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## The Release of Heavy Metals from Hardening Slurries with Addition of Selected Combustion By-products

**Uwalnianie metali ciężkich z zawiesin twardniejących z dodatkiem wybranych ubocznych produktów spalania**

In Poland, in recent years, there has been a rapid accumulation of sewage sludge - a by-product in the treatment of urban wastewater. This has come about as a result of infrastructure renewal, specifically, the construction of modern sewage treatment plants. The more stringent regulations and strategic goals adopted for modern sewage management have necessitated the application of modern engineering methodology for the disposal of sewage sludge. One approach is incineration. As a consequence, the amount of fly ash resulting from the thermal treatment of municipal sewage sludge has grown significantly. Hence, intensive work is in progress for environmentally safe management of this type of waste. The aim of the experiment was to evaluate the possibility of using the fly ash that results from municipal sewage sludge thermal treatment (SST) as an additive to hardening slurries. This type of hardening slurry with various types of additives, e.g. coal combustion products, is used in the construction of cut-off walls in hydraulic structures. The article presents the technological and functional parameters of hardening slurries with an addition of fly ash obtained by SST. Moreover, the usefulness of these slurries is analysed on the basis of their basic properties, i.e. density, contractual viscosity, water separation, structural strength, volumetric density, hydraulic conductivity, compressive and tensile strength. The mandated requirements for slurries employed in the construction of cut-off walls in Floyd embankments are listed as a usefulness criteria. Subsequently, leaching tests were performed for heavy metals in the components, the structure of the hardening slurries. An experiment showed leaching of hazardous compounds at a level allowing their practical application. The article presents the potential uses of fly ash from SST in hardening slurry technology. It also suggests directions for further research to fully identify other potential uses of this by-product in this field.

**Keywords:** hardening slurry, fly ash from thermal treatment of municipal sewage sludge, cut-off walls

### Introduction

After the Polish accession to the European Union (EU), the criteria and procedures concerning water and sewage management, as well as industrial or municipal waste, have been significantly tightened. The National Programme for Urban Wastewater Treatment (KPOSK) involves the construction of modern sewage

treatment plants. From 2004 to 2014, a more than 14% increase in the number of municipal sewage treatment plants (from 2875 in 2004 to 3288 in 2014) was recorded [1]. However, the rise in the number of highly efficient specialist facilities has resulted in a relatively fast increase in the amount of the major by-product of the sewage treatment process, i.e. municipal sewage sludge. According to the data from the Central Statistical Office of Poland [1], the amount of municipal sewage sludge produced in 2014 alone has reached 556.0 thousand tonnes d.m. This is a huge amount, but less than the expected 651.0 thousand tonnes d.m. [2]. Thus, the steady annual increase in the amount of municipal sewage sludge at around 2.0÷2.5%, poses enormous problems for its safe management.

The data presented in the literature [1, 3] clearly indicate a decrease in the amount of sludge stored in landfills (from about 16% in 2008, to about 5% in 2014) and an increase in the amount of sludge processed thermally (from about 1% in 2008, to about 15% in 2014). The above trends also match the goals set by the municipal sewage sludge management directives [4]. These are:

- reducing (or abandoning) sewage sludge storage,
- increasing the amount of municipal sewage sludge processed before re-introduction to the environment, as well as the amount of sludge recycled by thermal methods,
- maximizing the use of biogenic substances contained in the sludge, while meeting all the requirements related to health and chemical safety.

Still, experts in the field of waste agree on the current absence of a clear strategy for municipal sewage sludge management in Poland [5], as well as on the need for development and investments in modern thermal methods [6].

