* was ≥50% that in the control sample. Then it was necessary to determine EC_{50} (concentration that causes adverse effects in 50% of the test organisms for a binary effect such as mortality or a specified sublethal effect), values and make a preliminary and main test. It was necessary to carry out verification tests. Reference substance was $K_2Cr_2O_7$, $EC_{50,\ 48\ hours}$ = 0.81 $mg \cdot dm^{-3}$ (limit – 1.5 $mg \cdot dm^{-3}$) and validity of the test% of immobilised individuals in control was 5% (limit – 10%) [18–22].

The tests of growth inhibition of higher cultivated plant (Sinapis alba). In the test comparison was made between the seeds of white mustard (*Sinapis alba*) grown on a filter paper with an undiluted aqueous extract and seeds grown on a filter paper with reconstituted water.

In the preliminary test filter paper was placed in Petri dishes. Then 10 cm^3 of undiluted aqueous extract was added to the Petri dish and 30 seeds were regularly dispersed

on a rectangular grid of 5×6 cm. Three tests were conducted in parallel. The control sample was made similarly using 10 cm^3 of reconstituted water. The values measured after 72 h were used to calculate the arithmetic mean of the control and individual bottom ash samples. Percentage inhibition (stimulating) the growth of root of the higher culture plants was calculated according to the equation:

$$I_i = \frac{L_k - L_v}{L_k} \times 100 \tag{6}$$

where: L_v is the average length of the roots in the extract at the tested concentration, mm, L_k is the average length of the roots in the control sample, mm.

The preliminary test was negative if the inhibition of the root growth was <30% (respective stimulation below 75%) of that of the control. Further testing was not performed in this case. The preliminary test was positive if the inhibition of root growth was ≥30% (respective stimulation ≥75%) of that of the control.

If the inhibition of root growth was ≥30% and <50% (respective stimulation ≥75%) of that of the control, then the next test was not performed. If the inhibition was ≥50% of that of the control, then the basic test was performed and the half-maximal inhibitory concentration (IC_{50}) as a measure of the effectiveness of a substance in inhibiting a specific biological or biochemical function was determined. The validity of the test germination in the control sample was 97.5% (limit ≥90%), for the reference $K_2Cr_2O_7$, $IC_{50,\ 72\ h}$ was 30.50 mg·dm^{-3} (limit 4.1–85 mg·dm^{-3}). The root length was measured by steel calibrated instrument [19, 23, 24].

Statistical analysis. The software STATISTICA (Version 10, StatSoft Company, Tulsa, USA), ANOVA – Analysis of Variance was used to evaluate the results of ecotoxicological tests. The graphical presentation of the ANOVA results was performed with 95% confidence intervals for average values of immobilisation and inhibition for individual samples.

3. RESULTS AND DISCUSSION

The important parameter for use in construction is mainly carbon content (loss on ignition – LOI). This parameter is specified by the Directive 2000/76/EC *On the incineration of waste* [25]. The LOI was higher than 5% of the dry weight of the bottom ash (Table 2). The exceeded value of LOI was caused by the variability of the gasification process (change of reactor temperature during the incineration of 300–1000 °C). This resulted in different LOI values for samples. pH affects a number of other factors. Decreasing pH values can increase the solubility of amphoteric metals (Pb, Zn), thereby increasing their leachability.

Table 2

Basic chemical parameters of the bottom ash

Sample	pH measured	pH range[a]	LOI [d.w. %]	LOI limit[b] [d.w. %]	Solid content [wt. %]
1	9.24	9.5–12.0	11.34	5.00	96.55
2	9.60		7.69		97.87

[a]The pH range for bottom ash from thermal treatment of MSW.
[b]The LOI limit specified by the Directive 2000/76/EC.

