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Release of critical metals from furnace wastes using the process of bioleaching in various variants

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Abstract: The aim of this work was to determine the influence of various variants of bioleaching on effectivity of releasing chosen critical metals: rhodium, cadmium, indium, niobium and chromium from ashes which are a byproduct of municipal waste and sewage sludge thermal processing. The research was conducted in 3 variants that considered different process factors such as temperature (24ºC and 37ºC), mixing intensity and aeration. After 5 days of the process the analyses were made of metals content, sulfate concentration, pH, general number of bacteria number, index of sulfur oxidizing bacteria. The best results of bioleaching were achieved by running the process at the temperature of 24ºC with aeration. The efficiency of rhodium and cadmium release from the byproduct of municipal waste thermal processing was above 90%. The efficiency of indium and chromium release reached 50–60%. Only niobium leached better in mixing conditions. The byproduct of sewage sludge thermal processing was far less susceptible to bioleaching. The highest effectivity (on a level of 50%) was reached for indium in temperature of 24°C with aeration. The efficiency of bioleaching depended on waste's physiochemical properties and type of metal which will be released. Aeration with compressed air had a positive influence on the increase of sulfur oxidizing bacteria what corresponded with almost double increase of sulfate concentration in leaching culture. Such conditions had a positive influence on the increase of the efficiency of bioleaching process. Heightening the temperature to 37°C and slowly mixing did not impact bioleaching in a positive way.

Introduction

One of the methods of waste disposal is their thermal processing. The byproduct of that process is the production of significant amounts of furnace waste. In Europe there are 37 million Mg of ashes containing heavy metals yearly (Sushil and Batra 2006). Furnace waste can contain a lot of precious and rare metals which could be reused in various branches of economy. Intensive development of the industry and new technologies are related to a rising need for metals which are in limited supply. In the year 2010 a report concerning European Union's critical mineral resources was introduced. Said minerals are those for which there are no natural and derivative sources in the EU countries and are impossible to produce on different stages. To determine the level of criticality two factors are used: significance for the industry and risk related with supplying. There were 14 mineral materials described in the report, 12 of which were metals (Smakowski 2011). In 2017 that list has been updated and now it contains 27 critical materials: cobalt, indium, niobium and metals from the platinum group (rhodium, iridium, platinum, ruthenium, palladium) (Communication from the Commission to the European Parliament on the 2017). According to Smakowski, chromium is one of the most important resources for Polish and European industry (Smakowski 2011). One of the methods of releasing the metals from wastes is bioleaching. Literature data confirm the effectiveness of that method in removing metals from sewage sludge, ashes from power plants and electroplating and electronic refuse disposal. Microbiological processes were also used to regain heavy metals from low percentage ores (Ilyas et al. 2010) and recovery of copper, gold, uranium, germanium, gallium and thorium on the industrial scale (Karwowska and Łebkowska 2008). In the biohydrometallurgical processes microorganisms which produce organic acids, complexing compounds and, above all, bacteria capable of oxidizing the sulfur compounds and creating sulfuric acid are used (Ishigaki et al. 2005, Karwowska 2007, Rohwerder et al. 2003). The research conducted on bioleaching has shown that the best results can be reached by using mixed microorganism cultures which can be sourced from activated sludge of a municipal waste disposal plant (Ishigaki et al. 2005, Yahya and Johnson 2002). Availability of oxygen is a vital factor with huge impact on biohydrometallurgical processes (Andrzejewska-Morzuch and Karwowska 2012). Oxygen deficiency has a negative impact on metal release (Zagury et al. 2001). Filali-Meknassi et al. have observed that higher concentration of dissolved oxygen had a significant influence on acidification and the increase of redox potential during the research on metal release from sewage sludge after the process of fermentation (Filali-Meknassi et al. 2000). Another factor which has a significant influence on bioleaching is temperature. Microorganisms which oxidize sulfur compounds are active in temperatures ranging from below 5°C to above 80°C (Cruz et al. 2010, Kupka et al. 2007, Liu et al. 2003, Olson et al. 2003). Mesophilic bacteria are the most widely used, especially strains such as *A. ferrooxidans*, *A. thiooxidans* or *Leptospirillum ferrooxidans*. Xiang et al. have conducted bioleaching in the temperature of 28°C (Xiang et al. 2000). Andrzejewska-Morzuch and Karwowska have observed that metals were released more intensively in the temperature of 37°C than 22°C (Andrzejewska-Morzuch and Karwowska 2012). There is evidence in the literature confirming possibility of use of thermophilic and extremely thermophilic microorganisms in the process of metal removal (Bullock 2000, d'Hugues et al. 2002). The process of mixing is another important element that has substantial influence on bioleaching intensity. Mixing the leaching culture helps its oxidation and heat transfer (Andrzejewska-Morzuch and Karwowska 2012). Due to differential contents and properties of various refuse types it is essential to choose an adequate variant of bioleaching process taking into consideration such parameters as: temperature, oxidation, and mixing. Attempts to use microbiological leaching processes to remove and salvage metals from refuse of different kinds are made in many research facilities all over the world.

