Role of Mineralogical Identification in Enrichment Processes of Waste Materials

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Summary

Article presents the results of research on the recovery of metals from the slimes originated from the current production of ZGH Boleslaw as well as deposited for many years in settling ponds originated from the operation of the abandoned Zinc Electrolysis Plant in Szopienice. These slimes contain high contents of various metals, including also precious metals, e.g. silver. Publication highlights the importance of identifying apart from granulometric and chemical composition also mineral composition of these wastes. The identification of mineral composition of the investigated slimes was carried out using the Jeol JXA 8230 Electron Probe Microanalyzer (EPMA).

On the basis of the performed waste material characteristics and occurring useful minerals an enrichment method was developed. Technological tests on the recovery of silver, zinc and lead were conducted using flotation process. For the recovery of lead occurring in the form of lead sulphate a hydrometallurgical treatment of slime before flotation process was carried out with the aim of transition of one part of lead sulphate into easier to float lead sulphide.

Keywords: mineralogical identification, microanalyzer, flotation, slimes,flotation reagent

Introduction

Currently, becoming increasingly important is a possibility of recovery of metals and other useful minerals from waste materials resulting from exploitation, enrichment and processing (e.g. metallurgical, chemical) mineral ores. Transformations due to the metallurgical smelting, acid action, long-lasting dumping, etc. influence the complicated phase composition of minerals occurring in the waste materials. It is characterized by the presence of diversified replacements of elements in the lattice structure, which are unencountered under natural conditions. Therefore, the waste materials should be submitted to the identification investigations before the reprocessing, with a particular attention given to the investigation of mineral composition. The mineralogical studies of the waste materials should be carried out mainly from the standpoint of the needs for their enrichment and technology being developed. Therefore, a wide range of qualitative and quantitative identification of the occurring components, together with determination of their grain size and forms of occurrence should be carried out.

The identification of mineral composition of the waste permits to conclude that there is a possibility of their economic use, and therefore it should be one of the first stages on the way of studies into their enrichment. Chemical and granulometric analysis does not give a complete characteristics of the waste. Very efficient in obtaining a full material image is X-ray microanalysis method [Monaco et al., 2007]. The X-ray analysis in microareas allows both identification of basic minerals occurring in the investigated specimen and determination of forms of occurrence of the accompanied elements.

Article presents the results of investigations carried out by means of X-ray microanalysis of slimes after leaching of roasted zinc blende originated from the current production of ZGH Boleslaw as well as deposited for many years in settling ponds originated from the operation of the abandoned Zinc Electrolysis Plant in Szopienice. These slimes contain high contents of various metals, including also precious metals, e.g. silver.

The results of mineralogical studies of the slimes help with the effective choice of conditions for their processing, with the aim of obtaining the most advantageous indices of recovery of the useful metals.

In the article, the results of enrichment process of these materials and hydrometallurgical treatment for the recovery of interesting metals, are also described.

Description of the investigated material

Zinc slimes were submitted to the chemical and granulometric analyses. The granulometric analysis was carried out by means of a classical sieve analysis, whereas the grain size below 45μm was measured using Malvern Mastersizer 2000 particle size analyzer. Results of the granulometric analysis are shown in Tab. 1.

The chemical composition of the delivered slimes was as follows:

- ZGH Boleslaw: about 20% Zn, 250ppm Ag, 11% Pb and over 38% Fe
- HMN Szopienice: about 17.5% Zn, 380-400ppm Ag, about 10.5% Pb and about 32% Fe

Methodology of the mineralogical studies

The studies of the slimes after zinc electrolysis were performed with application of X-ray microanalyzer (EPMA) JXA 8230 by Jeol equipped with three crystal spectrometers, semiconductor detector with energy analyzer and EBSD camera (electron backscatter diffraction).

The test samples were in the forms of metallographic specimens prepared by polishing of slime particles with application of diamond pastes after their earlier immersion in the conductive resin. The oxide samples are poor conductors of both electricity and heat, what under the influence of electron beam results in a shift of weakly bound positive

ions towards the middle of the sample and in the evaporation of some elements. In order to improve the conductivity and eliminate these effects the samples were sprayed with thin gold coating.

The imaging of the area selected for the studies was performed with both secondary electrons (SEI – Secondary Electron Imaging) and backscattered electron composition, where contrast depends on average atomic number. In order to determine changes of elements concentration the distribution maps were examined by wavelength dispersion spectroscopy (WDS). The points selected in the electron images were subjected to qualitative and quantitative X-ray microanalyses using both energy dispersive spectroscopy (EDS), characterized by short analysis time, and also wavelength dispersion spectroscopy. The wavelength dispersive technique is characterized by significantly better analytical capabilities when compared with energy dispersion method (Pownceby et al., 2007). The resolving power (possibility to distinguish two adjacent lines in the spectrum) of the crystal spectrometers is more than ten times better than the resolving power of the energy dispersive spectrometers. In case the lines of different elements have equal energy (e.g. SKα and PbMα) the proper qualitative analysis may be performed by wavelength dispersion only. WDS technique is also characterized by significantly better element detection limits than EDS method (Szummer et al.,1994). Due to statistical character of the measurement the standard deviation of the single count is taken for the measurement uncertainty, the percentage relative uncertainty is in the range from 0.5% to about 5% depending on the element concentration. The following spectrally pure elements: Mn, Fe, Cu, Zn, Ag, Cd and compounds: Al_2O_3, $CaSiO_3$, FeS_2, PbTe, InAs were used as the reference materials.