Table 3

The results of the sieve analysis of the bottom ash samples

Sieve size [mm]	Weight balance [kg]		Proportion balance [wt. %]		Overall proportion overflow [wt. %]	
	Sample 1	Sample 2	Sample 1	Sample 2	Sample 1	Sample 2
8	0.00	0.00	0.0	0.0	100	100
5.6	0.18	0.12	28.2	18.0	72	82
4	0.04	0.03	5.8	3.7	66	78
2	0.04	0.03	7.0	4.0	59	74
1	0.05	0.03	8.5	4.3	51	70
0.5	0.05	0.03	8.3	5.0	42	65
0.25	0.07	0.07	10.9	9.7	31	55
0.125	0.07	0.20	10.7	29.3	21	26
0.063	0.12	0.16	19.2	23.6	1.4	2.5
Bottom	0.01	0.01	1.1	1.8	–	–
Percentage of fines passing the 0.063 mm sieve [wt. %]		Sample 1		1.4		
		Sample 2		2.5		

Fig. 2. Bottom ashes from municipal solid waste incineration:
a) 5.6 mm sieve, b) 0.125 mm sieve, c) 0.063 mm sieve

The results of the sieve analysis are shown in Table 3. The largest fraction of sample 1 was retained on the 5.6 mm sieve. The material contained large particles of unburned

material, which cannot be used (Fig. 2a). The largest fraction of material of sample 2 was retained on the 0.125 mm sieve (Fig. 2b) and another large proportion was retained on the 0.063 mm sieve (Fig. 2c). Material retained on the 0.125 mm and 0.063 mm sieves was completely burnt and can be used for analysis. Both samples contained residues under the 0.25 mm sieve which are further usable. Figures 3, 4 illustrate the grading curves of the samples of bottom ashes.

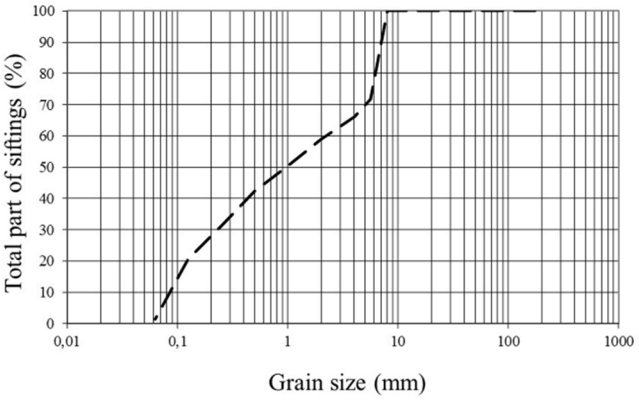

Fig. 3. Grading curve for sample 1

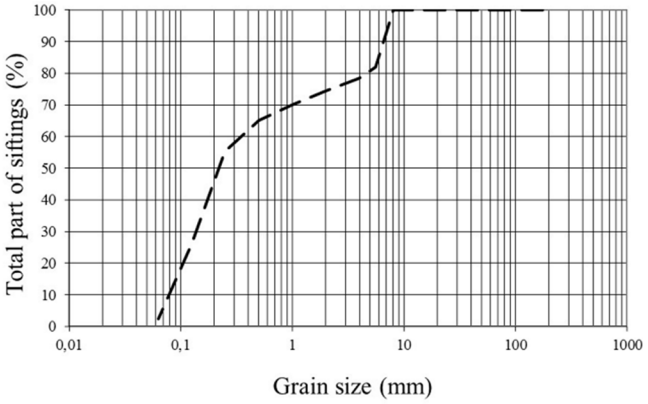

Fig. 4. Grading curve for sample 2

The preliminary test of acute toxicity on *Daphnia magna* and test of growth inhibition of higher cultivated plant (*Sinapis alba*) was used to assess the toxicity of the aqueous extract. This test served as a validation test. The results of the preliminary tests are shown in Tables 4, 5.

In the preliminary test of acute toxicity on daphnia, both sample 1 and sample 2 showed mortality after 24 hs (Table 4). During the test, the mortality of the daphnia was

>50% of that of the control. The test was therefore positive and consequently, verification, basic tests and determination of the EC_{50} should have been carried out.

Table 4

Results of the test of acute toxicity on *Dafnia magna*

Sample		No. of embedded organisms	Mortality after 24 h		Mortality after 48 h	
			No.	[%]	No.	[%]
Control	A	20	1	5	1	5
	B		0	0	1	5
	C		0	0	0	0
1	A		20	100	20	100
	B		20	100	20	100
	C		20	100	20	100
2	A		20	100	20	100
	B		20	100	20	100
	C		20	100	20	100

A, B, C – parallel samples.

