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SELECTED PROPERTIES OF FLOTATION TAILINGS WASTES DEPOSITED IN THE GILÓW AND ŻELAZNY MOST WASTE RESERVOIRS REGARDING THEIR POTENTIAL ENVIRONMENTAL MANAGEMENT

WYBRANE WŁAŚCIWOŚCI ODPADÓW POFLOTACYJNYCH ZDEPONOWANYCH W ZBIORNIKACH GILÓW I ŻELAZNY MOST W ASPEKCIE MOŻLIWOŚCI ICH ZAGOSPODAROWANIA PRZYRODNICZEGO

Wastes originating from copper mining, particularly generated during the ore enrichment process constitute a considerable percentage of industrial wastes in Poland, which theoretically are supposed to provide a raw material base but remain wholly unmanaged. Wastes from copper flotation are deposited on the waste dumps which pose a significant eco-toxicological hazard. The paper discussed the results of analysis of selected post-floatation waste properties deposited in the Żelazny Most and Gilów reservoirs from the perspective of their environmental management.

Keywords: flotation tailings, copper mining, heavy metals, Phytotoxkit^{TM}, Microtox$^{®}$

Odpady pochodzące z górnictwa miedzi, szczególnie powstające w procesie wzbogacania rudy, stanowią znaczny odsetek odpadów przemysłowych w naszym kraju, które teoretycznie zasilić mają bazę surowcową i w całości pozostają niezagospodarowane. Odpady z procesu flotacji miedzi są deponowane na składowiskach i stanowią zagrożeniem ekologicznym i ekotoksykologicznym dla środowiska przyrodniczego i człowieka. Niekorzystne oddziaływanie składowisk odpadów poflotacyjnych na środowisko to przede wszystkim deformacje terenu, zanieczyszczenia gleb zanieczyszczeń roślinności metalami ciężkimi, oraz zanieczyszczenie wód powierzchniowych i podziemnych. W artykule zostały omówione niektóre właściwości fizykochemiczne oraz ekotoksykologiczne odpadów poflotacyjnych deponowanych w zbiornikach Żelazny Most i Gilów pod kątem ich biologicznego zagospodarowania. Próbki pobranych osadów, po odpowiednim przygotowaniu, poddano ocenie wybranych parametrów fizycznych, analizie chemicznej oraz serii testów ekotoksykologicznych. Właściwości ekotoksykologiczne osadów oceniono przy użyciu baterii biotestów składającej się z dwóch biotestów pracujących na pięciu organizmach (tab. 1). W teście Phytotoxkit do pomiaru toksyczności osadu wykorzystano trzy rośliny: *Sorghum saccharatum*,

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Lepidium sativum i *Sinapis alba*. Mierzonym parametrem było zahamowanie kiełkowania nasion oraz długości korzeni w badanym osadzie poflotacyjnym w porównaniu do osadu kontrolnego.

W badanych osadach poflotacyjnych dla zawartości miedzi (Gilów i Żelazny Most), ołowiu (Żelazny Most) stwierdzono znaczne przekroczenie dopuszczalnych norm dla gruntów kategorii B i C (Dz. U. z 2002 r. nr 165, poz. 1359). Większą zawartość analizowanych pierwiastków wykazano w odpadach zdeponowanych w zbiorniku Żelazny Most niż Gilów. (tab. 4). Większą rozpuszczalność badanych pierwiastków śladowych wykazano w odpadach poflotacyjnych zdeponowanych w zbiorniki Gilów. Nieznacznie niższą toksyczność wobec roślin testowych wykazały odpady poflotacyjne zbiornika Gilów w porównaniu do osadów zbiornika Żelazny Most (Rys. 3).

Słowa kluczowe: odpady poflotacyjne, górnictwo miedzi, metale ciężkie, Phytotoxkit^{TM}, Microtox$^{®}$

1. Introduction

Industrial wastes constitute over 90% of the total amount of wastes produced in Poland, of which over 80% is generated in the mining, metallurgical and power industries. Flotation tailings, which form mainly during the copper extraction process make up the highest percentage of wastes produced during the mineral deposits exploitation (Przeniosło, 2006). Polish copper mining industry deposits 100% of the wastes on landfills. Flotation tailings, despite numerous concepts of their management, are still non-utilised. Prospectively they are supposed to provide a raw material base in case of resources depletion but they may also be an arduous ballast for the environment (Azimi & Osanloo, 2011; Łuszczkiewicz, 2000).