Results of the mineralogical studies

The first stage of studies of the slimes after zinc electrolysis was identification of the basic compounds contained in both waste materials. Figure 1 presents typical image of surface of the slime sample after zinc electrolysis from ZGH Boleslaw with the marked analysis points. On the basis of qualitative analyses (Fig. 2) and results of the measurement of chemical composition presented in Table 2 the presence of sphalerite, franklinite, anglesite and gypsum grains in the slime was observed.

In order to determine the occurrence forms of silver and lead the element distribution maps in selected areas of the sample were examined. In the areas of increased concentration of the particular element the qualitative and quantitative X-ray microanalyses were carried out. From the element distribution maps presented in Fig. 3 and 4 results that silver with copper surround some of sphalerite grains. The average size of this kind of grains (Fig. 5) is about 50µm, whereas the rim thickness does not exceed 5µm (Fig. 6). The point analysis from the areas of higher Cu and Ag concentration (Fig. 7, Tab. 3) indicates partial substitution of zinc atoms by copper and silver atoms in the sphalerite. Maximum content of copper reaches 35.7wt.%, silver concentration is significantly lower and it is in the range from 3.3% to 5.1%.

On the basis of the element distribution maps presented in Fig. 8 it can be found that lead occurs in the small grains (maximum diameter 2-3µm). The qualitative and quantitative analyses (Fig. 9, 10, Tab. 4) indicate that this is an anglesite (lead sulphate). No presence of silver was discovered in it. In neighbourhood of anglesite grains the oxide compounds of silicon, aluminium, zinc and iron occur.

Tab. 1 Grain size analysis of the zinc slimes

Tab. 1 Analiza wielkości ziarna szlamów cynkowych

Grain class [mm]	ZGH Boleslaw	Szopienice smelter
	Yield [%]	Yield [%]
0.50 - 0.30	-	12.48
0.30 - 0.20	0.70	1.37
0.20 - 0.10	1.51	1.91
0.10 - 0.075	2.43	1.46
0.075 - 0.045	7.25	3.00
0.045 - 0.036	6.76	13.44
0.036 - 0.025	10.13	11.77
0.025 - 0.015	18.21	17.06
0.015 - 0.005	28.00	21.06
-0.005	25.01	16.45
Σ	100.00	100.00

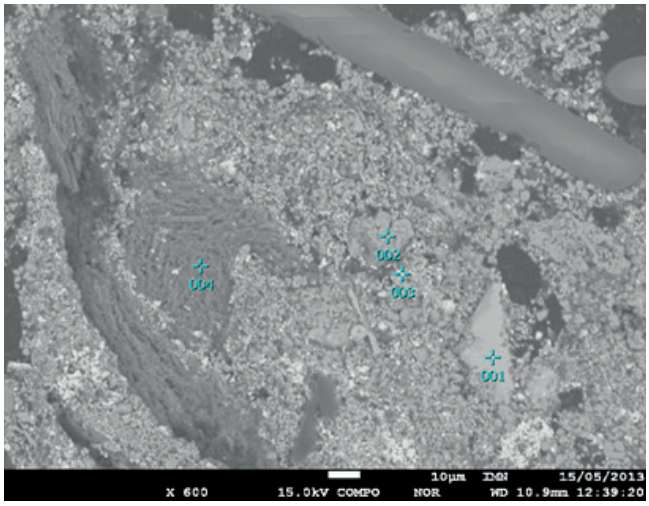


Fig. 1 Electron image (composition) of surface of the slime sample from ZGH Boleslaw with the selected analysis points: 1 - sphalerite, 2 – franklinite, 3 – anglesite, 4 – gypsum

Rys. 1 Obraz elektronowy (skład) powierzchni próbki szlamu z ZGH Bolesław z analizą: 1 - sfalerytu 2 - franklinitu 3 - anglezytu 4 – gipsu

Results of the technological tests

Studies into a possibility of metal recovery from slimes after acid leaching of zinc blende were mainly conducted in terms of silver recovery and later zinc and lead. When developing a method for the recovery of silver, its mineralogical forms were taken into account. In view of the fact that silver occurred in both slimes in the form of complex sulphide structures (Ag-Cu-S, Ag-Cu-S) as thin layers (rims) on the sphalerite, the flotation as the basic method for the silver recovery was applied. The flotation flowsheet is shown in Fig. 11. The flotation reagents were the same as used in flotation of the sulphide minerals. The following reagents were used:

- collecting reagents – amyl xanthate, (X-23)-reagent of thiocarbamate type and (LIB)-reagent of diethyl dithiophosphate type
- frothing reagent – Corflot-mixture of polyglycols
- reagents depressing iron – (ON)-apolar reagent and $CuSO_4$ -reagent used for activation of sphalerite

Results of flotation of the slimes from ZGH Boleslaw and Szopienice smelter are shown in Tab. 5, 6 and Fig. 12.