The sewage sludge thermal treatment technique (SSTT) makes it possible to change the chemical and biological composition of sludge, to neutralise pathogens and to significantly decrease the weight or volume of sludge. The thermal methods of dealing with municipal sewage sludge include incineration, co-incineration and other processes, such as wet oxidation, pyrolysis, gasification and vitrification [7]. Over the last years, the use of these methods has increased in Poland, and therefore the amount of coal combustion products (CCP), such as ash, has increased. Unfortunately, the sewage sludge incineration process does not eliminate the high content of phosphorous and heavy metals in sewage sludge. Therefore, research continues towards developing effective, environmentally safe methods of managing/using ash from the thermal treatment of sewage sludge. A popular solution to processing fly ash from SSTT is their use in the ceramic industry or construction, e.g.: solidification of concrete blocks or sintering to a granulated form [8]. The main purpose of these methods is a safe and economical immobilization of hazardous compounds within the obtained material structure. It should be noted that research on hardening slurries containing other types of coal combustion products, e.g.: conventional ash resulting from fluidised bed combustion, showed an improvement in the hydraulic conductivity of hardening slurries under both capillary and diffusive conditions [9].

The aim of the experiment was to evaluate the possibility of using as an additive to hardening slurries, and in a manner safe for the natural environment, the fly ash that results from municipal sewage sludge thermal treatment (SSTT). This type of hardening slurry with various types of additives, e.g. coal combustion products, is used in the construction of cut-off walls in hydraulic structures.

## 1. Type and properties of hardening slurries

In hydraulic structures, cut-off walls are normally constructed by way of narrow (trench) spatial excavations. The excavations are first expanded by the addition of bentonite and water slurries, and are then filled with cohesive soil, modified local soils, concrete, as well as loam-concrete or so-called hardening slurries.

A hardening slurry is defined as a slurry which hardens over time and contains cement or another binder and additional materials, such as loam (bentonite), granulated blast furnace slag or fly ash, fillers and admixtures [10]. If chemical admixtures are excluded from the slurry compositions, the remaining components are of a mineral character. Some of these are by-products of certain waste technology processes.

The slurries used or tested in Poland can be classified in terms of the types of their components [11]. These are:

- cement-bentonite-water,
- cement-bentonite-water with chemical admixtures,
- cement-bentonite-water with additives, such as sand, hard or lignite coal ash, hard or lignite coal fluidised bed combustion ash, blast furnace slag,
- bentonite-water with additives, such as lignite coal ash, hard coal ash, lime,
- cement-bentonite-water with additives, the so-called “company mixes”.

Information on the basic properties of slurries can be found in the literature, e.g. [11, 12]. However, application of the coal combustion products requires, apart from typical tests, detailed compliance tests allowing to apply this type of material in the water and soil environment. From the point of view of environment protection, tests of immobilization of hazardous compounds contained in the used components (CCP) of hardening slurries become especially important.

## 2. Characteristics of ash from thermal treatment of sewage sludge

Sewage sludge ash is produced by the incineration of sewage sludge at a high temperature (about 600÷920°C), most often in the fluidised bed process. This process ensures a considerable reduction in the volume of material and an enhanced yield of thermal energy. Moreover, it results in ash with specific characteristics not found among the usual by-products of coal combustion. Owing to the high content of organic substances in sewage sludge, ash from the thermal treatment of sewage sludge may contain 0.3÷1.5% of total phosphorus [13], which negatively affects

(prolongs) the delay of the cement hydration process in concrete based on this additive [14]. The relatively high content of heavy metals is also problematic, so it is necessary to immobilise these (e.g. in hardening slurries).

The phosphorus content, as well as the presence of heavy metals indicate a very limited suitability of SSTT ash for the construction industry, and particularly for current concrete technology. Moreover, the high water demand and low pozzolanic activity of this ash preclude its use as an additive in ready-mix concrete. However, these properties open up the possibility of using it in hardening slurries, in which the ratio of w/s (water/dry components) and the resulting strength are subject to other assessment criteria.