The mining company, owned by KGHM, localized in the Legnica-Głogów Copper District, extracts over 30Mg copper ore annually, which contains about 2% of pure metal. Wastes formed during the floatation enrichment of copper ores make up about 94% of wastes generated by the company and currently deposited in the Żelazny Most reservoir, whereas until 1974 the flotation tailings wastes were stored in the Gilów reservoir.

Floatation process relies on recovery from the ore mainly copper and other minerals characterized by a high floatability. Because the content of these minerals in the ore is low, in view of their petrographic, mineral and chemical composition are similar to the spoil, but they still contain trace amounts of chemicals used during floatation process. Reservoirs of post-floatation wastes are aboveground, open facilities, situated in valley depressions, covering a huge area, which make impossible its use. Unfavourable effect of flotation tailings disposal sites on the environment involves in the first place the area deformation, soil and vegetation pollution with heavy metals, particularly cadmium and lead, pollution of surface and underground water. Pollutant migration to various components of the natural environment intensifies due to weathering, water and wind erosion (Kisielowska & Hołda, 2013; Kowalski, 2004; Chodak et al., 2006; Pieczaba et al. 2005).

"Żelazny Most" flotation tailings reservoir, with the area of 1395 ha and total capacity of 700M m^3 is currently the largest such facility in Europe and one of the largest in the world. The sedimentation basin is located in a natural valley between moraine hills, in the upper part of the Rudna River catchment. Wastes from ore enrichment plants receiving the spoil from the Lubin, Polkowice-Sieroszowice and Rudna mines are transported to the facility (Czaban et al., 2009). The "Żelazny Most" flotation tailings reservoir is constantly extended.

The "Gilów" post-floatation waste reservoir with the area of 600ha, reached the maximum damming level in June 1980 and was closed down, thus becoming the largest inactive facility of this type in Poland. Since that time it has been functioning as an emergency retentive facility for the "Żelazny Most" reservoir. Total amount of wastes deposited in the "Gilów" flotation tailings reservoir is 68M m^3. The area is subjected to water and forest reclamation activities, currently the reservoir is covered by forest and self-seeding plants (Krzaklewski, 1990). Two areas characterized by diversity in mineral composition and granulation may be distinguished in the "Gilów" reservoir area: the eastern part, where the post-mining wastes (coarse grained materials) from the "Lubin" mine are deposited and the central and western part, i.e. the area where wastes from ZG "Polkowice" are dumped. In these fine fractions are distinguished in the material granulation (Werno, 1996).

An important ecological and economic issue is a lack of management of flotation tailings, mainly due to their high heavy metal concentrations. Physicochemical and eco-toxicological properties of wastes deposited in the Gilów and Żelazny Most flotation tailings reservoirs were discussed and compared in the article.

2. Description of research method

Samples of flotation tailings were collected from the "Gilów" and "Żelazny Most" landfills using Egner's stick from the depth of 20cm in measurement points with determined coordinates (established using GPS). A total of 10 averaged samples were collected from each landfill. Following an appropriate preparation, the physical parameters of samples were determined and subsequently the samples were subjected to chemical analysis and a series of eco-toxicological tests. The above mentioned analyses were conducted in the laboratory of the Department of Agricultural and Environmental Chemistry at the Faculty of Agriculture and Economics, University of Agriculture in Krakow. In the air-dried material grain size distribution was determined with Casagrande's method in Prószyński's modification, pH in H_2O and 1 mol KCl · dm^{-3} and carbonate concentrations by Scheibler's method. Total content of selected trace elements (Zn, Cu, Ni, Cr, Pb, Cd, Fe and Mn) was assessed after sample mineralization in a muffle furnace and subsequently in a mixture of HNO_3 and $HClO_4$ (3:2), and their exchangeable forms soluble in 1 mol HCl · dm^{-3}. Extraction of metal soluble forms from flotation tailings was conducted using static method through a shaking of the wastes samples with the solution, at the sediment of solution ration 1:10 and extraction time 1 hour. Leachability of metals from the sediments was conducted according to Polish standard PN-Z-15009. *Solid wastes. Preparation of water extract.* The method relies on washing out pollutants from the examined sample using water with third degree of purity in static/quasi-dynamic conditions. Selected elements concentrations in the obtained solutions were assessed by means of ISP-AES method on JY 238 ULTRACE Jobin Von Emission apparatus. All laboratory analyses were performed in 6 replications. Obtained results were elaborated statistically and arithmetic mean were computed (Tab. 1).