Aim of the research

The aim of this work was to determine the influence of various variants of bioleaching, taking into account such factors as temperature, mixing and oxidizing, on effectivity of removing chosen critical metals: rhodium (Rh), cadmium (Cd), indium (In), niobium (Nb) and chromium (Cr) from ashes which are a byproduct of municipal waste and sewage sludge thermal processing.

Research methods

There were two types of ash waste used in the research:

- Ashes from municipal waste thermal processing (sample A)
- Ashes from sewage sludge thermal processing (sample B)

Metals content in the wastes is shown in Table 1.

Leaching culture has been prepared on a basis of activated sludge coming from a municipal wastewater treatment plant. The activated sludge was a source of active autotrophic and heterotrophic bacterial strains. The culture consisted of activated sludge and distilled water in 1:1 ratio and 1% of dusty sulfur. The process of leaching culture's adaptation was conducted in conditions of shaking (120 rpm) and temperature of 24°C in order to achieve pH level below 3 and index of sulfur oxidizing bacteria on a level of 10^{-6}. Metal bioleaching from the waste was conducted in 300 mL Erlenmeyer flasks containing 150 mL of leaching culture and 10 g of waste. The research was conducted in 3 variants of bioleaching and adequately in 3 control variants. The results for control variants were similar, so they were presented as an average value separately for every metal. The bioleaching process variants for samples A and B are shown in Table 2.

The process lasted for 5 days. Afterward the following analyses were made:

- Sulfate concentration – in accordance with PN-ISO 9280:2002 norm,
- General number of bacteria – in accordance with PN-EN ISO 6222 norm,
- Index of sulfur oxidizing bacteria – done using breeding method on a WR (water on rock) culture medium. The WR medium composition was: $(NH_4)_2SO_4$ (1.0 g), K_2HPO_4 (0.5 g), sulfur (10.0 g), 1% bromocresol purple solution (0.5 mL), 10% solution of yeast extract (few drops),
- pH – in accordance with PN-90/C-04540/01,
- Metals content – measurements were conducted using a Shimazu ICPE-9820 plasma spectrometer.

Table 1. Metals content in wastes [mg·kg^{-1} of the waste]

Type of waste	Rh	Cd	Nb	In	Cr
A	132.0	40.0	33.6	143.3	120.0
B	43.3	–	15.3	120.0	–

Table 2. The bioleaching process variants for samples A and B

Variant	Temperature	Speed of mixing	Aeration with compressed air
1	24°C	120 rpm	No
2	24°C	*	Yes
3	37°C	50 rpm	No

* In case of variant 2, mixing was done by aeration.

Determination of the elemental composition was performed by ICP-OES plasma excitation spectrometry. It allows for marking several dozen of elements at the same time and is characterized by high sensitivity and precision. To mark the metals the following reagents were used:

- nitric acid of 65% PURANAL purity made by Honeywell,
- deionized water of Mili-Q purity made by Merck,
- argon gas of 99.998% purity.