Table 1 shows the concentrations of heavy metals in the ash from SSTT and binder (cement) used in the experiment. The marking was performed mineralizing an ash sample (using HNO<sub>3</sub> and HClO<sub>4</sub> acids in a proportion of 3:1). The heavy metal concentrations were then measured using Atomic Absorption Spectrometry (AAS) [15].

Table 1. Heavy metals concentration in components of hardening slurry

Item	Marked metal	SSTT ash mg/kg d.m.	Cement CEM I 32.5R mg/kg d.m.
1	Cd	11.25	10.95
2	Cu	599.17	39.73
3	Ni	39.10	12.90
4	Pb	31.60	47.90
5	Zn	2302.00	151.50

### 3. Creating formulae of hardening slurries

The hardening slurries used in this study were prepared from the following ingredients:

- tap water,
- sodium bentonite *BDC*,
- Portland cement CEM I 32.5R,
- SSTT fly ash.

Table 2 shows the composition of these slurries (percentages of components in 1.0 m<sup>3</sup> of the slurry) used in the experiment. For each formula, the water/cement ratio (w/c) and water/dry ingredients ratio (w/s) are listed.

Table 2. Formulae of hardening slurries (as the percentages of components in 1.0 m<sup>3</sup> of the slurry)

No.	Formula	Water	Bentonite	Ash	Cement	w/c	w/s
1	R1	66.6	2.7	20.0	10.7	6.25	2.00
2	R2	54.6	2.2	32.3	10.9	5.00	1.20

## **4. Methods of testing hardening slurries**

### **4.1. Tests of technological properties (in liquid state)**

The first part of the experiment included compliance tests of hardening slurries with regard to their practical application. The marked technological and functional parameters were referenced to the limit values for hardening slurries characterising the demands in relation to anti-filtration barriers executed in flood embankments [12]. As part of marking the technological parameters of the tested hardening slurries, the following was measured: density [16], conventional viscosity [16] daily water setting [17], structural strength [16]. The mentioned technological properties were determined immediately after the execution of a batch of a given slurry formula.

Hardening slurry test cylinders were prepared in PVC and steel moulds (cylinder) of 8 cm in diameter and 8 cm in height. Before the slurry set, the samples were kept under a foil covering in the laboratory. After 2-3 days, the samples were submerged into water. The samples were left under water until the moment of measurement. The water temperature was  $18 \pm 2^\circ\text{C}$ .

### **4.2. Tests of functional properties (after hardening)**

As part of marking the functional parameters, the following were determined: volumetric density [18], hydraulic conductivity [19], tensile splitting strength tests [20]. All these parameters were marked after 28 days of slurry maturation.

### **4.3. Tests for the leaching of heavy metals from the hardening slurries structure**

The dried samples of hardening slurry of the two tested formulae (R1 and R2) were assessed after 7, 14 and 28 days of maturation. In this work, the maturing time of the samples is significant due to binder (cement) hydration, as the formation of a tight C-S-H matrix is strongly correlated with the maturity time. The time periods utilized in the experiment: 7, 14 and 28 days of sample maturation result from the adoption of that employed in the research of materials that are based on hydraulic binders. In order to extract heavy metals, the following solutions were used: water ( $\text{H}_2\text{O}$ ), hydrochloric acid (HCl), and ethylenediaminetetraacetic acid (EDTA). Leaching of heavy metals (zinc, copper, lead, nickel, and cadmium) was determined in a 10 g sample after first rinsing with  $50 \text{ cm}^3$  of the stated solvent, and then 10 g of each sample was shaken with  $100 \text{ cm}^3$  of the relevant extraction solvent for six hours. The suspensions were subsequently centrifuged and filtered, and the obtained extract was analysed for metal content using Flame Atomic Absorption Spectroscopy (FAAS) [21]. All the heavy metal markings were executed during one test run, which was later repeated. The results of both test runs provided the average value.