Table 5

Basic statistical characteristics and results of the tests of growth inhibition in white mustard (*Sinapis alba*)

		Control	Sample 1	Sample 2
Length root, mm		5.40	4.23	0.57
Inhibition, %	average	–	21.74	89.41
	standard error	–	0.91	0.62
95% confidence intervals	lower limit	–	19.22	87.68
	upper limit	–	24.26	91.13
Number of repetitions		–	5	5
Evaluation		–	negative	positive

In the preliminary test of the white mustard seeds (*Sinapis alba*), sample 1 displayed inhibition below 30% (Fig. 5), therefore the test is negative and it is not necessary to conduct further testing. Sample 2 displayed inhibition higher than 50% compared with the control; therefore the test was positive.

The contents of metals were compared with the average values in samples of three MSW incineration of data published by the Nordic Council of Ministers (Table 6). The zinc content in both samples was higher than the compared value. Elevated copper content was found in sample 1. The contents of Cd, Cr, Pb and Ni in sample 2 was higher than that in sample 1. The increased concentration of these elements in both samples was sensitive for the aquatic test with daphnia. In the case of terrestric test on seeds of white mustard (*Sinapis alba*), only sample 2 was positive – inhibition of up to 89.41%.

Used bioassays are relevant combination of biological tests to assess the hazard property of H 14 (ecotoxicity) as shown by studies Pandrad et al. [26, 27].

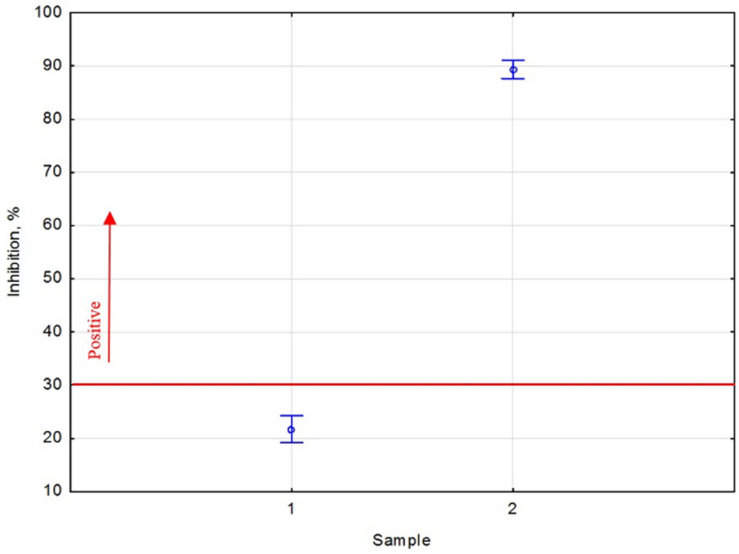

Fig. 5. 95% confidence intervals for inhibition by aqueous extract from incinerated bottom ash; root growth inhibition test of *Sinapis alba*

Table 6

Total metals content in bottom ash [mg·kg^{-1}]

Sample	As	Cd	Cr	Cu	Pb	Ni	Zn	Hg
1	7	2.7	323	4 410	167	121	3 798	0.0135
2	6	8.1	752	1 850	231	347	4 652	0.0132
Average after [28][a]	47	3.7	600	2 700	990	210	3 300	<0.1

[a]Average content of metals in the bottom ash sample from three thermal treatment of MSW plants in [28].

4. CONCLUSIONS

The study of the properties of ash from municipal solid waste incineration is an important source of information for suitable use of ash. The samples were of variable granulometric composition. Higher content of Cu, Cd, Cr, Pb and Ni has been demonstrated. The tested samples were alkali. The samples were assessed from the viewpoint of ecotoxicity, using the aquatic test of immobilisation of *Daphnia magna* and terrestric test of growth inhibition of the root of *Sinapis alba*. It seems that immobilisation and

inhibition proved by these tests were caused by the transition of some elements from the immobile phase into the mobile one (e.g., Cr, Cu, Ni at pH > 7 [29, 30]) during the preparation of water infusion used in testing. The unsuitability of raw bottom ash for the possible use as a secondary material was confirmed. Biological tests are an important tool for the verification whether the specific incinerated waste from the municipal waste incineration is only waste or ecological secondary raw material.

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