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## THE IMPACT OF COMBUSTION TECHNOLOGY OF SEWAGE SLUDGE ON MOBILITY OF HEAVY METALS IN SEWAGE SLUDGE ASH

### WPŁYW TECHNOLOGII SPALANIA KOMUNALNYCH OSADÓW ŚCIEKOWYCH NA MOBILNOŚĆ METALI CIĘŻKICH Z POPIOŁÓW

**Abstract:** Sewage sludge ashes from grate furnace and fluidized bed furnace were used in this research. This research was carried out to investigate the impact of combustion technology on sewage sludge speciation of heavy metals from sewage sludge ash. This was achieved by conducting a sequential chemical extraction procedure Community Bureau Reference (BCR). This study indicated that heavy metals in sewage sludge ash were dominant in immobile fractions. Moreover, it was stated that the combustion technology of sewage sludge did not have a significant influence on the mobility of heavy metals in ashes.

**Keywords:** sewage sludge ash, heavy metals, mobility

## Introduction

The accumulation of contamination in municipal sewage sludge [1], prevailing norms [2-7], at the simultaneous increase in the amount of formed sewage sludge [8] and the growth of ecological awareness among citizens lead to the increase of sewage sludge neutralized with thermal methods [9-11]. The thermal disposal of sewage sludge includes main methods: combustion (grate furnace, fluidized bed, smelting and rotary furnace), co-combustion (with: coal, other fuels, municipal solid waste), alternative processes (for instance, pyrolysis) [12, 13]. In Poland the dominant combustion technology of sewage sludge is fluidized bed furnace, 8 - installations from all 11 installations [11-14]. According to the European Committee Reference Document (BREF), the incineration with the fluidized bed was considered as the Best Available Technology (BAT) for the sewage sludge incineration [15].

The advantages resulting from sewage sludge incineration include the decrease in weight and volume of wastes, the thermal destruction of toxic organic compounds, the recovery of energy and the minimalization of odour [16]. However, the incineration of

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sewage sludge is not a waste-free method because the forming ash constitutes approximately 30% of the initial sludge mass [13]. The sewage sludge ash, whose chemical and mineral composition depends on *ia* the technology of sewage treatment, the technology of thermal sewage sludge utilization, can represent a potential threat to the natural environment [17].

According to [18, 19], the main elements of the sewage sludge ash are Si, Al, Ca, Fe and P. The phase composition of sewage sludge includes quartz, whitlockite and hematite. Aluminum is typically present in feldspar and amorphous glassy phases [20].

The elements of sewage sludge ash which represent a potential threat are heavy metals. During incineration, most of the heavy metal compounds are vaporized, but later they condense on the surface of the sewage sludge ash particles [13, 21]. Van de Velden et al [22] in a study on an industrial scale lasting over a year, stated that 20% of Hg, 93% of As and almost 100% of Cd and Pb remained in the sewage sludge ash.

In order to define the influence of heavy metals on the environment it is not sufficient to determine only their total content in sewage sludge ash. The total content indicates the degree of environment pollution without providing the information on metal mobility and the resulting natural environment hazard. The mobility of metals depends on the chemical form in which they occur in a particular element of the environment [23-26]. The determination of particular metal forms requires their separation which can be performed by sequential extraction. One of the sequential extraction procedures used is the one developed by the European Community Bureau Reference (BRC) consisting of the following stages [27-30]:

- Stage I:  $\text{CH}_3\text{COOH}$  extraction - aiming at identification and measurement of assimilable and carbonate-bound metals content (FI fraction - exchangeable).
- Stage II:  $\text{NH}_2\text{OH}\cdot\text{HCl}$  extraction - aiming at identification and measurement of the content of metals bound with amorphous iron oxide and manganese oxide (fraction FII - reducible).
- Stage III:  $\text{H}_2\text{O}_2/\text{CH}_3\text{COONH}_4$  - aiming at identification and measurement of the content of organometallic and sulfide fractions (fraction FIII - oxidizable).
- Stage IV: mineralization of residual fraction (after stages FI - FIII) with the mixture of concentrated acid (HCl, HF and  $\text{HNO}_3$ ) - aiming at identification and measurement of the content of metals bound with silicates (fraction FIV - residual).