In the flotation of slimes from ZGH Boleslaw, in order to depress the iron (pyrite), an apolar reagent ON was added. The addition of ON resulted in a slight decrease of iron content in the concentrate and an increase of silver content. There was no effect of the addition of $CuSO_4$ on the activation of sphalerite flotation. One of the reasons can be the mentioned cover of sphalerite with a thin layer of silver.

As a result, in this flotation a summary Zn-Ag bulk concentrate was obtained (K1+K2 or K1+K2+K3) with about 30% Zn and about 800 to more than 900ppm Ag content at recovery 30-32% for Zn and 77% to more than 81% for Ag.

In the case of flotation of slimes from Szopienice smelter, a summary concentrate (K1-K3) with 2400 ppm Ag content at recovery over 85.5% was obtained.

Because the lead in the form of sulphate was not recovered by means of flotation method, it was decided in the subsequent series of tests to precede the flotation by a appropriate preparation of material through its two-stage hydrometallurgical treatment [Wójtowicz at al., 1997].

A leaching method was applied for the slimes originated from Szopienice smelter. It consisted in the first stage in

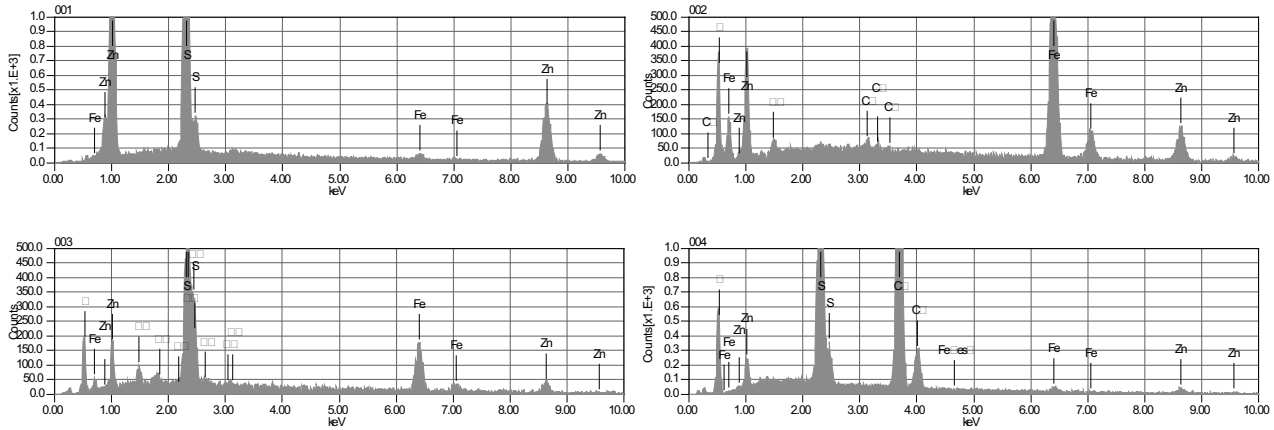


Fig. 2 Qualitative analyses (energy spectrum) of the points selected in the Fig. 1

Rys. 2 Analizy jakościowe (widma energii) punktów zaznaczonych na Rys. 1

Tab. 2 Results of the chemical composition measurements of the points selected in Fig. 1

Tab. 2 Wyniki pomiarów składu chemicznego elementów wyselekcjonowanych w Rys. 1

Element	Grain 1 sphalerite		Grain 2 franklinite		Grain 3 anglesite		Grain 4 gypsum	
	wt. %	at. %	wt. %	at. %	wt. %	at. %	wt. %	at. %
O	-	-	21.9	50.7	22.9	59.8	46.2	66.8
Al	-	-	0.7	1.0	1.3	2.1	-	-
S	33.6	50.7	-	-	5.9	7.7	22.3	16.1
Ca	-	-	-	-	-	-	26.3	15.2
Fe	1.8	1.6	49.9	33.1	19.4	14.5	1.2	0.5
Zn	64.5	47.7	25.8	14.6	13.2	8.4	4.2	1.5
Cd	-	-	1.8	0.6	-	-	-	-
Pb	-	-	-	-	37.2	7.5	-	-

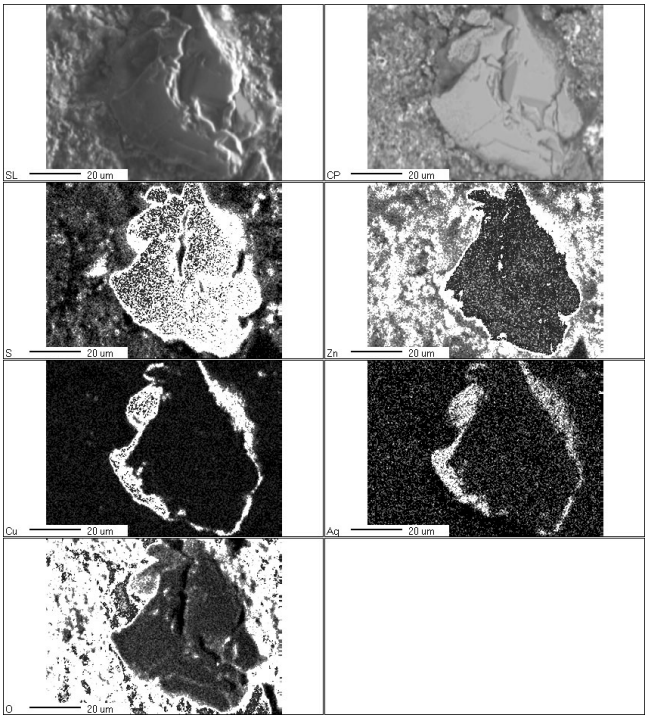


Fig. 3 Electron images of the sphalerite grains and element distribution maps (S, Zn, Cu, Ag, O, respectively) of the zinc slime sample from ZGH Boleslaw