TABLE 1

Total content of elements in flotation tailings

Parameters	Żelazny Most [mg · kg^{-1} d.m.]							
	Cu	Pb	Zn	Cd	Cr	Ni	Mn	Fe
Mean	2610.00	1132.00	255.60	1.00	5.28	21.19	541.00	2597.00
SD	192.22	73.71	16.49	0.09	0.66	0.53	21.91	66.39
Minimum	2470.00	1020.00	231.00	0.90	4.55	20.65	505.00	2535.00
Maximum	2840.00	1190.00	277.50	1.10	6.25	21.75	565.00	2710.00

TABLE 1. Continued

Parameters	Gilów [mg · kg^{-1} d.m.]							
	Cu	**Pb**	**Zn**	**Cd**	**Cr**	**Ni**	**Mn**	**Fe**
Mean	989.00	169.10	42.27	0.09	2.55	3.48	489.40	1207.00
SD	106.03	21.03	3.33	0.04	0.39	0.76	49.63	144.33
Minimum	815.00	138.00	36.90	0.05	2.20	2.75	410.00	1000.00
Maximum	1080.00	190.50	45.60	0.15	3.10	4.50	540.00	1315.00

Toxicity of the flotation tailings were assessed using a bio-tests composed of two bio-tests operating on four organisms (Tab. 2). Phytotoxkit™ applied for the waste toxicity assessment uses three plants: *Sorghum saccharatum*, *Lepidum sativum* and *Sinapis alba*. The measured parameter was inhibition of seed germination and root length in the tested flotation tailings in comparison with the "control sediment". The test enables a fast and automated measurement of root length using computer techniques of image analysis and provides repeatable results. Acute toxicity of flotation tailings samples was tested for *Vibrio fischeri* luminescent bacteria using M500 analyzer, following the PN-ISO11348-2:2002 procedure. Water extract was prepared by pouring 4 volumes of distilled water over 1 volume of post-floatation waste and mechanic shaking for 24 hours (Loureiro et al., 2005, Wolska & Mądrzycka, 2009). Characteristic feature of *Vibrio* bacteria is giving out a considerable portion of metabolic energy on luminescence. Luminescent bacteria generate light in visible range as an effect of their normal metabolic processes. Any change in the metabolism under the influence of toxic substance causes a change in the generated light intensity. The changes are directly proportional to biological activity of a given substance (Microtox, 1992). Luminescence measurement was conducted prior to and after the bacteria suspension incubation with the analysed sample.

TABLE 2

Bioassay battery

Trophic level	Test	Organisms	Endpoint	Type of test, time
Producers	Phytotoxkit™	*Sorghum saccharatum, Lepidium sativum, Sinapis alba*	Germintaion and growth inhibition	Chronic (3 dni)
Destruents	Microtox®*	*Vibrio fischeri*	Luminescence inhibition	Acute (15 min.)

*) PN-ISO11348-2:2008

The system of risk assessment developed by Persoone et al. (2003) was used to estimate flotation tailings toxicity:

PE (Percent toxic effect) < 20% no significant toxic effect, class I, no acute hazard
- 20% ≤ PE < 50% significant toxic effect, low toxic sample, class II, low acute hazard
- 50% ≤ PE <100% significant toxic effect, toxic sample, class III, acute hazard
- PE – 100% (single test), class IV, high acute hazard
- PE – 100% All tests, class V, very high acute hazard

The system is based on two values: position in a 5-grade risk scale and significance of the result for each class. At the first stage screening tests were conducted on the composts. The data on toxicity of flotation tailing samples were then expressed as percentage effects (PE) of growth inhibition, luminescence on the effect criterion of respective test procedure scoring system (Persoone et al. 2003). After determining the percent effect for each biotest, the sample was classified as one of five classes on according to the highest toxicity indicated by at least one test.

3. Description of the results of research and discussion

According to PN (Polish Society of Soil Science, 2008), sand fraction dominated in the flotation tailings deposited in the "Żelazny Most" and "Gilów" reservoirs (Tab. 3). Sand fraction constituted 80% of the wastes originating from the Gilów site and 89% of the wastes from the Żelazny Most site. The contents of silt fraction was about 17% in the wastes from the Gilów reservoir and 9% in wastes from Żelazny Most. Clay fraction made up, respectively 3% of granulometric composition of wastes from Gilów and 2% of the sediments from the Żelazny Most reservoir.