The literature, according to the authors' knowledge, does not provide numerous research results of heavy metals speciation from environmental sewage sludge ashes samples.

The aim of the study was to evaluate the influence of municipal sewage sludge incineration technology on the mobility of heavy metals accumulated in sewage sludge ashes. This matter is important from the perspective of a potential risk connected with the environmental exposure of heavy metals which is strictly connected with the content of mobile forms.

## Materials

The material for the study was sewage sludge ash obtained from two Polish wastewater treatment plants.

### **Sewage sludge ash from combustion in fluidized bed**

One of the wastewater treatment plants with combustion of sewage sludge in fluidized bed is the installation in Sitkowka-Nowiny. This wastewater treatment plant receives wastewater from the sewer system in Kielce - the capital of this region, Sitkowka-Nowiny municipalities and part of the Maslow municipality. The nominal flow capacity of this sewage treatment plant equals 270 000 PE. The total amount of wastewater influent is municipal wastewater 85 and 15% of industrial waste - mainly from the food and metal industries. This wastewater treatment plant releases around 12 000 Mg/a of sewage sludge. The maximum amount of combustion sludge equals 88.8 Mg/d. This thermal sludge utilization installation is composed of the following main elements:

- reception and storage of sewage sludge,
- sludge drying in discs dryer - drying at 215°C, after drying, sewage sludge has 36% d.m. Dried sludge after leaving the drier is directed to the mixing tank. In this tank dried sludge is mixed with screenings, sand and fat;
- combustion in fluidized bed - combustion at 850°C.

### **Sewage sludge ash from combustion in grate furnace**

One of the Polish sewage treatment plants in which the sewage sludge is incinerated in grate furnace is the wastewater treatment plant in Olsztyn. The sewage is transported to the wastewater treatment plant by means of a separate sewage system. The capacity of the wastewater treatment plant equals 350 000 PE. The amount of sewage sludge intended for combustion equals 760 Mg d.m. The maximum capacity of sewage incineration plant equals 15 Mg/d. The installation of sewage sludge combustion consists of a belt drier and a combustion chamber with a grate. Sewage sludge is incinerated at 850°C.

## **Methods**

The chemical content of the studied sewage sludge ash was defined with the use of the X-ray fluorescence spectroscopy XRF method. The phase contents of sewage sludge ash were determined with the use of the X-ray diffraction XRD method. Grain size distribution of sewage sludge ash was conducted by laser diffractometr. SEM analysis of the sewage sludge ashes was performed with the Microscope - QUANTA FEG 250.

### **Mobility of heavy metals from sewage sludge ash**

The BCR tests were applied to determine the heavy metal fraction in sewage sludge ashes (SSA).

*Sample collection and pre-treatment.* The tests were conducted in accordance with the four-step BCR sequential extraction procedure [27], introducing a change in the method of residual fraction mineralization, *ie* aqua regia was used in the process of mineralization [27].

Step one: acid soluble/exchangeable fraction (FI). A 2 g sample of the sewage sludge ash was placed in a 100 cm<sup>3</sup> test-tube for centrifuging. Then, 40 cm<sup>3</sup> 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 ash with a centrifuge (4000 rpm). The content of the water soluble metals was marked in the liquid.

Step two: reducible fraction (FII). The sewage sludge ash was washed in 20 cm<sup>3</sup> of distilled water (shaken and centrifuged). Subsequently, 40 cm<sup>3</sup> of 0.1 M hydroxylamine hydrochloride solution of pH = 2, was added to the 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 ash was carried over quantitatively to a quartz evaporating dish and 10 cm<sup>3</sup> 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<sup>3</sup> of 8.8 M hydrogen peroxide solution to the sewage sludge ash. After drying, the sample was transferred to test tubes to be centrifuged and then 50 cm<sup>3</sup> 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 ash was separated from the extract. Fraction III metals were marked in the solution.

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

The heavy metals in the obtained extracts were determined using an optical spectrometer with inductively coupled plasma ICP Perkin-Elmer Optima 8000.

