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JOLANTA LATOSIŃSKA*, JAROSŁAW GAWDZIK*

EFFECT OF INCINERATION TEMPERATURE ON THE MOBILITY OF HEAVY METALS IN SEWAGE SLUDGE ASH

Mobility of heavy metals has been investigated in sewage sludge as well as in ashes obtained by incineration of this sludge at the temperature range of 850–1000 °C. In the ashes, heavy metals created the most durable connections with immobile fractions which did not have a major significance in a toxicological aspect. A clear influence of temperature on the forms of occurrence of heavy metals in fractions has not been proved. In the range of applied incineration temperatures, only the mobilities of copper and zinc increased with the increase of incineration temperature.

1. INTRODUCTION

The existing norms in EU limit the deposit of municipal sewage sludge on the landfills [1], as well as their environmental usage (Table 1) [2, 3]. Moreover, the realisation of Council Directive 91/271/EEC [4] requirements contributes to the growth of produced sewage sludge (SS). The situation requires an increase of SS incineration.

The methods of thermal SS utilization include combustion, co-incineration of sludge with other waste (e.g., municipal waste) and co-fuelling coal-fired power plants or cement kilns [6]. Using of multiple hearth and fluidized bed furnace are the most popular technologies of the thermal processing of sewage sludge. According to the European Committee Reference Document (BREF) incineration with the fluidized bed was considered as the 0,best available technology (BAT) for the sewage sludge incineration [7]. The incineration does not constitute the zero-waste disposal method since approximately 30% of the solids remain as ash [8]. It is estimated that 1.2 million tonnes of incinerated sewage sludge ash (ISSA) are produced in North America and the EU per year [9].

*Kielce University of Technology, Civil Engineering and Environmental Protection Department, al. Tysiąclecia Państwa Polskiego 7, 25-314 Kielce, Poland; corresponding author J. Latosińska, e-mail: jlatosin@tu.kielce.pl

Table 1

Admissible contents of heavy metals [mg/kg d.m.] in municipal sewage sludge designated for environmental use in accordance to the current standard and planned changes

Metal	Admissible contents of heavy metals in sewage sludge designated for use					
	In agriculture			The Ordinance of the Minister of Environment [3]		
	1986/278/EEC – valid [2]	ENV/E.3/LM – suggested changes [5]		Agriculture and land reclamation for agriculture	Land reclamation for other purposes	Adaptation of lands for particular needs[a]
		Year 2015	Year 2025			
Pb	750–1200	500	200	750	1000	1500
Cd	20–40	5	2	20	25	50
Hg	16–25	5	2	16	20	25
Ni	300–400	200	100	300	400	500
Zn	2500–4000	2000	1500	2500	3500	5000
Cu	1000–1750	800	600	1000	1200	2000
Cr	–	800	600	500	1000	2500

[a]Based on waste disposal plans, land development plans or decisions on the conditions and the land development, for cultivation of compost plants, for cultivation of plants which are not designed for consumption and fodder production.

ISSA containing inorganic components such as Al_2O_3, SiO_2 and in flux (i.e. Fe_2O_3, CaO, MgO) has been used in construction materials to improve their properties (e.g. pozzolanic properties) [10]. According to many authors, ISSA can be used as addition to the production of bricks, tiles, cement mortars, asphaltic paving mixes and aggregate. Moreover, due to the pozzolanic properties, the ashes from sewage sludge can be used in geotechnics [11, 12]. Ashes generated during combustion of sewage sludge contain significant amounts of heavy metals. The concentrations of heavy metals in sewage sludge ashes determine the manner of their utilisation. In the case of exceeding the heavy metals admissible limits, wastes are considered hazardous. One of the criteria for determining the possibility of depositing ash from the sewage sludge on a particular landfill is the content of heavy metals in water extracts [13] (Table 2).

The presence of heavy metals in sewage sludge, especially their high concentration, is caused by the contribution of industrial sewage (e.g. tannery, glaze and metallurgic wastes) in an overall stream of urban sewage. Moreover, heavy metals originate from domestic sewage, surface flushing and the corrosion of sewerage pipes [6]. Heavy metals are present in sewage sludge in the following states: dissolved, precipitated, co-precipitated with metal oxides, adsorbed or associated on particles of biological remains. They can appear in forms of oxides, hydroxides, sulfides, sulfates, phosphates, silicates, organic combinations in the shape of humic groups and compounds with polysaccharides [14].