## 5. Test results

### 5.1. Technological and functional properties of hardening slurries

Table 3 shows the values of technological (in liquid form) and functional parameters (after hardening) of the investigated hardening slurries. For particular parameters, the limit values characterising the requirements for flood embankment anti-filtration barriers constructed from hardening slurries were shown.

Table 3. Technological and functional parameters of the tested hardening slurry formulae

No.	Parameter	R1	R2	Limit value [12]
technological parameters				
1	density, g/cm <sup>3</sup>	1.28	1.43	1.20÷1.60
2	conventional viscosity, s	38	50	max 50
3	water setting, %	4.0	3.0	max 4.0
4	structural strength after 10 min, Pa	1.40	1.87	min 1.40
functional parameters				
5	volumetric density, g/cm <sup>3</sup>	1.33	1.39	max 1.60
6	hydraulic conductivity, m/s	$3.71 \cdot 10^{-6}$	$8.84 \cdot 10^{-7}$	$\leq 10^{-8}$
7	compressive strength, MPa	0.13	0.21	0.5÷2.0

Table 4. Values of heavy metals leached from the tested hardening slurry formulae

No.	Solvent	Formula	Maturation time days	Value of the leached metals in relation to the hardening slurry mass				
				Cd	Cu	Ni	Pb	Zn
				mg/kg d.m.				
1	H <sub>2</sub> O	R1	7	0.05	0.05	0.05	0.69	0.14
2			14	0.05	0.05	0.46	1.335	0.16
3			28	0.05	0.05	0.595	1.475	0.235
4		R2	7	0.05	0.265	0.075	1.12	0.175
5			14	0.05	0.19	0.05	1.025	0.185
6			28	0.05	0.31	0.05	0.585	0.265
7	HCl 0.1 M	R1	7	0.05	0.05	0.475	1.24	0.285
8			14	0.05	0.05	0.42	1.3	0.48
9			28	0.06	0.05	0.62	1.2	0.195
10		R2	7	0.05	1.235	0.51	1.83	0.05
11			14	0.09	1.865	0.46	1.595	0.05
12			28	0.075	1.48	0.42	1.79	0.055
13	EDTA 0.1 M	R1	7	0.475	1.6	2.76	10.965	22.9
14			14	0.53	1.85	2.67	11.225	47.35
15			28	0.545	1.905	2.69	11.975	33.5
16		R2	7	0.36	24.95	3.24	8.59	38.1
17			14	0.355	22.6	2.91	8.275	26.2
18			28	0.38	24.05	3.275	9.32	24.95
19	limit value, mg/kg d.m. [22]			0.50	20	10	20	60
20	limit value, mg/kg d.m. [23]			1.0	25	20	40	80

## 5.2. Leaching of heavy metals from the hardening slurries

Table 4 lists the values of the marked heavy metals as leached from a hardening slurry matrix for the tested formulae. The results of heavy metal leaching are presented as heavy metal values in relation to the used mass of hardening slurry. For comparative purposes, Table 4 contains the limit values of heavy metals in soft and very soft soils, in a layer of up to 30 cm according to [22, 23].

## 6. Discussion of test results

When analysing the received values of tested hardening slurry technological and functional parameters in respect to the formulated criterion, it should be noted that both formulae met all the requirements in relation to desired properties when in the liquid form. However, as hardened slurry mass, in the case of functional parameters, for both tested formulae, the exceeding of the criterion in relation to the value of acceptable hydraulic permeability and compressive strength was noted. This means that the tested slurries should not be used during flood embankment anti-filtration barrier construction due to too low tightness of the chemical structure and strength of the matrix framework. Still, in the presented form, the material can be used during, for example, void filling in mining damage recoveries or in site restoration.

Despite this, the possibility of the release of heavy metals contained in the ingredients of various formulae from the slurries structure must be assessed. The potentially weaker structure of internal matrix framework of both tested formulae enables the easy leaching of hazardous compounds into the external environment (e.g. soil or groundwater).