The mobility of heavy metals in studied sewage sludge ash was evaluated on the basis of stability indicator  $I_r$ . When a metal has an easy soluble and exchangeable form, the  $I_r$  value is close to zero. In the case when  $I_r \approx 1$ , metal is dominant in the stable forms, mainly residual. The  $I_r$  values between zero and one indicate a differential contribution of metal both in mobile and immobile forms. The mobility indicator was expressed with a formula [31]:

$$I_r = \sum \frac{i^2 F_i}{k^2}$$

where:  $i$  - the next stage of sequential extraction,  $k$  - the maximal number of extractions,  $k = 4$ ,  $F_i$  - the percentage content of metal in every fraction of sequential extraction.

## Results and discussion

Chemical content of sewage sludge ash is presented in Table 1. The main compounds of studied ashes are SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, CaO, MgO, P<sub>2</sub>O<sub>5</sub>. The concentration of SiO<sub>2</sub> in SSA1 equalled 35.72%, and SSA2-25.77%. Adam et al stated similar contribution regarding ashes from sewage sludge combustion in fluidized bed furnace [32]. Si and Al in sewage sludge ash mainly derive from treated sewage *ie* the material carried by pluvial water, cleaning of streets and erosion of the system of piping [33, 34]. A higher content of SiO<sub>2</sub> in SSA1 in comparison to SSA2 results from the combustion technology where during the sewage sludge combustion in the fluidized bed furnace, a part of sand leaves the

chamber together with the sludge ash. A high content of CaO in the studied sewage sludge ashes was similar to the results of studies conducted by Merino et al [33]. A high contribution of phosphorus is caused by the use of detergents by the citizens [33, 34] and coagulants in a technological line of sewage treatment.

Table 1

Chemical composition of sewage sludge ash

Component	Sewage sludge ash from Sitkowska-Nowiny (SSA1)	Sewage sludge ash from Olsztyn (SSA2)
	[%]	[%]
SiO <sub>2</sub>	35.72	25.77
Al <sub>2</sub> O <sub>3</sub>	6.70	9.54
Fe <sub>2</sub> O <sub>3</sub>	9.56	5.12
CaO	17.46	20.70
MgO	4.51	4.48
SO <sub>3</sub>	1.21	0.30
K <sub>2</sub> O	1.61	1.87
Na <sub>2</sub> O	0.52	0.55
P <sub>2</sub> O <sub>5</sub>	19.0	21.58
TiO <sub>2</sub>	1.00	0.77
Mn <sub>2</sub> O <sub>3</sub>	0.14	0.13
SrO	0.05	0.10
ZnO	0.37	0.41
BaO	0.11	0.09
CuO	0.06	0.13
Total organic carbon	0.09	5.39

Cumulative distribution and density distribution of SSA1 and SSA2 was shown in Figures 1 and 2. SSA1 was quite a cohesive material with mean particle size of approximately 80 μm. SSA2 was characterized by mean particle size of approximately 1500 μm.