Table 2

The permissible limits of heavy metals leaching
– the waste acceptance criteria for landfill of the particular type [13]

Metal	Permissible leaching limits [mg/kg d.m.]		
	Landfill for hazardous waste	Landfill for inert waste	Landfill for various hazardous and inert waste
Cu	100	2	50
Cr	70	0.5	10
Ni	40	0.4	10
Cd	5	0.04	1
Pb	50	0.5	10
Zn	200	4	50

Many studies have shown that an activated sludge process removes substantial quantities of metals from influent wastewater. Insoluble metals or metals adsorbed onto settling particles are removed during primary settling; finely suspended or dissolved metals are adsorbed on the sludge flocs during secondary treatment and thus removed during secondary settling [15].

Chemical and biological methods are known for reducing heavy metal content in SS. Chemical methods are mostly based on the use of inorganic or organic acids to solubilise the metals. Acid thermal hydrolysis effectively reduces the concentrations of most heavy metals, with exception of Cu and Pb. Fenton peroxidation releases Cd, Cu, Ni and Zn to a large extent. It cannot be used however for a reduction of Pb and Hg [16]. Biological methods include the solubilisation of the metals by the metabolic action of microorganisms [17].

Removal of heavy metals from SS is inapplicable due to high costs, operational difficulties and low efficiency [18]. Concentrations of heavy metals are not a sufficient index of their actual environmental hazard evaluation. The mobility and bioavailability of heavy metals in the environment also depend on the form of their occurrence.

In order to evaluate mobilities of heavy metals, including the ones from sewage sludge ashes, the speciation analysis method is used [19]. A basic form of speciation is sequential extraction, i.e. classification of an analyte according to the defined physical and chemical properties.

The sequential extraction following procedure recommended by the Community Bureau of Reference (BCR) leads to obtaining four fractions of metals, which differ from each other in the mobilities of heavy metals occurring in them [20]:

- I fraction (mobile) – interchangeable metals undergoing retention and combined with carbonates.
- II fraction (prone to reduction) – absorbed metals on the developed surface of precipitating hydrated iron and manganese oxides in oxygen-free conditions; are mobile, however their release usually proceeds slowly.

• III fraction (prone to oxidation) – sulfides and organometallic compounds.

• IV fraction (immobile) – metals combined with aluminosilicates and/or immobilised in crystalline structures.

The purpose of the research was to explain the impact of varying incineration temperatures on mobilities of heavy metals from ISSA. In this study, sewage sludge was incinerated at: 850, 900, 950, 1000 °C. The accepted range of incineration temperatures of sewage sludge was chosen due to the fact that temperatures in the range 850–950 °C are the most frequently used and according to the applicable regulations, including those on the exhaust gas temperature [21]. The processes during the thermal treatment of sewage sludge at temperatures lower than 850°C are presented by [6]. The portion of heavy metals fraction in the obtained sewage sludge ashes was examined.

2. MATERIALS AND METHODS

Sewage sludge from the Skarżysko-Kamienna waste water treatment plant was used in the tests. The flow capacity of the sewage-treatment plant equals 50 000 PE. The sewage sludge was obtained after the anaerobic digestion, dehydration and drying (solar drier). Its moisture of 59% and pH – 6.9.

The element contents of sewage sludge was marked with a microanalysis method on the X-ray analyser Rőntec GmbH (Fig. 1).

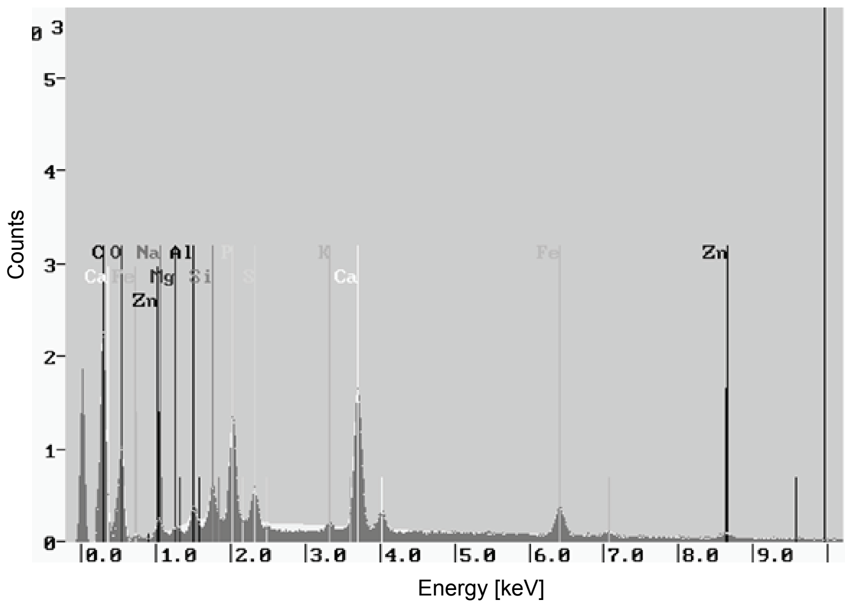


Fig. 1. XRF spectrum of stabilised sewage sludge from the Skarżysko-Kamienna treatment plant