On the basis of the data contained in Table 4, average values were calculated of the content of the marked heavy metals released by way of extraction with relevant solutions, from the formulae of the tested hardening slurries. The results are shown in Figures 1-5.

In the case of the analysis of the leached nickel (Fig. 1), in accordance with [22, 23], the limit value of the set comparative criterion was not exceeded in any of the formulae, however, the highest concentration of the marked metal was recorded in the case of extraction of both formulae with a solution of ethylenediaminetetraacetate acid (EDTA).

In the case of cadmium, an exceeding of the average value of the leached metal from the acceptable criterion according to [22], was recorded for the R1 formula EDTA solution extraction (Fig. 2). In the case of the R2 formula, in which the w/c and w/s ratios were lower, no such exceeding was recorded. In the case of extraction with a water (H<sub>2</sub>O) and a hydrochloric acid (HCl) solution, the values of leached cadmium were similar for both analysed formulae. Regarding the leached lead values, these do not exceed the acceptable criterion (Fig. 3). Lower levels of leached lead were recorded for the R2 formula in the case of extraction with a water or an EDTA solution, while the reverse situation occurred in the case of

activity with a HCl acid solution. The highest values of Pb leached from the hardening slurry matrix (for the R1 and R2 formulae) were recorded in the case of EDTA solution activity.