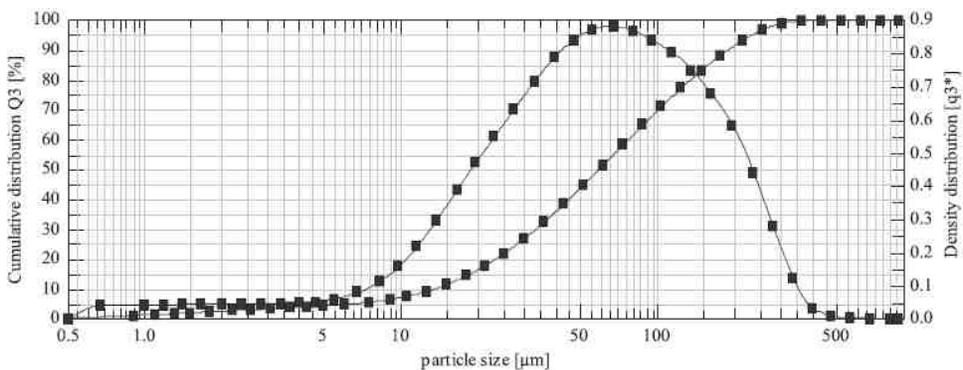


Fig. 1. Cumulative distribution and density distribution of SSA1

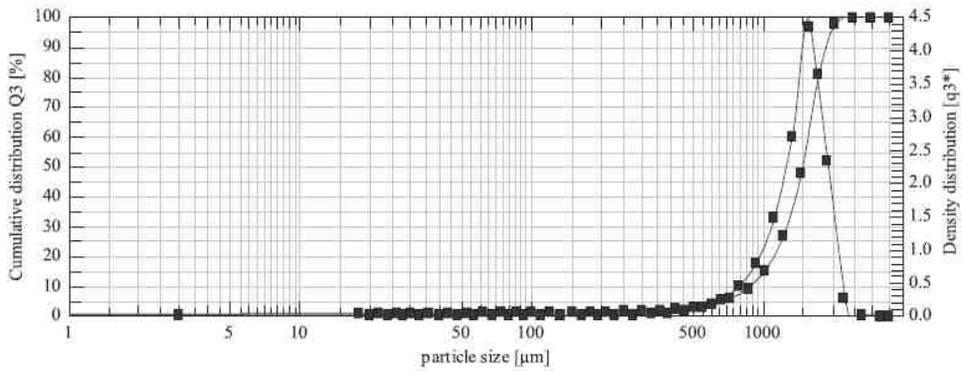


Fig. 2. Cumulative distribution and density distribution of SSA2

Figure 3 presents irregular surfaces of sewage sludge ash grains. Spherical forms, abundantly present in ashes from coal combustion [35-36], in studied sewage sludge ashes were very rare.

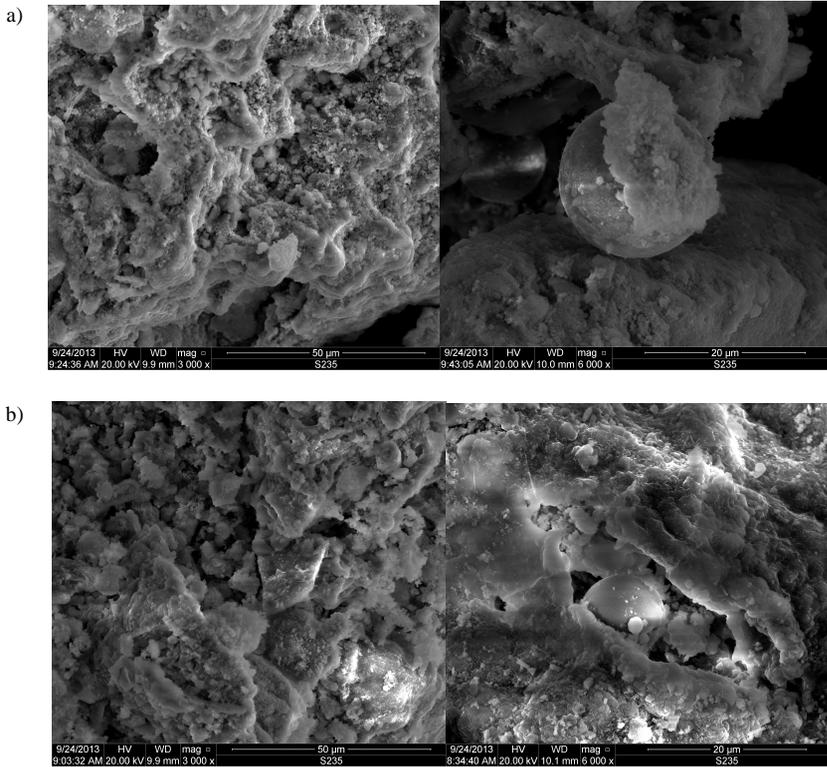


Fig. 3. SEM micrographs of the sewage sludge ash: a) from Sitkowa-Nowiny, b) from Olsztyn

The statistical results of each fraction of heavy metals in samples are listed in Table 2.