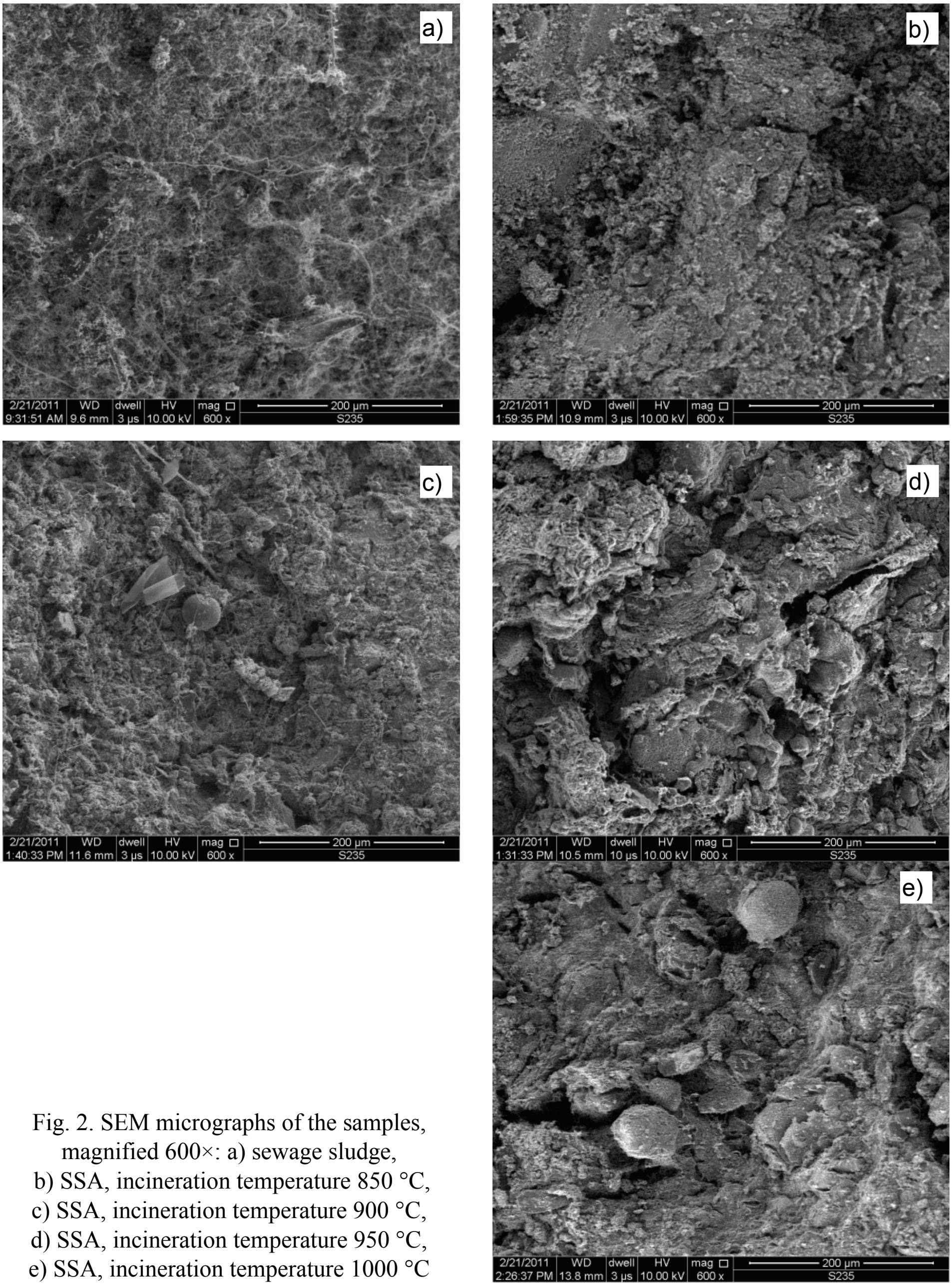


Fig. 2. SEM micrographs of the samples, magnified 600×: a) sewage sludge, b) SSA, incineration temperature 850 °C, c) SSA, incineration temperature 900 °C, d) SSA, incineration temperature 950 °C, e) SSA, incineration temperature 1000 °C

The ashes were obtained as a result of sewage sludge thermal treatment. The sewage sludge was dried in a laboratory drier at 105 °C. Then, it was incinerated in an electric laboratory furnace at: 850, 900, 950 and 1000 °C. The period of time in which the sewage sludge was kept in the furnace resulted from the furnace characteristics. At the maximum temperature, the sewage sludge was kept for 60 min. After the incineration, the samples remained in the furnace until they cooled down 20 °C. This research did not include presence of sand in the bed – SiO_2 which plays an important role in the process of thermal treatment [16].