TABLE 3

Granulometric composition of flotation tailings

Flotation tailings	Granulometric fractions and formation [%]				Category of formation % fraction < 0.02
	1-0.1	0.1-0.02	< 0.02	Determination by PN*	
Żelazny Most	89	9	2	Sand	Very light
Gilów	80	17	3		

*) Polish Society of Soil Science, 2008

Selected physicochemical properties were presented in Tables 2 and 3. Generally a low diversification of the investigated parameters value was demonstrated as shown by low values of variation coefficient. Analyzed flotation tailings reveled close to a neutral pH (Gilów) and alkaline pH (Żelazny Most) whereas pH value ranged from 8.07 to 8.20 (Żelazny Most) and from 7.27 to 7.66 (Gilów) (Tab. 4). The wastes revealed a high content of calcium carbonate, ranging from 7.56 to 16.23% (Tab. 4).

TABLE 4

pH and content of calcium carbonate in flotation tailings

Żelazny Most	pH		$CaCO_3$	Gilów	pH		$CaCO_3$
	H_2O	KCl	%		H_2O	KCl	%
Mean	8.17	8.07	12.58	Mean	7.55	7.81	13.79
SD	0.05	0,11	0.24	SD	0.16	0.08	0.34
Minimum	8.07	7.87	7.56	Minimum	7.27	7.76	8.79
Maximum	8.20	8.14	14.67	Maximum	7.66	7.84	16.23

Buffering ability is the system capacity to counteract pH changes under the influence of acids or bases. In the presented investigations the waste buffering ability was assessed after adding to the 20 g waste: 2, 4, 6, 8 and 10 cm^3 of 0.1 $mol \cdot dm^{-3}$ HCl. All samples were completed with water to 20 cm^3 and subsequent measurement of pH value changes. Data in Figure 1 show that the waste collected from the Gilów storage site revealed stronger buffering abilities than the waste taken from the Żelazny Most post-floatation waste reservoir. A decrease in pH values in the Gilów wastes fell within the 2 to 6% range, whereas in the Żelazny Most wastes from 1 to 13%. Undoubtedly this fact was influenced by a relatively higher carbonate content in the flotation tailings collected from the Gilów than from the Żelazny Most reservoir and therefore a better effect of carbonate buffer in these wastes.

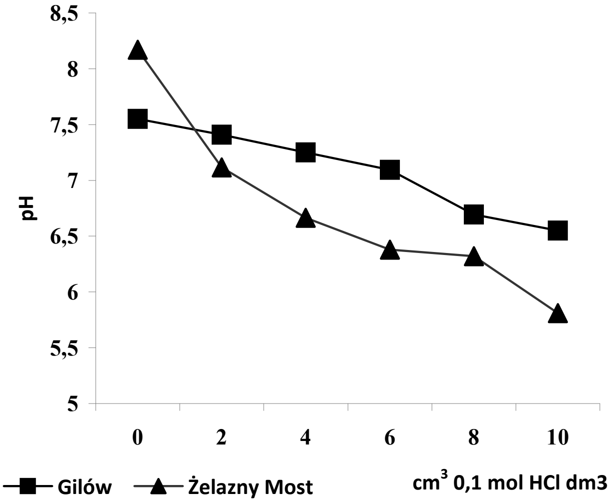

Fig. 1. Buffering abilities of flotation tailings

Total contents of trace elements in the flotation ranged as follows: 815-2840 mg Cu; 138-1190 mg Pb; 36.90-277 mg Zn; 0.05-1.10 mg Cd; 2.20-6.25 mg Cr; 2.75-21.75 mg Pb; 410-565.0 mg Mn; 1000-2710 mg $Fe \cdot kg^{-1}$ d.m. (Tab. 1).

A larger content of the analyzed elements was assessed in the wastes deposited in the Żelazny Most reservoir than in Gilów. Almost 3 times more of Cu; 7-times more of Pb; 6 times more of Zn and Ni; 11-times more of Cd and twice more of Cr and Fe and one time more of Mn were determined in the flotation from the Żelazny Most reservoir in comparison with the wastes from the Gilów reservoir (Tab. 4). Regarding their quantities in the analyzed wastes, trace elements formed the following sequence: Cu > Fe > Pb > Mn > Zn > Ni > Cr > Cd (Żelazny Most) and Fe > Cu > Mn > Pb > Zn > Ni > Cr > Cd (Gilów) (Tab. 4). It is worth mentioning that a bigger diversification in the trace element content was determined in the wastes from the Gilów than the Żelazny Most reservoir. Variation coefficient ranged from 8 to 46%. (Tab. 1).