The analysis of data obtained with the use of sequential extraction BCR leads to the conclusion that in SSA1 heavy metals occur mainly in a residual fraction FIV. In a mobile fraction FI, the highest recorded value was the one of zinc - 71.651 mg/kg d.m. The FI fraction also contained copper - 11.98 mg/kg d.m., nickel - 2.9722 mg/kg d.m. The content of chrome, cadmium, lead in fraction I was below 1.0 mg/kg d.m. Fraction II similarly to fraction I contained the highest value of zinc - 72.961 mg/kg d.m., secondly of copper - 8.2368 mg/kg d.m., nickel - 3.8101 mg/kg d.m. and cadmium - 0.7432 mg/kg d.m., lead - 0.1587 mg/kg d.m., chrome - 0.082 mg/kg d.m. In a temporally immobile fraction III, the highest value was of lead - 301.89 mg/kg d.m. The smallest number of organic connections and sulfides were found for cadmium - 3.7626 mg/kg d.m. and chrome - 1.4879 mg/kg d.m. In fraction IV the largest number of connections with silicates was formed with zinc - 336.85 mg/kg d.m. and copper - 290.99 mg/kg d.m. In a residual fraction, no cadmium was observed.

Table 2

Statistical contents<sup>1</sup> of each fraction of heavy metals in samples

Speciation	Heavy metals [mg/kg d.m.]					
	Cu	Cr	Cd	Ni	Pb	Zn
Sewage sludge ash from waste water treatment plant in Sitkowa-Nowiny						
FI	11.98 ±0.2085	0.3316 ±0.019	0.9002 ±0.0036	2.9722 ±0.0202	0.0094 ±0.094	71.651 ±1.0246
FII	8.2368 ±0.0799	0.082 ±0.0788	0.7432 ±0.0056	3.8101 ±0.0419	0.1587 ±0.1786	72.961 ±0.3502
FIII	85.419 ±1.2642	1.4879 ±0.1278	3.7626 ±0.0497	9.9175 ±0.0238	310.89 ±2.0208	256.21 ±2.8183
FIV	290.99 6.4018	73.918 ±5.2851	0±0.0000	28.897 ±0.052	112.47 ±0.9672	336.85 ±5.4907
ΣFI-IV	396.6258	75.8195	5.40	45.5968	423.5281	737.672
Sewage sludge ash from waste water treatment plant in Olsztyn						
FI	8.5196 ±0.2258	0.2943 ±0.0336	0.3324 ±0.0137	4.2244 ±0.0317	0.1601 ±0.1222	56.578 ±0.7355
FII	1.1143 ±0.0761	0.2740 ±0.0487	0.2673 ±0.0164	3.3392 ±0.0170	0.6368 ±0.1129	58.484 ±1.3334
FIII	58.099 ±1.7197	0.5404 ±0.0796	0.2545 ±0.0190	5.7693 ±0.0756	1.4961 ±0.2166	78.275 ±0.2192
FIV	757.26 ±16.8869	110.01 ±10.484	0.0000 ±0.0000	44.576 ±0.3923	31.936 ±0.3417	410.09 ±5.3722
ΣFI-IV	824.9929	111.1187	0.8542	57.9089	34.229	603.427

<sup>1</sup>Results are expressed as the mean ± standard deviations

In studied SSA2, heavy metals occurred mainly in immobile connections with silicates - fraction IV. In fraction I the highest amount, similarly to SSA1, was observed for zinc - 56.578 mg/kg d.m. The amount of chrome, nickel and lead were below 0.4 mg/kg d.m. In mobile fraction II the highest value was found for zinc - 58.484 mg/kg d.m., secondly for nickel - 3.3392 mg/kg d.m., copper - 1.1143 mg/kg d.m. The content of lead, chrome, cadmium did not exceed 0.7 mg/kg d.m. In fraction III the highest value was found for zinc - 78.75 mg/kg d.m. The smallest amount in fraction III was observed for cadmium -

0.2545 mg/kg d.m. In fraction IV, the highest amount was found for copper - 757.26 mg/kg d.m. In a residual fraction, similarly to SSA1, no cadmium was observed.