SEM analysis of the sewage sludge and the sewage sludge ashes was performed with Microscope – QUANTA FEG 250 (Fig. 2).

The BCR tests were applied to determine the heavy metal fraction in sewage sludge and in sewage sludge ashes (SSA). The sequential extraction method used allows one to determine the mobility degree of metals which are present in the matrix. The analytics suggested by European Community Bureau of Reference was used.

Sample collection and pre-treatment. The tests were conducted in accordance with the four-step BCR sequential extraction procedure [20], introducing a change in the method of residual fraction mineralisation, i.e. aqua regia was used in the process of mineralisation [20].

Step one: acid soluble/exchangeable fraction (FI). A 2 g sample of sewage sludge/sewage sludge ash was placed in a 100 cm^3 test-tube for centrifuging. Then, 40 cm^3 of 0.11 M acid solution was added. The sample was shaken for 16 h at room temperature. The extract was separated from the sewage sludge/sewage sludge ash with a centrifuge (4000 rpm). The content of the soluble metals in the water was marked in the liquid.

Step two: reducible fraction (FII). Sewage sludge/sewage sludge ash was washed in 20 cm^3 of distilled water (shaken and centrifuged). Subsequently, 40 cm^3 of 0.1 M hydroxylamine hydrochloride solution of pH = 2, was added to the sewage sludge /sewage sludge ash. Nitric acid was used for the correction of the pH value. The procedure was the same as in step one, the mixture was shaken and centrifuged. Fraction II metals were marked in the liquid.

Step three: oxidation fraction (FIII). The sewage sludge/sewage sludge ash was carried over quantitatively to a quartz evaporating dish and 10 cm^3 of 30% hydrogen peroxide was added. The content of evaporating dish was heated in a water bath at 85° C for 1 h. The process was repeated with the addition of 10 cm^3 of 8.8 M hydrogen peroxide solution to the sewage sludge/sewage sludge ash. After drying, the sample was transferred to test tubes to be centrifuged and then 50 cm^3 of ammonium acetate solution was added (1 M, pH = 2; nitric acid was used to correct the pH value). The sample was shaken for 16 h and afterwards the sewage sludge/sewage sludge ash was separated from the extract. Fraction III metals were marked in the solution.

Step four: residual fraction (FIV). The sewage sludge/sewage sludge ash was washed and dried to a solid state. The mineralisation of the residual fraction was conducted with aqua regia; 30 cm^3 of concentrated hydrochloric acid and 10 cm^3 of concentrated nitric acid were added carefully to a 300 cm^3 conical flask together with 0.5 g of sludge/ash. The conical flask was heated for 30 min and subsequently evaporated to dryness. After cooling, 25 cm^3 of 5% hydrochloric acid were added. The sewage sludge/sewage sludge ash was dissolved, carried over to a metal measuring flask and topped up with 50 cm^3 of distilled water. Then the sample was mixed and strained to a dry dish. In the filtrate the metal forms, fraction IV, were marked.

The heavy metals in the obtained extracts were determined in accordance with ISO 9001:2000 using a Perkin-Elmer 3100 FAAS-BG atomic absorption spectrophotometer (impact bead). Each test was repeated four times.

3. RESULTS AND DISCUSSION

The element content of the sewage sludge from the Skarżysko-Kamienna treatment plant is presented in Table 3. The results of sewage sludge speciation analysis are presented in Fig. 3.

Table 3

Content of elements in sewage sludge

Element	Content [%]
C	30.61 ± 5.61
O	59.65 ±10.38
Na	0.27 ± 0.13
Mg	0.01 ± 0.04
N	1.81± 0.22
Al	0.21 ± 0.05
Si	0.46 ± 0.05
P	1.62 ± 0.09
S	0.50 ± 0.05
K	0.03 ± 0.04
Ca	2.90 ± 0.16
Fe	1.39 ± 0.15
Zn	0.54 ± 0.17

The examined sewage sludge cannot be agriculturally used due to the excess of the admissible contents of heavy metals (Cd, Cr). Its fertilizer usage of the examined sludge is also excluded by the excess of the admissible metal concentration: Cd, Pb, Ni, Cr. The introduction of the suggested heavy metal limit changes (Table 1) similarly excludes the environmental usage of sewage sludge; the excess of Pb, Cd, Ni, Zn, Cu, Cr limits (Fig. 3).