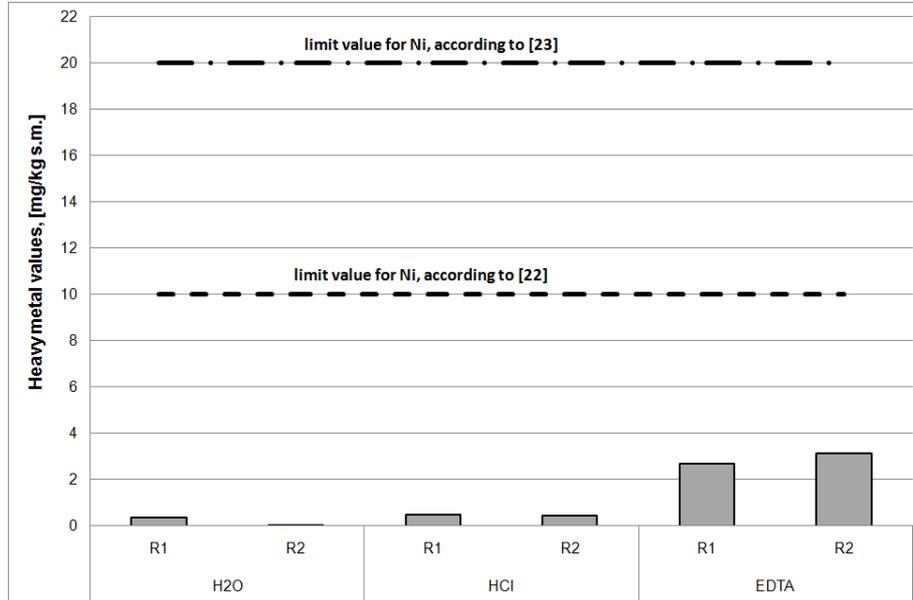


Fig. 1. Content of nickel (Ni) leached from the tested hardening slurry formulae

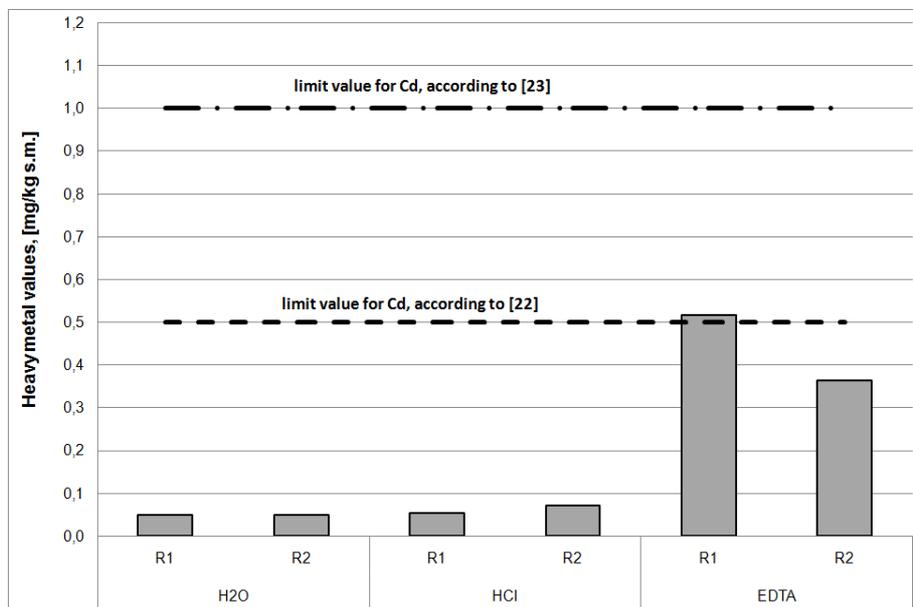


Fig. 2. Content of cadmium (Cd) leached from the tested hardening slurry formulae