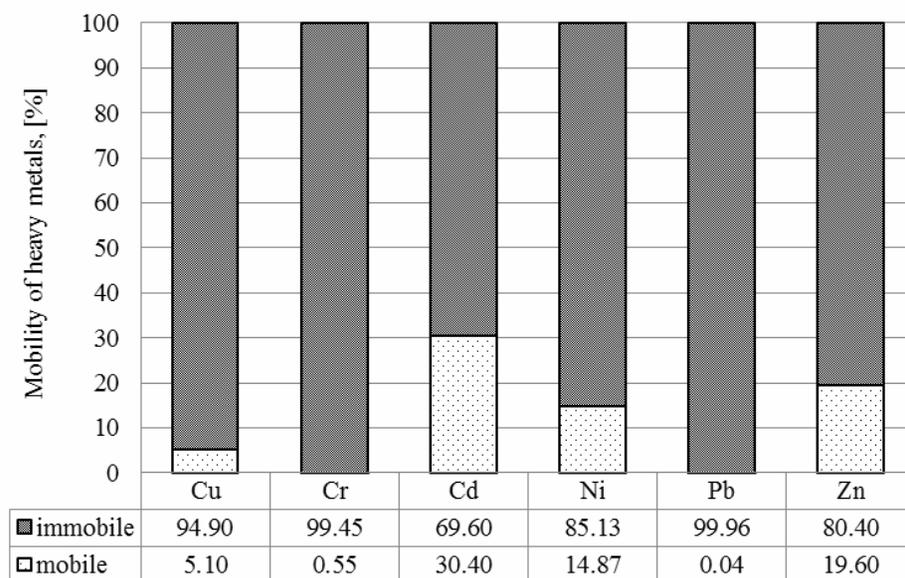


Fig. 4. The mobility analysis of heavy metals in sewage sludge ash from Sitkowka-Nowiny, fraction I + II - mobile, fraction III + IV - immobile

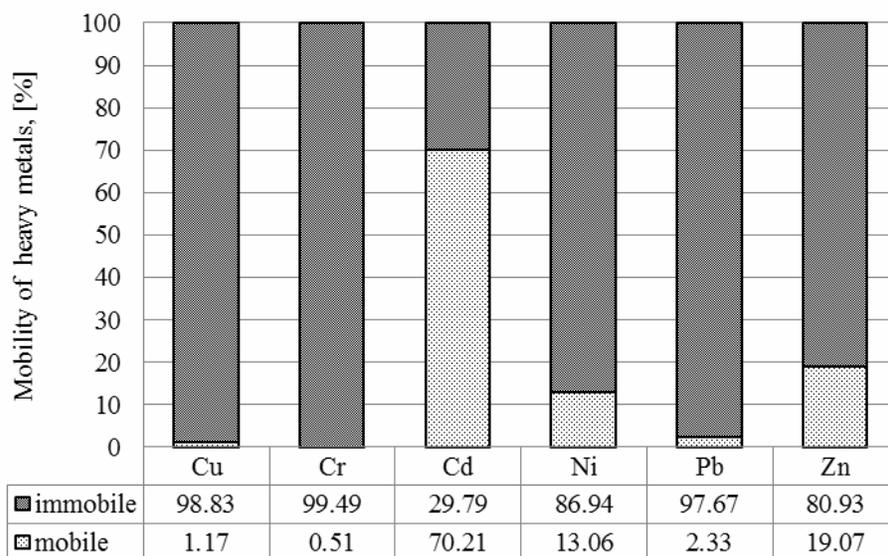


Fig. 5. The mobility analysis of heavy metals in sewage sludge ash from Olsztyn, fraction I + II - mobile, fraction III + IV - immobile

The percentage contribution of heavy metals in mobile fractions (I+II) and immobile (III+IV) for SSA1 and SSA2 is presented on Figures 4 and 5. The metal with the highest percentage contribution in the mobile fraction for both studied ashes was cadmium 30.4% for SSA1, 29.79% for SSA2. The heavy metal with a similar percentage contribution of the mobile fraction (I+2) in SSA1 and SSA2 was zinc, 19.6% and 19.07% respectively.