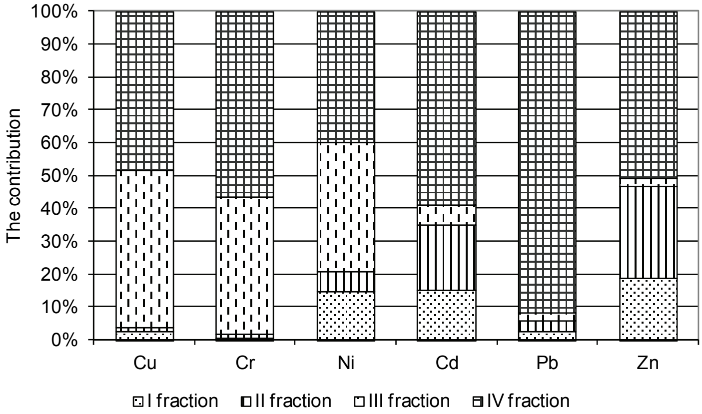
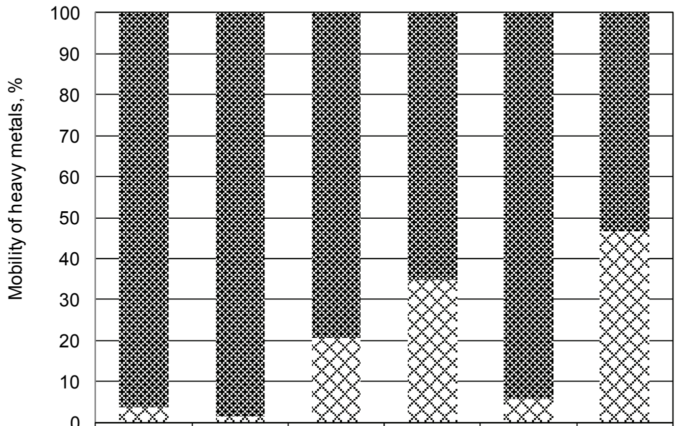


Fig. 3. The contribution per cent of heavy metals in particular fractions in sewage sludge



	Cu	Cr	Cd	Ni	Pb	Zn
Immobile HMs	96.19	98.42	79.31	65.17	94.14	53.30
Mobile HMs	3.8	1.6	20.7	34.8	5.9	46.7

Fig. 4. The mobility analysis of heavy metals in sewage sludge:
fraction I + II – mobile, fraction III + IV – immobile

The mobility of heavy metal analysis in sewage sludge form the Skarżysko-Kamienna treatment plant is presented in Fig. 4. In the examined sewage sludge, the content of mobile fractions (fractions I, II) together with the residual fraction (fraction IV) and the conditionally mobile fraction (fraction III) was minor. Chrome and copper were characterized by a significant immobility in the sewage sludge. The residual fraction contained a large portion of lead, while zinc (fraction I) made the greatest number of mobile connections. The per cent contribution of heavy metals in particular fractions (1–4) for sewage sludge ashes is presented in Figs. 5–8. The statistical results of each fraction of heavy metals in samples are listed in Table 4.