Rys. 3 Obraz elektronowy ziaren sfalerytu i mapa rozkładu pierwiastków (odpowiednio S, Zn, Cu, Ag, O) w próbce szlamu cynkowego z ZGH Bolesław

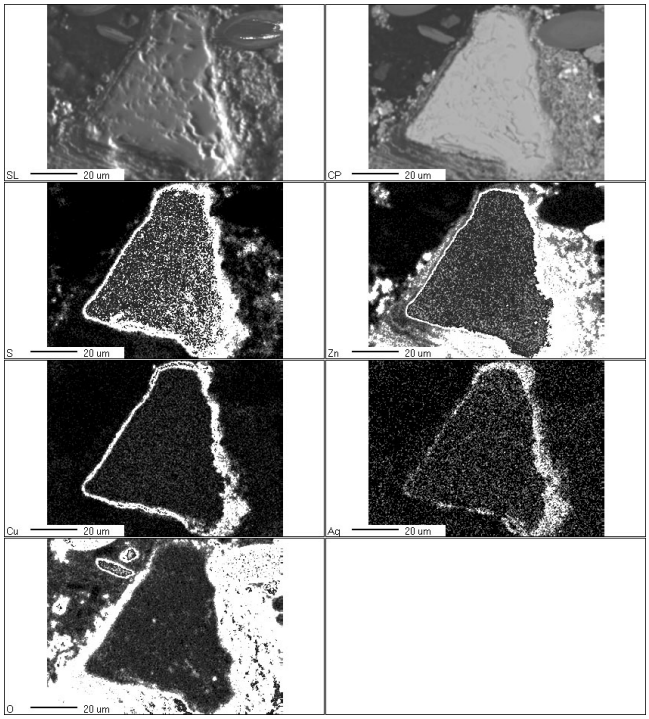


Fig. 4 Electron images of the sphalerite grains and element distribution maps (S, Zn, Cu, Ag, O, respectively) of the zinc slime sample from Szopienice smelter

Rys. 4 Obraz elektronowy ziaren sfalerytu i mapa rozkładu pierwiastków (odpowiednio S, Zn, Cu, Ag, O) w próbce szlamu cynkowego z huty Szopienice