Working conditions of ICP-OES emission spectrometer are shown below:

- generator sensitivity – 27.12 MHz, generator's power – 1.20 kW,
- total argon flow – 10.00 L·min^{-1},
- support argon flow – 0.70 L·min^{-1},
- secondary argon flow – 0.60 L·min^{-1},
- exposure time – 30 sec,
- number of expositions – 3,
- duration of system washing – 30 sec,
- rotation of the peristaltic pump – 20–60 rotations·min^{-1}.

The efficiency of the process was calculated according to the formula:

$$X = \frac{100 \cdot C_r \cdot V_s}{m_s \cdot M_s}$$

Where:
X – bioleaching efficiency [%],
C_r – concentration of metal release in a leaching culture [mg·L^{-1}],
V_s – volume of leaching culture [L],
m_s – mass of waste sample taking into experiment [kg],
M_s – metal content in waste [mg·kg^{-1}].

Results of the research

The results of metal bioleaching from the ash A samples are presented in Figures 1–5. The results from the ash B samples are presented in Figures 6–8. In the case of ash A sample the bioleaching effectivity of four metals out of five was the highest in the variant 2 in the temperature of 24°C with aeration. The

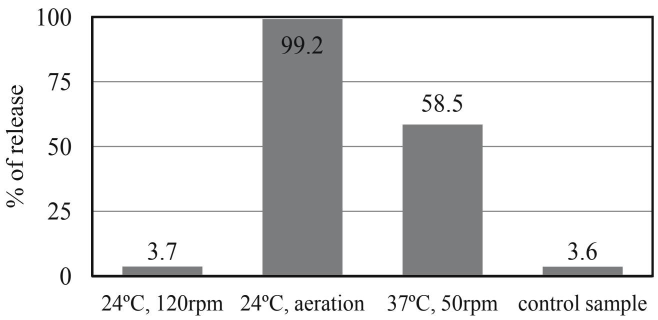

Fig. 1. Efficiency of rhodium release from ash sample A

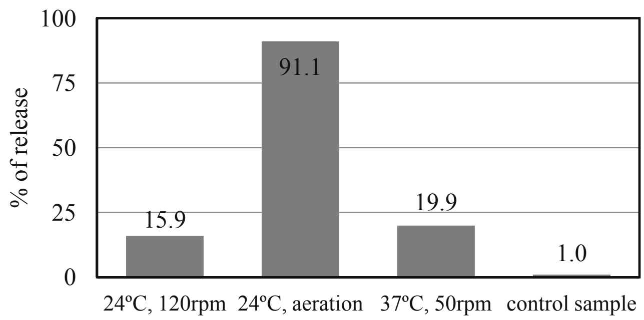

Fig. 2. Efficiency of cadmium release from ash sample A

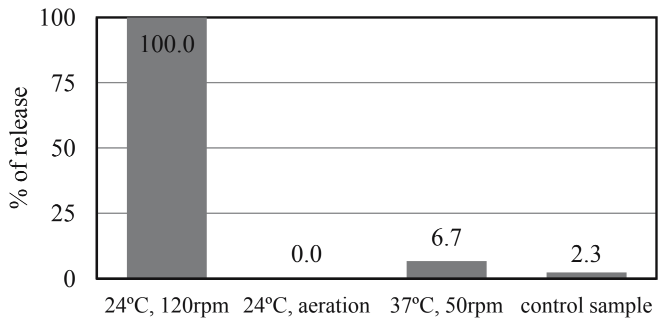

Fig. 3. Efficiency of niobium release from ash sample A

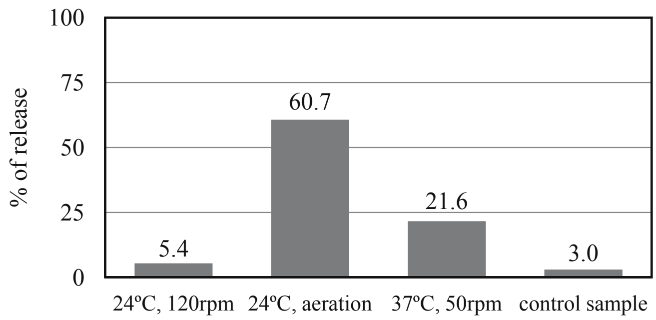

Fig. 4. Efficiency of indium release from ash sample A

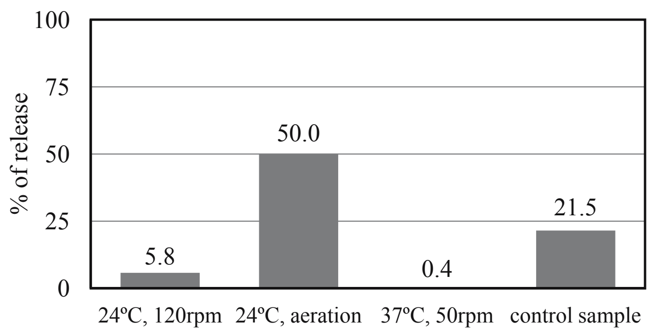

Fig. 5. Efficiency of chromium release from ash sample A

release of rhodium and cadmium was above 90%. The release of indium and chromium – respectively 60% and 50%. In the case of niobium the highest effectivity was observed in variant 1 in temperature of 24°C, mixed. The effectivity for this metal reached 100%. In the case of variant 3 in the temperature of 37°C in conditions of slow mixing, the effectivity of metal releasing amounted for rhodium almost to 60%, for cadmium and indium to around 20%, for niobium to nearly 7% and for chromium was lower than 1%. Metal releasing from the control variant was lower than 4% for rhodium, cadmium, indium and niobium. Chromium was an exception and its release amounted to a bit more than 20%.