Table 4

Statistical contents[a] of each fraction of heavy metals in samples

Speciation	Heavy metals [mg/kg d.m.]					
	Cu	Cr	Cd	Ni	Pb	Zn
Sewage sludge						
Fraction I	17.2 ± 0.2	12.1 ± 0.2	3.6 ± 0.1	18.9 ± 0.1	13.7 ± 0.6	1484 ± 12
Fraction II	8.3 ± 0.1	20.6 ± 0.2	4.8 ± 0.1	8.0 ± 0.1	16.6 ± 0.7	2187 ± 19
Fraction III	320.1 ± 0.3	872 ± 0.9	1.5 ± 0.1	51.4 ± 0.6	12.2 ± 0.5	199.8 ± 1.5
Fraction IV	324.7 ± 0.3	1169 ± 7	14.2 ± 0.3	51.7 ± 0.1	475 ± 9	3990 ± 35
ΣFI…IV	670 ± 0.9	2074 ± 8.3	24.1 ± 0.6	129.9 ± 0.9	518 ± 11	7861 ± 68
Sewage sludge ash 850 °C						
Fraction I	27.1 ± 0.2	11.8 ± 0.2	9.4 ± 0.2	53.6 ± 1.0	59.0 ± 3.0	8317 ± 75
Fraction II	17 ± 0.2	0.0 ± 0.1	9.2 ± 0.2	22.3 ± 0.8	0.0 ± 0.5	4817 ± 23
Fraction III	401.9 ± 3.1	138.9 ± 0.2	0.0 ± 0.1	5.16 ± 0.2	20.7 ± 2.5	54.8 ± 0.4
Fraction IV	8668 ± 25	23432 ± 55	62.9 ± 0.5	964 ± 7	1812 ± 35	24100 ± 221
ΣFI…IV	9114 ± 29	23584 ± 56	81.4 ± 1.0	1045 ± 9	1892 ± 41	37288 ± 320
Sewage sludge ash 900 °C						
Fraction I	32.5 ± 0.3	3.44 ± 0.2	2.9 ± 0.2	84.6 ± 0.6	47.3 ± 3.2	10656 ± 99
Fraction II	17 ± 0.2	0.0 ± 0.1	5.3 ± 0.2	24.8 ± 0.3	18.4 ± 2.0	5090 ± 22
Fraction III	529 ± 2.4	102.8 ± 0.6	0.0 ± 0.1	4.6 ± 0.2	19.1 ± 2.2	151.4 ± 0.5
Fraction IV	7525 ± 36	19623 ± 96	25.2 ± 0.5	1257 ± 8.8	839 ± 9	17146 ± 76
ΣFI…IV	8103 ± 39	19729 ± 97	33.5 ± 1.0	1371 ± 9.8	924 ± 17	33044 ± 198
Sewage sludge ash 950 °C						
Fraction I	307.6 ± 2.0	3.92 ± 0.2	5.9 ± 0.2	42.3 ± 0.3	41.4 ± 1.2	9682 ± 44
Fraction II	202.8 ± 1.8	0.0 ± 0.1	7.8 ± 0.2	15.0 ± 0.2	17.5 ± 0.4	5855 ± 23
Fraction III	231.4 ± 1.9	114.7 ± 0.7	0.0 ± 0.1	0.0 ± 0.1	16.0 ± 0.3	100.0 ± 0.4
Fraction IV	6573 ± 10	14003 ± 85	35.7 ± 0.7	608.7 ± 5.2	722 ± 11	10669 ± 50
ΣFI…IV	7315 ± 16	14121 ± 86	49.4 ± 1.2	666.1 ± 5.8	797 ± 13	26306 ± 118
Sewage sludge ash 1000 °C						
Fraction I	997.1 ± 9.9	0.48 ± 0.2	5.4 ± 0.2	39.4 ± 0.4	27.6 ± 3.3	6832 ± 22
Fraction II	391.6 ± 1.9	0.0 ± 0.1	11.6 ± 0.3	16.0 ± 0.3	32.1 ± 3.9	3988 ± 19
Fraction III	143.6 ± 0.8	91.4 ± 0.5	0.0 ± 0.1	11.4 ± 0.3	8.3 ± 0.6	88.8 ± 0.5
Fraction IV	6096 ± 31	13717 ± 78	49.5 ± 1.2	585.4 ± 4.4	809 ± 12	8668 ± 38
ΣFI…IV	7629 ± 44	13809 ± 79	66.6 ± 1.9	652.1 ± 5.3	877 ± 20	19577 ± 80

[a]Results are expressed as the mean ± standard deviations.

In the sewage sludge ashes, the smallest concentration of the tested heavy metals was found for cadmium, similarly as for the sewage sludge (Table 4). The increase of the temperature of incineration did not have a significant influence on the change of contribution per cent of fraction IV nickel in ash. For all the ash samples, the contribution of residual fraction (fraction IV) of nickel exceeded 72%. The contribution per cent of mobile fractions (fractions I and II) of nickel in ashes equalled less than 27% for all incineration temperature of sewage sludges.

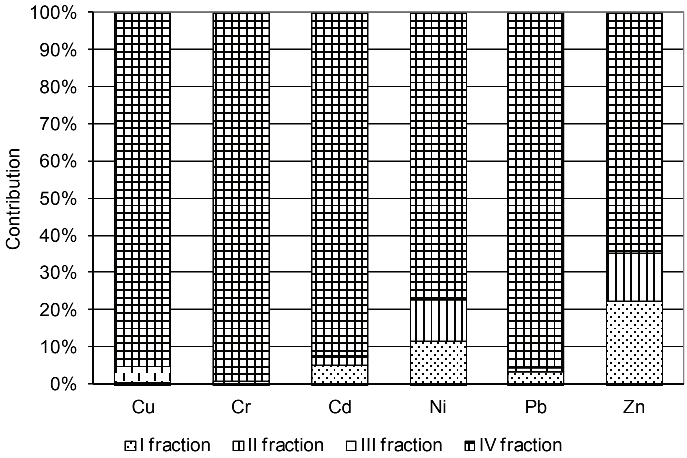


Fig. 5. Contribution of heavy metals in particular fractions in sewage sludge ash; incineration temperature of sewage sludge – 850°C