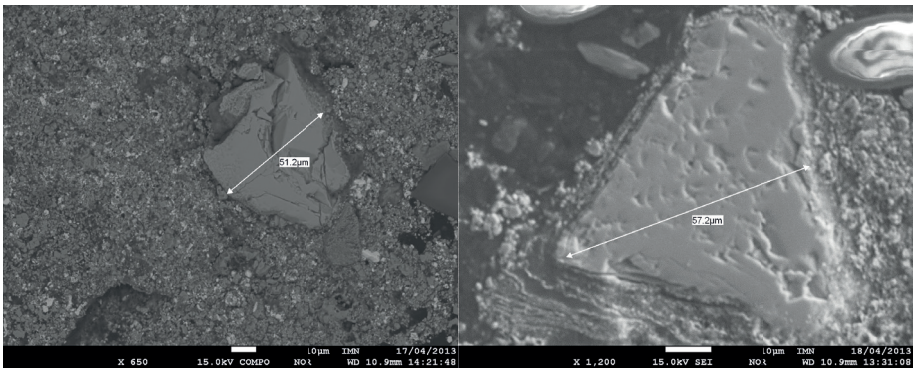


Fig. 5 Electron images of the sphalerite grains with size measurement

Rys. 5 Obraz elektronowy ziaren sfalerytu z pomiarem ich wielkości

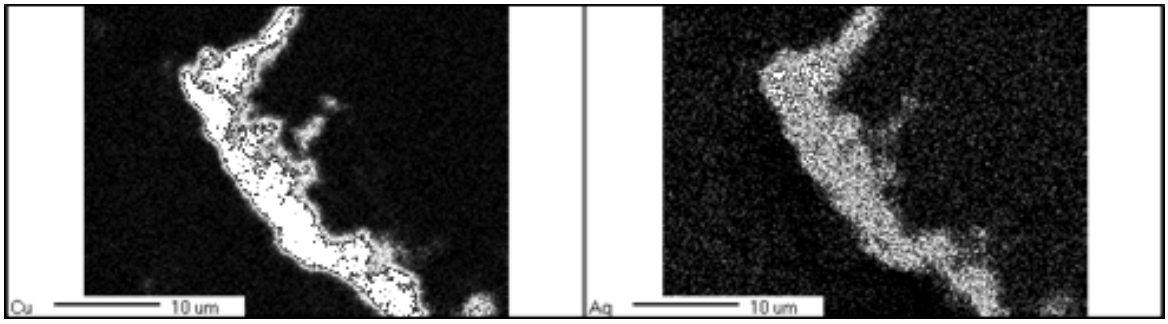


Fig. 6 Distribution maps of copper and silver, magnification 3000x

Rys. 6 Mapa rozkładu miedzi i srebra, powiększenie 3000x

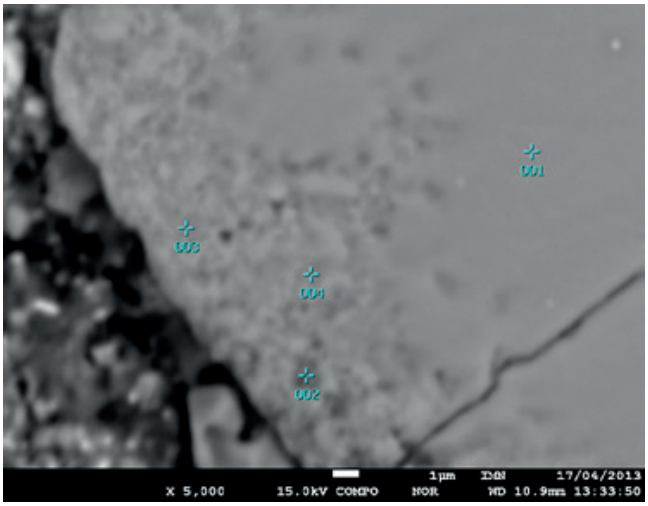


Fig. 7 Electron image (composition) of the sphalerite grain with the selected analysis points, magnification 5000x

Rys. 7 Obraz elektronowy (składu) ziaren sfalerytu z analizą wybranych punktów, powiększenie 5000x

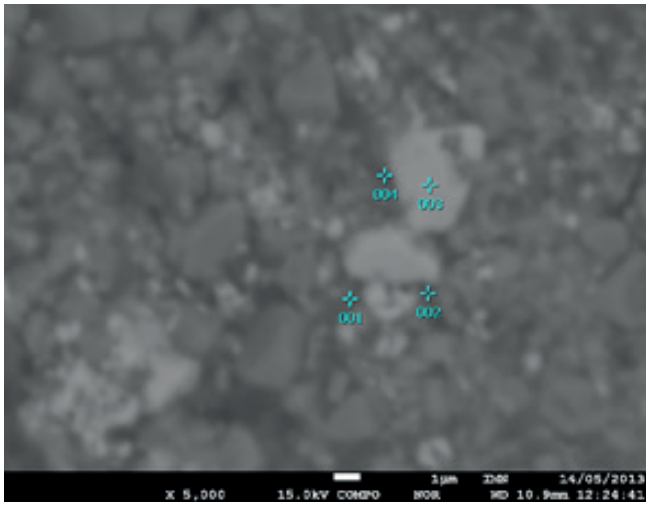


Fig. 9 Electron image (composition) of anglesite grains with the selected analysis points, magnification 5000x

Rys. 9 Obraz elektronowy (składu) ziaren anglezytu z analizą wybranych punktów, powiększenie 5000x

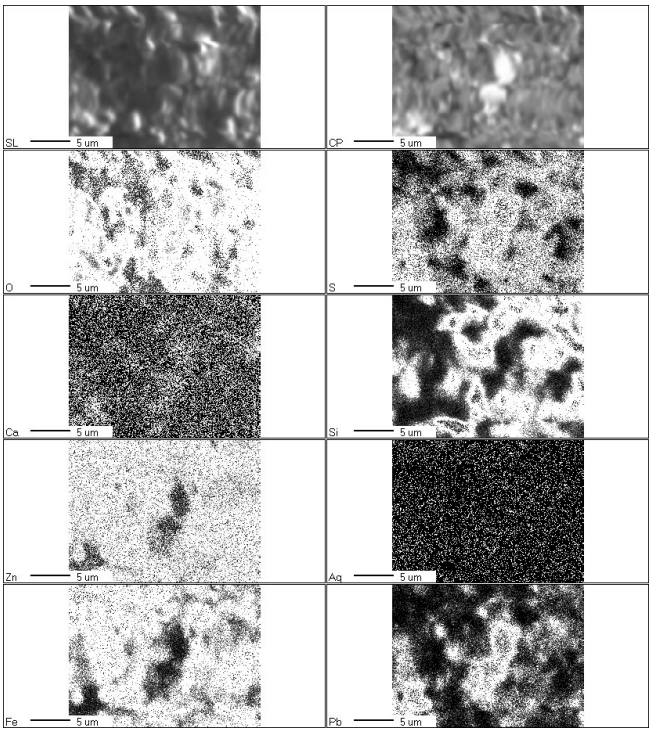
Tab. 3 Results of the chemical composition measurements of the points selected in Fig. 7

Tab. 3 Wyniki pomiarów składu chemicznego wybranych punktów z Rys. 7

Element	Point 1		Point 2		Point 3		Point 4	
	wt. %	at. %	wt. %	at. %	wt. %	at. %	wt. %	at. %
S	32.9	49.9	32.8	50.0	32.4	49.5	32.8	49.9
Fe	1.6	1.4	2.1	1.9	1.8	1.6	1.9	1.7
Zn	65.6	48.8	46.2	28.8	21.7	8.4	26.2	19.6
Cu	-	-	13.7	32.7	32.7	25.2	35.7	27.4
Ag	-	-	5.1	2.3	4.3	2.0	3.3	1.5