An important parameter enabling an assessment of elements bioavailability is determining the content of their readily soluble forms. Trace element solubility in 1 mol $HCl \cdot dm^{-3}$ in the post-floatation wastes from the Żelazny Most reservoir reached the highest maximum value for Mn – 86%, then for Cu (36%) >Pb (26%) > Fe (24%) > Cr (21%) > Cd (17%) > Zn (13%) and the minimum level for Ni not exceeding 4% of their total content (Fig. 2). In the waste from the Gilów waste reservoir, solubility of trace elements was from 13% for Cd to 99% for Mn in relation to their total content and formed the following sequence: Mn > Pb (89%) > Fe (50%) > Cu (45%) > Ni (39%) > Cr (35%) > Zn (16%) > Cd (13%) (Fig. 2). A better solubility of the analyzed trace elements was revealed in the post-floatation waste deposited in the Gilów reservoir. Numerous investigations have shown that waste reaction may affect heavy metals mobility in the environment (Jasiewicz, Antonkiewicz 2009, Kaszubkiewicz et al. 2007). Metals may pass into less readily soluble forms at higher pH values. Assessed alkaline reaction of flotation tailings in the Żelazny Most reservoir may influence a diminished solubility of a majority of analyzed elements. In these conditions the are precipitated sediments CO_3^{2-}, OH^- and hydrosols. Therefore, both lower content of trace elements and better bioavailability and diversification in the flotation tailings in the Gilów reservoir in comparison with Żelazny Most reservoir undoubtedly results from reclamation processes initiated in this dumping site and their gradual activating under the influence of life process of plants.

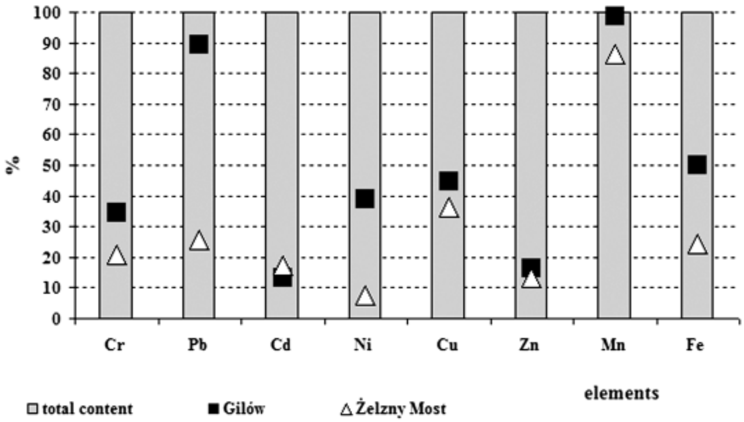

Fig. 2. Percentage of forms soluble in 1 mol dm^{-3} HCl trace elements in the total content in flotation tailings

An assessment of the waste eco-toxicological properties focused on the measurement of inhibition of germinating process and root growth of the test plants: *Sorghum saccharatum*, *Lepidium sativum* and *Sinapis alba*. Among the test plants species *Sinapis alba* proved the most sensitive to phytotoxic properties of the analyzed flotation tailings, *Sorghum saccharatum* was less sensitive and *Lepidium sativum* the least (Fig. 3).

The degree of *Sinapis alba* seed germination inhibition was from 6 to 13%, for *Sorghum saccharatum* seeds from 5% to 9%, whereas for *Lepidium sativum* only between 4 and 6%. The degree of young roots growth inhibition was between 4 and 6% for *Lepidium sativum,* from 23 to 34% for *Sorghum saccharatum* and between 58 and 63% for *Sinapis alba*. The flotation tailings from the Gilów reservoir revealed a slightly lower toxicity for the test plants in comparison with the Żelazny Most waste (Fig. 3). In the test, almost all water extracts prepared from the flotation tailings from the Żelazny Most reservoir caused almost 10% decrease in the luminescence of *Vibrio fischeri* bacteria, however none of the examined samples caused a percentage effect – PE < 20%. On the other hand, the water extracts from flotation tailings from the Gilów reservoir revealed a higher toxicity for *Vibrio fischeri* bacteria – reaching almost 50%, which