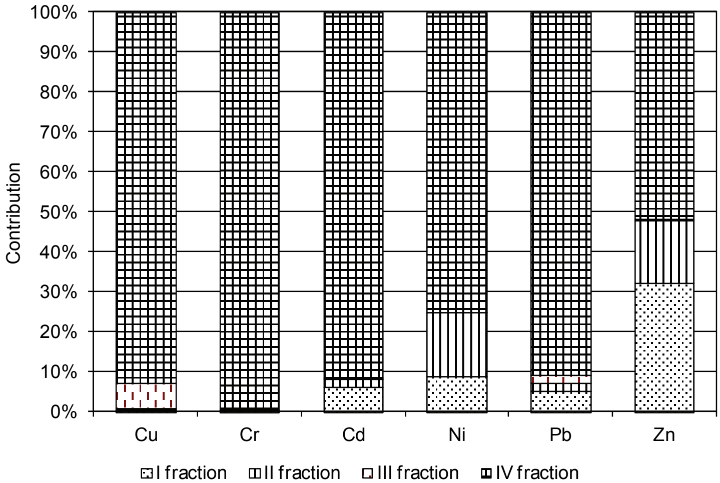


Fig. 6. Contribution of heavy metals in particular fractions in sewage sludge ash; incineration temperature of sewage sludge – 900 °C

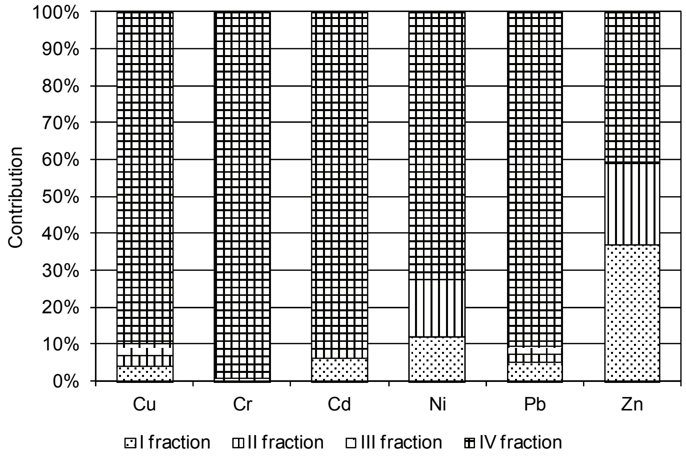


Fig. 7. Contribution of heavy metals in particular fractions in sewage sludge ash incineration temperature of sewage sludge – 950 °C

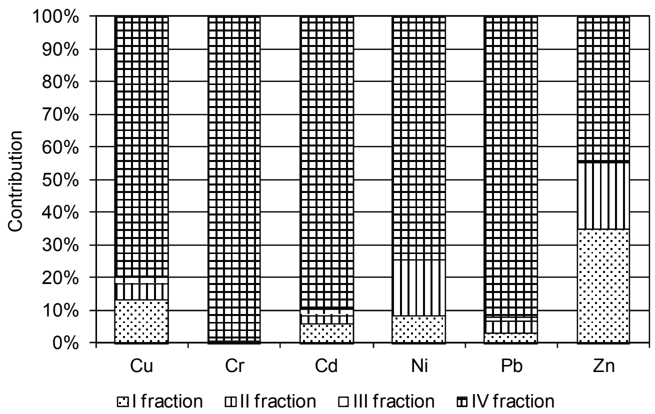


Fig. 8. Contribution of heavy metals in particular fractions in sewage sludge ash; incineration temperature of sewage sludge – 1000 °C

The increase of the incineration temperature caused a decrease of lead concentration of ashes from 1892 mg/kg d.m. (incineration temperature 850 °C) to 797 mg/kg d.m. (incineration temperature 950 °C). The dominant lead fraction in all samples of examined ashes was immobile fraction (fraction IV) and its contribution equalled more than 91%. The increase of incineration temperature did not have any influence on the per cent contribution of chrome fraction in ashes (Fig. 9).

The increase of the incineration temperature caused the decrease of chrome concentration in the examined ashes. However, it did not have any influence on the change of chrome fraction contribution in ashes. The dominant chrome fraction in ashes was fraction IV (immobile), its contribution equalled more than 99%. Copper had high immobility in ashes obtained at 850 °C and 900 °C. However, for ashes obtained at 950 °C and 1000 °C, the mobility of copper clearly increased (Fig. 9).

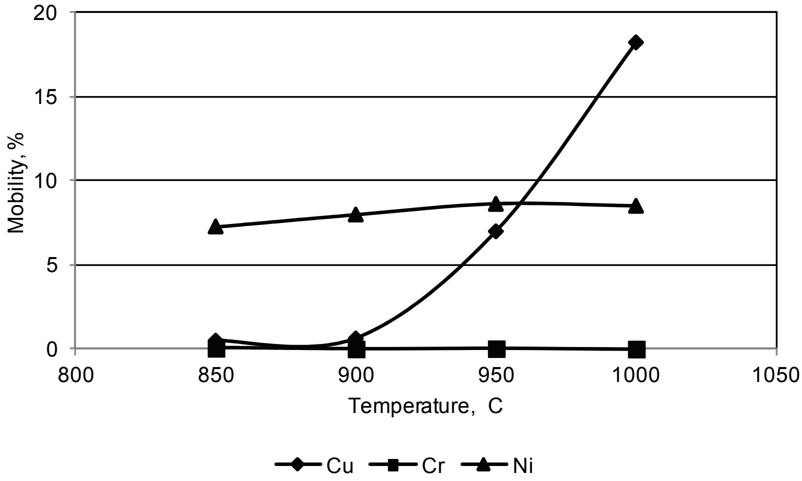


Fig. 9. Dependence of mobility of Cu, Cr and Ni in the sewage sludge ashes on the incineration temperature of sewage sludge