Fig. 8 Electron images of anglesite grains and element distribution maps (O, S, Ca, Si, Zn, Cu, Fe, Pb, respectively)

Rys. 8 Obraz elektronowy ziaren anglezytu i mapa rozkładu pierwiastków (odpowiednio S, Ca, Si, Zn, Cu, Fe, Pb)



Tab. 4 Results of the chemical composition measurements of the points selected in Fig. 9

Tab. 4 Wyniki pomiarów składu chemicznego wybranych punktów z Rys. 9

Element	Point 1		Point 2		Point 3		Point 4	
	wt. %	at. %	wt. %	at. %	wt. %	at. %	wt. %	at. %
O	27.0	58.7	22.2	54.6	23.7	67.8	26.0	55.5
Al	1.5	1.9	1.7	2.5	-	-	3.9	5.0
Si	14.1	17.4	6.1	8.5	0.1	0.2	8.2	10.0
S	6.5	7.1	7.3	9.0	10.2	14.6	6.6	7.1
Fe	7.6	4.7	15.5	11.0	2.7	2.2	13.1	8.0
Zn	7.8	4.2	13.1	7.9	2.5	1.7	20.9	11.0
Pb	35.5	6.0	34.1	6.5	60.7	13.4	21.4	3.5

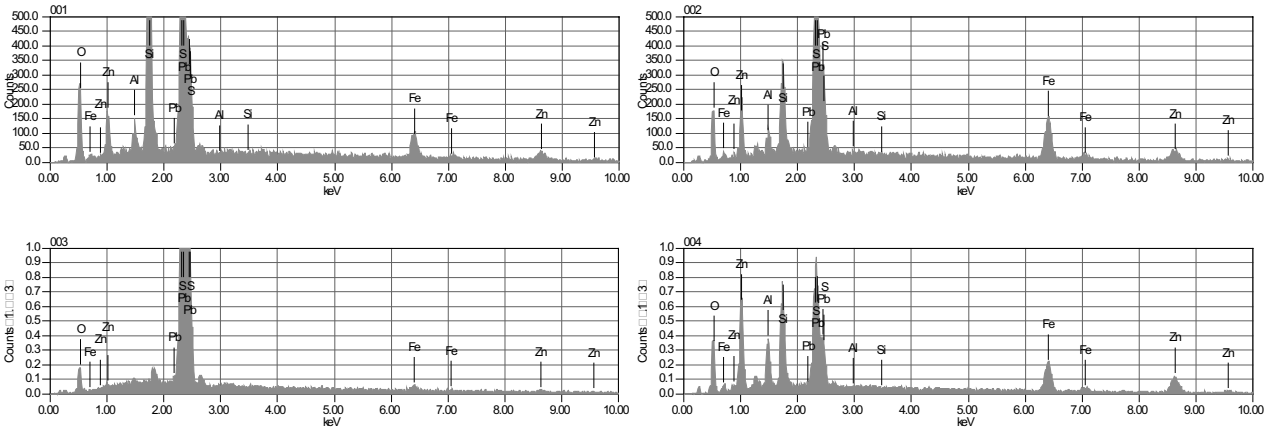


Fig. 10 Qualitative analyses (energy spectrum) of the points selected in the Fig. 9

Rys. 10 Analizy jakościowe (widma energii) punktów zaznaczonych na Rys. 9

removing remnants of the soluble zinc by acid leaching and digesting a considerable part of iron, and in the second stage in introducing a sulphide sulphur into the material in order to build it into the structure of lead minerals (surface sulphidisation).

This sulphidisation caused transition of one part of the lead sulphate into an easier to float lead sulphide (galena). It was also expected that, because a part of silver as the mineralogical investigations have revealed is associated with lead, it should improve the results of silver flotation. The results of slime flotation after hydrometallurgical treatment are shown in Tab. 6.

As a result of this flotation an improvement of the lead flotation was obtained, both in the scope of quality and recovery (increase of the lead recovery from 23% to 67%), however a satisfying result in the scope of silver flotation was not obtained.

Summary

The performed investigations of slimes are one of the examples indicating the importance of application of X-ray microanalysis method for the mineral identification of waste materials. This technique allows to detect elements with 0.01 wt.% content (for the method with wavelength dispersion – WDS) in areas of 1 μm3 volume. Moreover, it gives a possibility of quantitative analysis based on reference materials of the measured elements and examination of the changes of concentration on a chosen surface of the preparation. From the obtained results it can be concluded about connections in which the individual elements can be found and size of individual components. It matters, especially in the case of mineral waste materials which are characterized by a rich mineral composition. Moreover, they are characterized by a strong transformation due to high temperature, weathering processes, etc. what favours the formation of many secondary minerals with untypical mineral composition not having their counterparts in nature.

The described studies constitute a valuable supplement to the granulometric, chemical and phase investigations. On their basis it can be concluded about possibilities of an utilisation of the mineral waste materials and their environmentally safe disposal. They also largely facilitate development of methods for recovery of metals or other components.