In the case of the ash B sample, the effectivity of bioleaching above 50% was reached for indium. The process was conducted in variant 2. The efficiency of niobium release process in variants 1–3 was on a similar level and amounted to almost 15%. The highest effectivity of microbiological bioleaching for rhodium was reached in variant 1 and amounted to around 20%. The effectiveness of bioleaching in the variants 2 and 3 did not differ much and amounted to10% and 8%, respectively, The release of metals under scrutiny in the control variant was below 6%.

The research of microorganism activity consisted of general number of bacteria, the index of sulfur oxidizing bacteria and sulfate concentration in different variants after 5 days of bioleaching. Total number of bacteria in leaching culture before adding the wastes was $2.1 \cdot 10^6$ CFU·mL^{-1}. After 5 days it was observed that the number of bacteria was on a similar level in the case of ash A sample in variants 1 and 3 and it was, respectively, $1.8 \cdot 10^6$ CFU·mL^{-1} and $1.6 \cdot 10^6$ CFU·mL^{-1}. In variant 2 the number of bacteria has risen to $8.4 \cdot 10^6$ CFU·mL^{-1}. The situation in the sample of ash B was substantially different. A general number of bacteria decreased in the case of variants 1 and 3 and was, respectively, $2.4 \cdot 10^3$ CFU·mL^{-1}, $1.2 \cdot 10^3$ CFU·mL^{-1}, and $6.2 \cdot 10^4$ CFU·mL^{-1} for variant 2. The initial index of sulfur oxidizing bacteria was 10^{-6}. After 5 days of leaching it rose to 10^{-7} in variant 2 and decreased in variants 1 and 3 to 10^{-5} for samples of ash A as well as ash B. The general number of bacteria and the index of sulfur oxidizing bacteria are shown in Table 3.