The highest nickel concentration – 1371 mg/kg d.m. – was found in ash incinerated at 900 °C. The concentration of nickel in ashes obtained at 950 °C and 1000 °C was 666.1 mg/kg d.m. and 652.1 mg/kg d.m, respectively. In all samples of examined ashes, nickel dominated in the immobile fraction (fraction IV) on the level above 89.9%.

The highest concentrations in ashes, in comparison to the remaining heavy metals tested, were found for zinc. The decrease of zinc concentration in ashes was noticed upon increasing incineration temperature. The increase of incineration temperature resulted in the decrease of zinc immobility. The mobility of zinc in ashes clearly changed and reached the maximum for ashes obtained at 950 °C (Fig. 10). For the ash obtained at 850 °C, the mobile fraction (fraction I) of zinc constituted 35.22% and for that obtained at 950 °C – 59.06%.

The highest number of mobile connections (fraction I) in sewage sludge ashes was created by zinc, and the highest number of immobile connections (fraction IV) was created by chrome.

Not only temperature of thermal treatment influences behaviour of heavy metals during incineration but also the volatility of metals. Cadmium and zinc are classified as volatile metals, chrome, copper and nickel as non-volatile ones, while lead as intermediate metal [20].

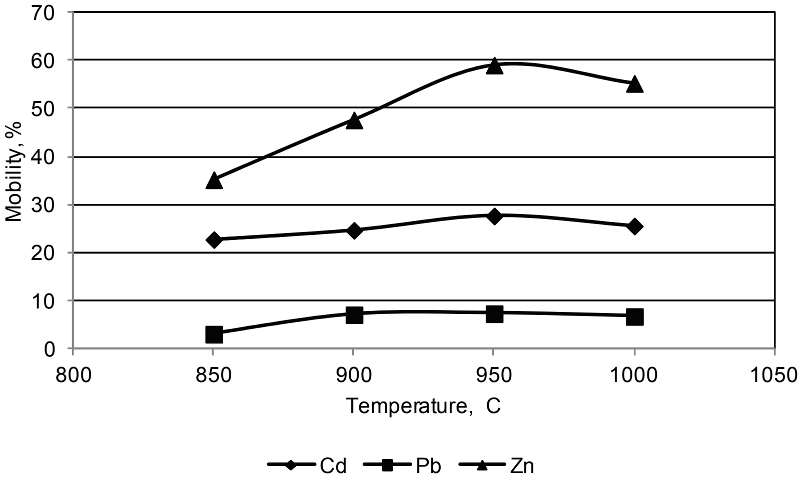


Fig. 10. Dependence of mobility of Cd, Pb and Zn in the sewage sludge ashes on the incineration temperature of sewage sludge

Increase of concentrations of heavy metals in ISSA (e.g. temperature of 850 °C) with respect to the SS may be arranged as follows: Cu > Cr > Ni > Zn > Pb > Cd (Table 4). This may indicate that Pb and Cd are relatively volatile metals. Similar results were observed by [16]. In the next stage of the research, use of the model acc Dewil is planned [16].

In the examined ashes, the accumulation of heavy metals was found mainly in fraction IV, unavailable for plants (Table 3). Due to the levels of heavy metals leached from sewage sludge ash (fraction I) in excess with respect to the admissible values according to [13] (Table 2), this waste cannot be deposited in a non-hazardous and inert landfill.

4. CONCLUSIONS

- In the ashes obtained from the incineration of sewage sludge from the Skarżysko-Kamienna treatment plant, the highest concentration was found for zinc, whereas the lowest for cadmium.
- Heavy metals created the most durable connections with immobile fractions which did not have any considerable significance in the toxicological aspect. The thermal treatment contributed to transfer of metals from organic fractions to residual fractions.
- In the range of incineration temperatures used, the mobility of copper and zinc from ashes increased together with the incineration temperature. The temperature from 850°C to 900 °C used in installations allows one to avoid the increase of copper and

zinc mobility. However, local increase of the incineration temperature, above 900 °C, in an installation can have a negative influence on the properties of the ash produced.

• The mobility of chrome, nickel, cadmium and lead of the examined sewage sludge ashes did not significantly depend on the incineration temperature of sewage sludge.

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