In the case of slimes from ZGH Boleslaw and Szopi-

Tab. 5 Conditions and results of flotation of slimes from ZGH Boleslaw

Tab. 5 Warunki i wyniki flotacji szlamu z ZGH Bolesław

Series	Conditions of flotation	Product	PRODUCT								CONCENTRATE								
			Yield [%]	Content				Recovery [%]				Yield [%]	Content				Recovery [%]			
				Zn [%]	Pb [%]	Fe [%]	Ag [ppm]	Zn	Pb	Fe	Ag		Zn [%]	Pb [%]	Fe [%]	Ag [ppm]	Zn	Pb	Fe	Ag
1	Amyl xanthate 50 [g/t] Corflot 10 [g/t] X23 30 [g/t]	C1	6.94	35.70	8.35	24.00	1430	12.02	5.21	4.36	38.53	6.94	35.70	8.35	24.00	1430	12.02	5.21	4.36	38.53
		C2	9.64	29.20	10.20	30.60	810	13.66	8.85	7.72	30.33	16.58	31.92	9.43	27.84	1069	25.68	14.06	12.07	68.86
		C3	5.51	25.60	10.70	31.70	516	6.84	5.30	4.57	11.04	22.09	30.34	9.74	28.80	931	32.52	19.36	16.64	79.90
		C4	4.11	18.80	13.40	39.20	272	3.75	4.95	4.21	4.34	26.20	28.53	10.32	30.43	828	36.27	24.31	20.85	84.24
		T	73.8	17.80	11.40	41.00	55	63.73	75.69	79.15	15.76	100.00	20.61	11.12	38.23	258	100.00	100.00	100.00	100.00
		F	100.00	20.61	11.12	38.23	258	100.00	100.00	100.00	100.00									
2	Amyl xanthate 100 [g/t] Corflot 35 [g/t] $CuSO_4$ 350 [g/t] X23 50 [g/t]	C1	13.77	32.20	9.41	25.90	1050	21.50	11.76	9.43	57.27	13.77	32.20	9.41	25.90	1050	21.50	11.76	9.43	57.27
		C2	9.90	23.80	12.10	32.80	514	11.42	10.87	8.58	20.15	23.67	28.69	10.53	28.79	826	32.92	22.62	18.02	77.42
		C3	4.00	19.40	13.10	36.40	387	3.76	4.75	3.85	6.13	27.67	27.35	10.91	29.89	762	36.69	27.38	21.87	83.55
		C4	9.13	18.40	12.90	37.00	151	8.15	10.69	8.94	5.46	36.80	25.13	11.40	31.65	611	44.83	38.06	30.80	89.01
		T	63.20	18.00	10.80	41.40	44	55.17	61.94	69.2	10.99	100.00	20.62	11.02	37.81	252	100.00	100.00	100.00	100.00
		F	100.00	20.62	11.02	37.81	252	100.00	100.00	100.00	100.00									
3	Amyl xanthate 75 [g/t] Corflot 25 [g/t] $CuSO_4$ 350 [g/t] X23 30 [g/t] ON 90[g/t]	C1	3.77	41.50	7.01	16.20	1641	7.68	2.38	1.60	26.91	3.77	41.50	7.01	16.20	1641	7.68	2.38	1.60	26.91
		C2	6.65	36.40	9.02	20.90	1184	11.87	5.40	3.65	34.21	10.42	38.25	8.29	19.20	1349	19.54	7.78	5.25	61.11
		C3	8.68	23.30	10.80	34.60	520	9.91	8.44	7.88	19.60	19.10	31.46	9.43	26.20	973	29.46	16.22	13.13	80.72
		C4	6.08	18.80	12.70	38.60	171	5.61	6.96	6.16	4.52	25.18	28.40	10.22	29.19	779	35.06	23.18	19.29	85.24
		T	74.82	17.70	11.40	41.10	45	64.94	76.82	80.71	14.76	100.00	20.39	11.10	38.10	230	100.00	100.00	100.00	100.00
		F	100.00	20.39	11.10	38.10	230	100.00	100.00	100.00	100.00									

Tab. 6 Results of slime flotation from Szopienice smelter

Tab. 6 Wyniki flotacji szlamu z huty Szopienice

Slime	Conditions of flotation	Pr.	Yield		CONTENT		RECOVERY		CONTENT		RECOVERY		CONTENT		RECOVERY	
			γ [%]	Σγ [%]	Zn [%]	Σ Zn [%]	Zn [%]	$\Sigma\varepsilon_{Zn}$ [%]	Pb [%]	ΣεPb [%]	Pb [%]	$\Sigma\varepsilon_{Pb}$ [%]	Ag [ppm]	ΣεAg [ppm]	Ag [%]	$\Sigma\varepsilon_{Ag}$ [%]
raw	LIB 550 [g/t] Corflot 110 [g/t] pH - 6.06 - 6.24	ΣC	14.26	14.26	36.60	36.60	30.07	30.07	7.48	7.48	10.33	10.33	2400	2400	85.55	85.55
		M	10.06	24.32	15.30	27.79	8.87	38.94	13.80	10.10	13.45	23.78	78	1439	1.96	87.51
		T	75.68	100.00	14.00	17.35	61.06	100.00	10.40	10.33	76.22	100.00	66	400	12.49	100.00
		F	100.00		17.35				10.33				400			
after leaching	LIB 400 [g/t] Corflot 40 [g/t] pH - 6.12	ΣC	37.77	37.77	23.10	23.10	61.22	61.22	14.50	14.50	49.01	49.01	1236	1236	86.86	86.86
		M	13.84	51.61	13.70	20.58	13.31	74.53	14.60	14.53	18.08	67.09	165	949	4.25	91.11
		T	48.39	100.00	7.50	14.25	25.47	100.00	7.60	11.17	32.91	100.00	99	537	8.89	100.00
		F	100.00		14.25				11.17				537			