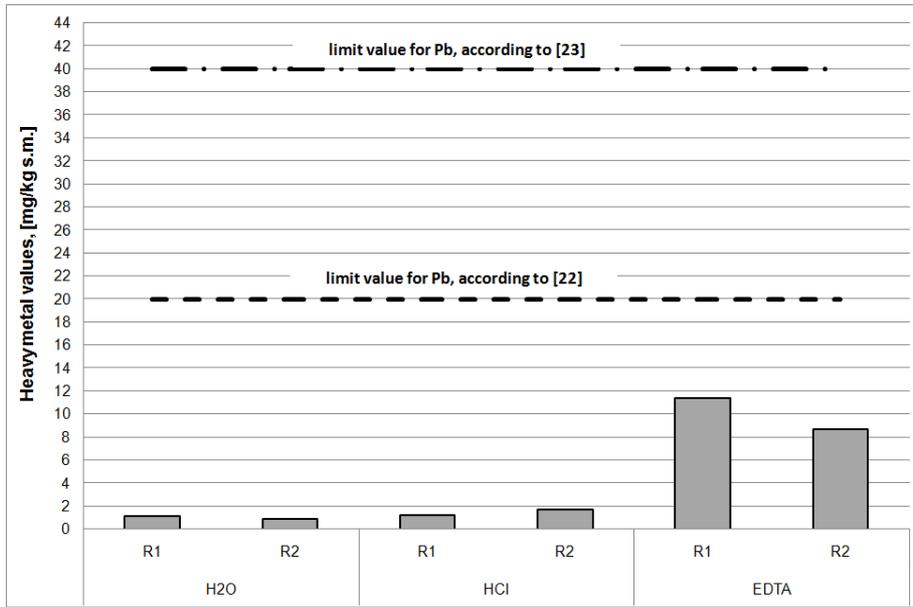


Fig. 3. Content of lead (Pb) leached from the tested hardening slurry formulae

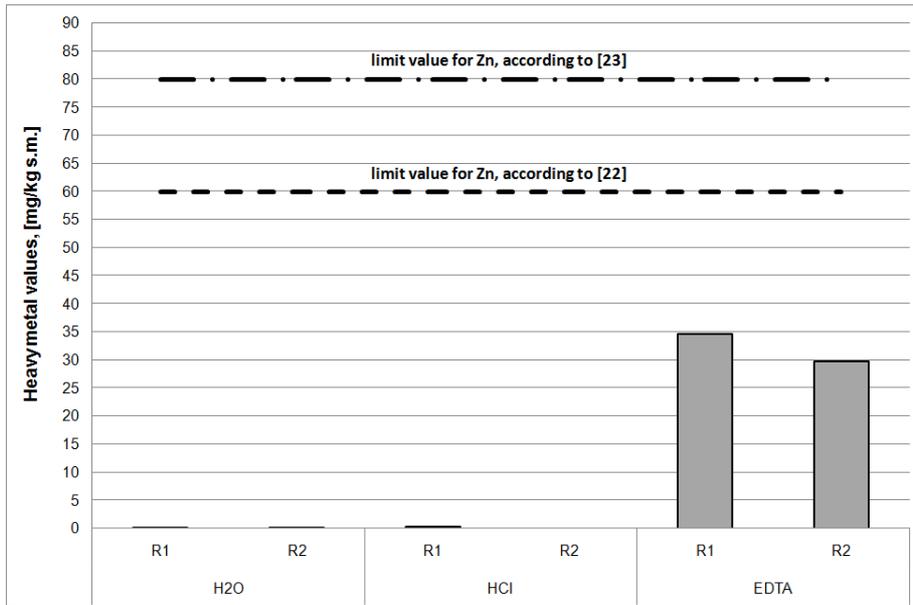


Fig. 4. Content of zinc (Zn) leached from the tested hardening slurry formulae

Figure 4 shows the concentrations of leached zinc from the tested hardening slurry formulae. In both analysed recipes, the maximum value established in the acceptable criterion was not exceeded, yet a definite difference exists in the share

of the leached metal in relation to the acting solutions. In the case of extraction with the EDTA solution, this value is tens times greater than in the case of other solutions. Moreover, for all the analysed solutions, the values of the leached metals were also higher in the case of the R1 formula.

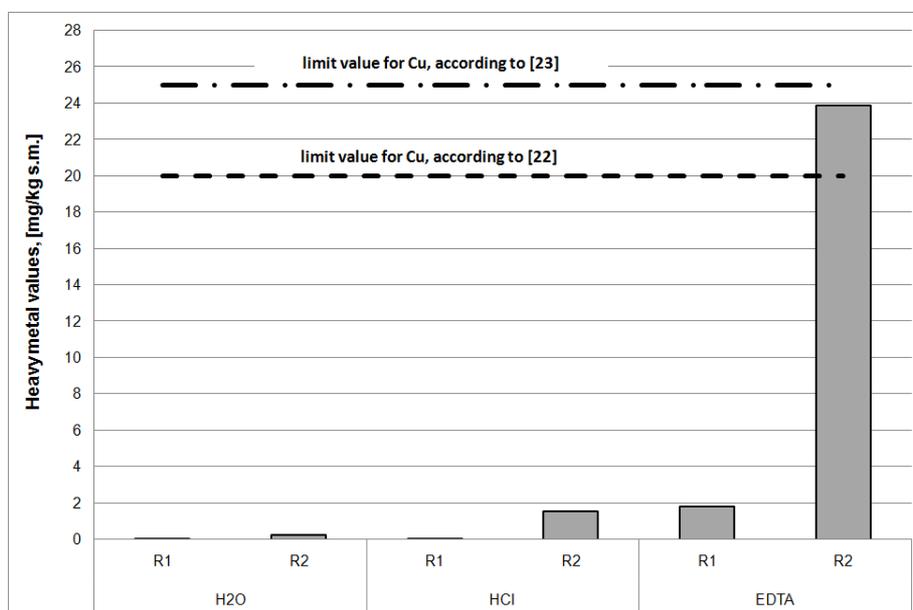


Fig. 5. Content of copper (Cu) leached from the tested hardening slurry formulae

The data presented in Table 4 and Figure 5 show an exceeding of the assumed limit value (based on the criterion [22]) for copper, in the case of the R2 formula as extracted with the EDTA solution. In other cases, the reported Cu values are below this criterion. Regarding the R2 formula, higher copper values were recorded for all the analysed extracting solutions.