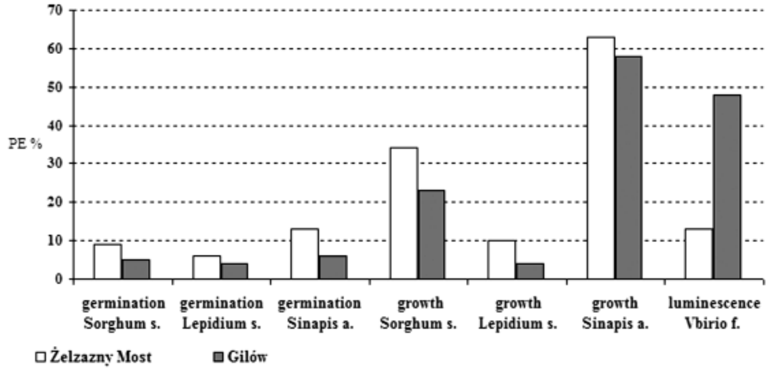

Fig. 3. Toxicity of flotation tailings

testifies a small hazard to the environment (20% ≤ PE < 50%) (Persone et al., 2003). *Vibrio fischeri* proved an organism more sensitive to pollutants in the water extract prepared from the Gilów post-floatation reservoir (Fig. 3). It is due to the fact that metals present in the Żelazny Most flotation tailings are characterized by a lower leachibility in H_2O in comparison with the flotation tailings from Gilów (Tab. 5). Heavy metal concentrations determined in water extracts of the waste from the Żelazny Most reservoir were very low, reaching from 0 (Cu) to 2% (Cd) in relation to their total content. However, metal leachability from the Gilów flotation tailings fluctuated from 0.11% (Pb) to 33% (Cd) of their total content.

TABLE 5

Percentage of forms soluble in H_2O trace elements in the total content in flotation tailings

Flotation tailings	Heavy metals leachability degree [%]					
	Cu	Pb	Zn	Cd	Cr	Ni
Żelazny Most	0	0,03	0,02	2,00	0,11	0,04
Gilów	1,26	0,11	0,90	33,00	2,75	1,15

Unfavourable impact of flotation tailings on the environment manifests itself in the first place as surface and undergroundwater pollution, dustiness due to aeolian erosion of flotation tailings containing heavy metals and infiltration of supernatants. Moreover, unmanaged flotation tailings cause a necessity of their storage and therefore exclusion from use of large area segments. Emission of dusts containing heavy metals leads to pollution of soils, which negatively affects vegetation development in the vicinity of the objects of this type (Angełow et al., 2000; Koszubkiewicz et al., 2006; Rybak et al.,2008). Therefore, it is important to identify physical, chemical and eco-toxicological properties of flotation tailings for the rational environmental management (Boularbah et al., 2005) . Kaszubkiewicz et al. (2007); Chodak et al. (2005); Kaszubkiewicz and Kawełko (2006) were among those researchers who investigated potential management of flotation tailings. Eco-toxicological research conducted using biotest revealed that *Sinapis alba* was the most sensitive to chemicals contained in the wastes. Among the test organisms used in the study in Sinapis alba was observed largest toxic effect (degree of young roots growth inhibition was 58-63%) (Fig. 3). *Sinapis alba* may be used for bioindication of reclaimed coal mining waste dumps. Moreover, a better sensitivity of a test based on root growth was determined. Other authors demonstrated a similar dependence claiming that germination ability is the indicator which is to low degree affected by heavy metal presence, oil derivatives, composts, and some polymers (Adam & Duncan, 2002; Baran et al., 2008; Smreczak & Maliszewska-Kordybach, 2003; An, 2004; Płaza et al., 2005; Kopeć et al., 2013). No highly or very highly hazardous samples (class IV or V) (PE = 100%) were found in the presented investigations. Analyzed post-floatation sediments characterized by acute hazard constituted class III.

4. Conclusions

One of crucial problems emerging during reclamation and management of the analyzed flotation tailings is high content of some heavy metals. In the studied flotation tailings, considerably exceeded contents of copper for B and C category grounds were registered (Gilów and Żelazny Most) and lead (Żelazny Most) (Journal of Low 2002, No. 165, item 1359). High pH of these wastes causes that most metals is unavailable to plants. Chemical pollutants which are readily soluble become activated by rainfall and penetrate into the soils. Weathering processes may cause mobilisation of further amounts of chemical components which translocate into the soil leading to a concentration of hydrogen ions in the washed wastes and therefore increased acidification. Acidification favours mobilisation of more chemicals as has been demonstrated in the presented research (Fig. 2). In result of waste acidification the content of soluble forms increases. This mechanism evidences a considerable mobility of heavy metals on these lanfills and serious hazard to the environment.

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