Fig. 11 Flowsheet of laboratory tests of slime flotation

Rys. 11 Schemat testów laboratoryjnych flotacji szlamu

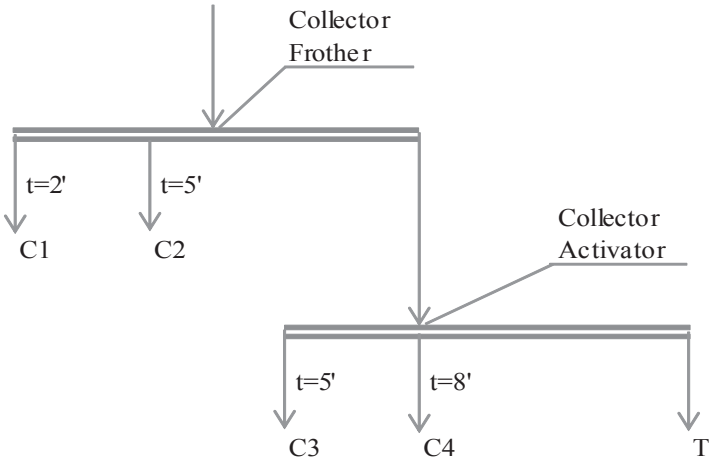
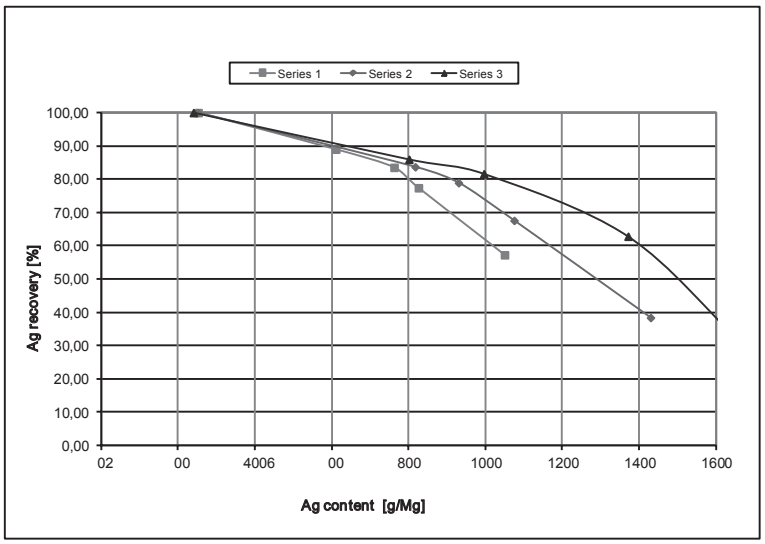


Fig. 12 Upgrading curves of silver in froth product for slimes from ZGH Boleslaw

Rys. 12 Rosnące krzywe wzbogacalności srebra w procesie flotacji szlamów z ZGH Bolesław



enice smelter, the studies were carried out in order to identify silver, but also zinc and lead.

For the recovery of lead occurring in the form of lead sulphate, a hydrometallurgical treatment of slimes was carried out before the flotation process with the aim of transition of one part of lead sulphate into easier to float lead sulphide.

Because the sphalerite, which as the microanalysis revealed, was covered with a thin layer of silver compounds, an application of copper sulphate for the zinc activation in the flotation process turned out to be pointless.

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Rola identyfikacji mineralogicznej w procesie wzbogacania materiałów odpadowych

W artykule przedstawiono wyniki badań nad odzyskiem metali ze szlamów pochodzących z bieżącej produkcji ZGH Bolesław oraz składowanych od wielu lat w osadnikach pochodzących z działalności nieczynnego Zakładu Elektrolizy Cynku w Szopienicach. Szlamy te zawierają wysokie zawartości różnych metali, w tym również metali szlachetnych, np. srebra. W publikacji podkreślono znaczenie rozpoznania, obok składu granulometrycznego i chemicznego, również składu mineralnego tych odpadów.

Identyfikację składu mineralnego badanych szlamów dokonano przy użyciu mikroanalizatorze rentgenowskim (EPMA) JXA 8230 firmy Jeol.

W oparciu o wykonaną charakterystykę odpadów i występujących w nim minerałów użytecznych opracowano metodę wzbogacania. Badania technologiczne nad odzyskiem srebra, cynku i ołowiu prowadzono przy zastosowaniu procesu flotacji. Dla odzysku ołowiu występującego w postaci siarczanu ołowiu prowadzono hydrometalurgiczną obróbkę szlamu przed procesem flotacji w celu przejście części siarczanu ołowiu w łatwiej flotowalny siarczek ołowiu.

Słowa kluczowe: identyfikacja mineralogiczna, mikroanaliza, flotacja, szlam, odczynnik flotacji