The most effective extractor in the case of the tested metals was the EDTA solution, and the forms seen during the extraction with the strongly chelating EDTA solution are that potentially available when under changing environmental conditions (pH, redox potential). All the analysed metals showed an affinity to the chelating solution by dispersing from the hardening slurry into solution. The tests showed little stabilizing nature with regard to the metal bindings in the slurry, and these results confirmed those of other researchers, as the tests of EDTA extraction of the relevant heavy metals were consistent with the tests conducted in various media (seabed sediments, soils, dusts) [24, 25]. According to various authors [26, 27], the mobility of metals bound in a solid phase results from the specific properties of the metal and the composition of the water in direct contact with the medium containing the metals, as well as from the variable nature of sediments. In our work, softer metal leaching was found for the applied HCl solution, while the weakest extraction was in the case of tap water.

## Conclusion

Considering all technological and performance parameters analysed above, it should be noted that none of the tested recipes met all the criteria in relation to the hardening slurries as applied in execution of sealings of flood embankments, yet, a potential field of practical applications for the presented formulae may be found in void filling in mining damage recoveries or in site restoration (renovation). Such application of the analysed material requires verifying the efficiency of immobilisation of the heavy metals contained in the hardening slurries. The data presented in the experiment reveal the greater pollution potential of the SSTT ash of heavy metals in relation to ordinary cement. Still, on the basis of the assumed criterion of the heavy metals content in soft and very soft soils [22, 23], it was observed that the limit value (according to [22]) was exceeded only in the case of cadmium and copper when the extracting solution was EDTA. The assumed research criterion is very strict and the conditions created during the test can be found in the natural environment.

In order to improve the tightness and strength of chemical framework of the studied slurries, the share of binder should be undoubtedly increased. This binder can be cement (as in this experiment) or another type of binder, e.g. ground blast furnace slag or fly ash resulting from the combustion of hard or lignite coal (additional Coal Combustion Products).

The proposed experiment in the direction of the use of fly ash from thermal treatment of municipal sewage sludge indicates a possible way of managing an environmentally worthless waste. Moreover, the use of fly ash in construction or mining technologies will reduce extraction of natural resources (sands, gravels).

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## Streszczenie

W ostatnich latach w Polsce odnotowano dość szybki wzrost ubocznych produktów w procesie oczyszczania ścieków komunalnych - osadów ściekowych. W 2014 roku wytworzono 556,0 tys. ton suchej masy komunalnych osadów ściekowych. Jest to pochodna rozwoju cywilizacyjnego Polski i budowy nowoczesnych oczyszczalni ścieków. Zaostrzające się przepisy oraz założone, strategiczne cele gospodarki ściekowej determinują rozwój nowoczesnych metod utylizacji osadów ściekowych, tj. technik termicznych. W wyniku takich działań ilość powstałych lotnych popiołów po spalaniu komunalnych osadów ściekowych znacząco rośnie. Trwają intensywne prace nad możliwością bezpiecznego dla środowiska zagospodarowania tego typu odpadu. W artykule przedstawiono badania nad możliwością

dodawania lotnych popiołów z TPOŚ jako składnika zawiesin twardniejących. Przeprowadzono badania właściwości technologicznych i użytkowych, których celem było określenie praktycznej przydatności zastosowanych w eksperymencie zawiesin twardniejących. Wykonano również badania wymywalności metali ciężkich ze struktury zawiesin (znajdujących się w składnikach receptur zawiesin). Przeprowadzony eksperyment wykazał wymywanie niebezpiecznych związków na poziomie umożliwiającym praktyczne ich zastosowanie. Artykuł przedstawia potencjalne pole wykorzystania wspomnianego ubocznego produktu spalania.

**Słowa kluczowe:** zawiesina twardniejąca, lotny popiół z termicznego przekształcania komunalnych osadów ściekowych, przesłony przeciwfiltracyjne, metale ciężkie