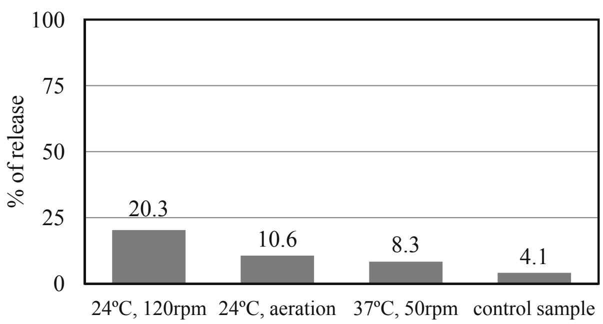

Fig. 6. Efficiency of rhodium release from ash sample B

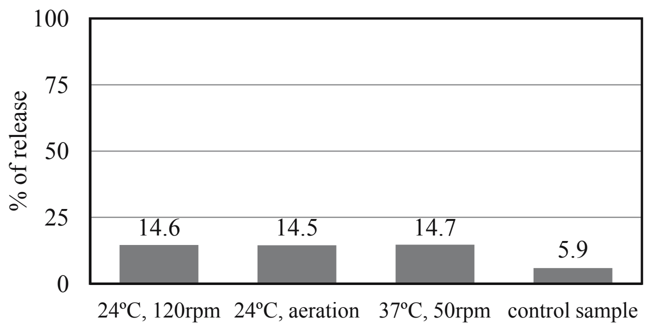

Fig. 7. Effectivity of niobium release from ash sample B

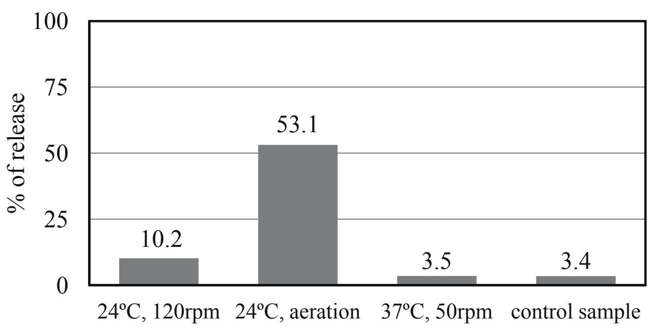

Fig. 8. Effectivity of indium release from ash sample B

Table 3. The general number of bacteria and the index of sulfur oxidizing bacteria of leaching

Waste	Variants	Number of bacteria	Index of sulfur oxidizing bacteria
A	1	$1.8 \cdot 10^6$ CFU·mL^{-1}	10^{-5}
	2	$8.4 \cdot 10^6$ CFU·mL^{-1}	10^{-7}
	3	$1.6 \cdot 10^6$ CFU·mL^{-1}	10^{-5}
B	1	$2.4 \cdot 10^3$ CFU·mL^{-1}	10^{-5}
	2	$6.2 \cdot 10^4$ CFU·mL^{-1}	10^{-7}
	3	$1.2 \cdot 10^3$ CFU·mL^{-1}	10^{-5}

Culture after 5 days of bioleaching

Initial sulfate concentration in the leaching culture before adding the waste was around 6000 mg·L^{-1} (sulfate concentration that was created during the adaptation process in the presence of sulfur). In variant 1 in temperature of 24°C with mixing and variant 3 in temperature of 37° with slow mixing the sulfate concentration decreased. The sulfate concentration rose almost twice from initial level in variant 2 with aeration in temperature of 24°C for both samples. In variant 2, there was much more oxygen needed for sulfate oxidation than in variants 1 and 3. The sulfate concentration in various variants of leaching cultivation after 5 days of bioleaching is shown in Figure 9.

The result corresponded with good results of rhodium, cadmium, indium and chromium bioleaching in sample A and indium in sample B.

In the last day of the process the pH of the leaching solutions was marked. Initial pH in the leaching culture before adding the waste was below 3. In the sample with waste A in variants 1 and 3 pH rose to 10 and in the case of variant 2 to 6. In the sample with waste B in variants 1 and 3 pH rose to 9 and in variant 2 to 8. Significant growth of the value during bioleaching resulted from highly alkaline qualities of both wastes.