The percentage contribution of heavy metals in mobile fraction (I+II) for sewage sludge ashes occurred in the following series of decreasing values:

- for SSA1: Cd > Zn > Ni > Cu > Cr > Pb,
- for SSA2: Cd > Zn > Ni > Cu > Pb > Cr.

The values of heavy metals stability indicator in the studied ashes is presented on Figure 6. The metal of lowest stability indicator value for SSA1 and SSA2 was cadmium 41.9% and 27.0% respectively. The heavy metal with the strongest bond was chrome for which the stability indicator value for SSA1 equaled 98.6% and for SSA2 it was 99.4%.

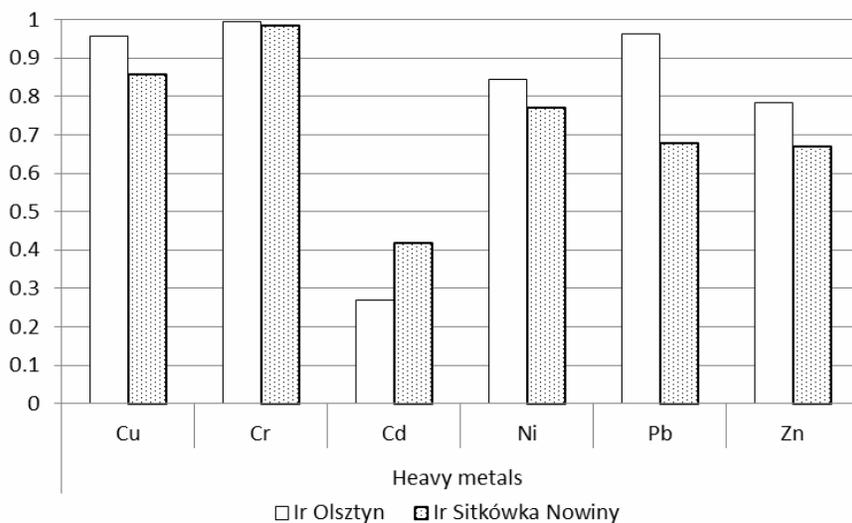


Fig. 6. The indicator of heavy metals stability of studied ashes

## Conclusions

In the studied sewage sludge ashes obtained from both the wastewater treatment plant with a combustion of sewage sludge in a grate furnace and in a fluidized bed, heavy metals are dominant in fractions which are considered as immobile. These were metals bound with aluminosilicates, sulfates and permanent organometallic connections.

The series of percentage contribution of heavy metals in sewage sludge ashes fractions differ only in the order of chrome and lead.

The mean stability indicator for SSA2 ash is 7% higher from the mean stability indicator for SSA1 ash. It occurs together with a smaller mobility of Cu, Cr, Ni, Pb and Zn in ash from the installation with grate furnace combustion. Only in the case of cadmium, the stability indicator for SSA2 is significantly smaller.

The conducted research indicates that the used technology of combustion of municipal sewage sludge did not have a greater influence on the mobility of heavy metals from sewage sludge ash.

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## **WPŁYW TECHNOLOGII SPALANIA KOMUNALNYCH OSADÓW ŚCIEKOWYCH NA MOBILNOŚĆ METALI CIĘŻKICH Z POPIOŁÓW**

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**Abstrakt:** Badania przeprowadzono w celu oceny wpływu technologii spalania osadów ściekowych na mobilność metali ciężkich z popiołów. W badaniach wykorzystano popioły z osadów ściekowych pobrane z oczyszczalni ścieków ze spalaniem osadów w piecu rusztowym i złożu fluidalnym. Zastosowano chemiczną procedurę ekstrakcji sekwencyjnej Community Bureau Reference (BCR). Stwierdzono, że metale ciężkie w popiołach z osadów ściekowych dominowały we frakcjach niemobilnych. Ponadto zauważono, że technologia spalania komunalnych osadów ściekowych nie miała znaczącego wpływu na mobilność metali ciężkich z popiołów.

**Słowa kluczowe:** popiół z osadów ściekowych, metale ciężkie, mobilność