Summary

The factors which influence the effectivity of biohydrometallurgic processes are: source of carbon, oxygen availability, pH, temperature, conditions of preliminary cultivation and type of grafting, microorganisms' resistance to metal ions, physiochemical parameters of leached material, percentage waste contribution in the leaching culture and duration of bioleaching (Guo et al. 2010, Wang et al. 2009, Xu and Ting 2004). In this research an attempt was made to assess the influence of different leaching culture variants on the effectivity of critical metal release from ashes which are a byproduct of thermal treatment of municipal waste (A) and sewage sludge (B). The variants were a combination of such parameters as: temperature, aeration, intensity and method of mixing. In Table 4 the amounts of metals obtained using bioleaching process in conversion to mg·kg^{-1} of waste are shown.

In the case of waste A the best results of microbiological leaching were achieved for rhodium, cadmium and niobium. Process effectivity reached above 90%, the amount of released metals was: Rh – 130.9 mg·kg^{-1}, Cd – 36.4 mg·kg^{-1} and Nb – 33.6 mg·kg^{-1}. The level of indium and chromium release was lower and reached 50–60%. 87.0 mg of indium and 60.0 mg of chromium were released from 1kg of waste A. Temperature of 24°C and aeration was favorable for bioleaching of rhodium, cadmium, indium and chromium. The only metal which was released in higher amount in the same temperature but with mixing was niobium. 33.6 mg of niobium was released from 1 kg of waste with effectivity of 100%. There are reports in the literature that heightening of the temperature is favorable for the process of bioleaching (Andrzejewska-Morzuch and Karwowska 2012). Because of that it was decided to create variant 3, which had temperature of 37°C and was mixed slowly. The highest effectivity of bioleaching (60%) was reached for rhodium from waste A. The efficiency of this

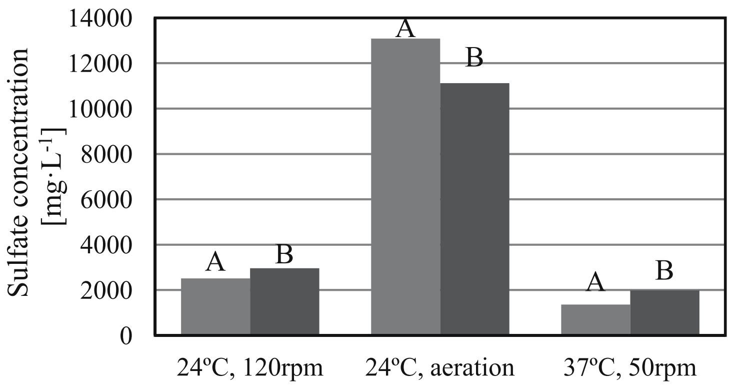

Fig. 9. Sulfate concentration in various variants of leaching culture after 5 days of bioleaching [mg·L^{-1}]

Table 4. Amounts of metals released from ashes samples A and B after 5 days of bioleaching [mg·kg^{-1}] culture after 5 days of bioleaching

Waste	Metal	24ºC, 120 rpm	24ºC, aeration	37ºC, 120 rpm
A	Rh	4.9	130.9	77.2
	Cd	6.4	36.4	8.0
	Nb	33.6	0.0	2.3
	In	7.7	87.0	31.0
	Cr	7.0	60.0	0.5
B	Rd	8.8	4.6	3.6
	Nb	2.2	2.2	2.2
	In	12.2	63.7	4.2

process was significantly lower than the one conducted in temperature of 24°C with aeration.

Ash sample B was less susceptible to bioleaching. The highest effectivity was reached for indium in the temperature of 24°C with aeration. 63.7 $mg \cdot kg^{-1}$ of this metal was released which equals 50% efficiency. In the case of rhodium and niobium the efficiency of the process was lower than 20% in all variants. The highest amounts of released metals were 8.8 $mg \cdot kg^{-1}$ for rhodium and 2.2 $mg \cdot kg^{-1}$ for niobium.

Waste samples A and B had alkalinizing properties. It is supported by an analysis of pH changes conducted during the last day of bioleaching. In the case of both wastes after 5 days of the process a strong alkalization of leaching cultures in variants 1–3 took place (pH 9–10). In the cultivations in variant 2, alkalization was less intensive. For waste A pH was 6, and for waste B – 8. Aeration caused a lowering of reaction in comparison with other cultivations. It was caused by a higher microorganism activity which was confirmed by a quantitative bacteria analysis and almost double rise in sulfate content compared to initial value (before waste addition). In the course of this research 314.4 mg of critical metals was released out of a 1 kg sample of byproduct of thermal processing of municipal waste in conditions of aeration in temperature of 24°C. Such conditions increased the effectivity of metal release. Because of the increasing need for critical metals and their limited supply in the environment it is crucial to conduct research on bioleaching optimization which causes an increase in metal recovery from waste.

Conclusions

Basing on this research one can formulate the following conclusions:

- Choosing the bioleaching variant should be conditional to waste's physiochemical properties and type of released metal. Wastes used in the research were of different susceptibility to metal bioleaching. Better results were achieved for waste A than B.
- Aeration with compressed air which also worked as mixing increased the efficiency of bioleaching and allowed for rhodium, cadmium, chromium and indium release from waste A and indium release from waste B. The efficiency of rhodium and cadmium release was above 90% and of indium and chromium respectively 60% and 50% in waste A. The efficiency of indium release was 50% in waste B.
- Aeration of the leaching culture had a positive influence on increase of sulfur oxidizing bacteria what corresponded with almost double increase of sulfate concentration (11 000–13 000 $mg \cdot L^{-1}$).
- Niobium as the only metal was released with high efficiency from waste A in the temperature of 24°C, mixed.
- Heightening the temperature to 37°C and mixing the solution slowly did not impact bioleaching in a positive way. The only exception was rhodium bioleaching from waste A, where efficiency of the process reached almost 60%.
- The furnace ash samples used in the research had strong alkaline properties and because of that the process of releasing the metals was hampered.

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Uwalnianie metali krytycznych z odpadów paleniskowych z wykorzystaniem procesu biołųgowania w różnych wariantach

Streszczenie: Celem niniejszych badań była ocena wpływu różnych wariantów biołųgowania na efektywność uwalniania wybranych metali krytycznych takich jak rod, kadm, ind, niob i chrom z odpadów będących produktami ubocznymi termicznego przetwarzania odpadów komunalnych i osadów ściekowych. Badania były prowadzone w 3 wariantach uwzględniających różne parametry procesu, takie jak: temperatura (24ºC i 37ºC), intensywność mieszania i napowietrzanie. Po 5 dniach prowadzenia procesu zostały wykonane oznaczenia: zawartości metali, stężenia siarczanów, pH, ogólnej liczby bakterii, miana bakterii utleniających siarkę. Najlepsze rezultaty osiągnięto przy prowadzeniu procesu w temperaturze 24°C, w warunkach napowietrzania. W przypadku próbki popiołu z termicznego przetwarzania odpadów komunalnych efektywność uwalniania rodu i kadmu wyniosła powyżej 90%, natomiast indu i chromu kształtowała się na poziomie 50–60%. Tylko niob ługował się lepiej w warunkach mieszania. Próbka popiołu ze spalania osadów ściekowych była mniej podatna na biołųgowanie. Najwyższą efektywność – na poziomie 50% osiągnięto dla indu w temperaturze 24°C, w warunkach napowietrzania. Efektywność zależała od typu odpadu oraz rodzaju uwalnianego metalu. Napowietrzanie sprzężonym powietrzem miało pozytywny wpływ na wzrost liczebności bakterii utleniających siarkę, co korespondowało z prawie dwukrotnym wzrostem zawartości siarczanów w hodowlach ługujących. Takie warunki sprzyjały zwiększeniu skuteczności procesu. Zastosowanie temperatury 37°C i powolnego mieszania nie wpłynęło korzystnie na biołųgowanie.