

Research article

Ammonia leaching of slag from direct-to-blister copper smelting technology

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Abstract: Smelting of copper concentrates in a flash furnace directly into a copper blister results in the production of slag with high copper and lead content. The recovery of these metals is carried out in an electric furnace, from which copper–lead–iron metallic alloy is then processed in a converter furnace. In the presented work, copper and lead extraction studies were carried out by hydrometallurgical route under leaching in ammonium acetate solutions. The obtained results indicate the possibility of receiving quite high copper leaching efficiency from slag with a relatively low yield for lead extraction. The use of ammonium acetate solutions with a concentration of 5 mol/dm³ leads to leaching from the slag over 80% of the copper contained in it after 90 min of the process carried out at a temperature of 70 °C. Model calculations related to the study of the kinetics of the process indicate that after 120 min of leaching it can be expected that 90% of the copper contained in the slag could be carried out into the solution.

Keywords: ammonia leaching; direct-to-blister copper flash smelting; copper recovery; leaching kinetics

1. Introduction

The technology of copper concentrates smelting in a flash furnace enables direct metal production, which has the properties of blister copper. Concentrates which contain chalcocite (Cu₂S) and bornite (Cu₅FeS₄) are melted in the flash smelting furnace. Polish copper concentrates mainly come from Rudna and Polkowice mines. The dried concentrate is fed into a flash furnace, and exothermic oxidation reactions of charge components, i.e. coal and copper and other metal sulfides,

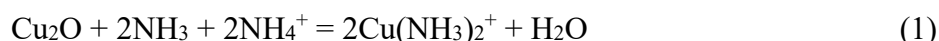
take place in the reaction shaft. In addition, there are endothermic dissociation reactions of calcium and magnesium carbonates and sulfates contained in dust, as well as melting processes of gangue. The flash smelting process of melting copper concentrates is characterized by much lower production costs than the previously used shaft process, and the continuous improvement of direct-to-blister copper production technology puts it at the forefront of the most modern metallurgical aggregates in the world [1]. One of the products of flash smelting of copper concentrates is slag with significant copper content (12–16%), which is the result of high oxidation of concentrate components in the reaction shaft of the flash furnace. This procedure is necessary to achieve a lead concentration below 0.3% in the copper blister. The slag undergoes the decopperization process by reducing oxide metal compounds in an electric furnace. As a result of this process, a Cu–Pb–Fe alloy is obtained, which is then converted. The presence of lead in the alloy causes difficulties in further metallurgical processing and adversely affects the durability of metallurgical aggregates [2].

The chemical composition of flash smelting slag depends on both the type of copper concentrates melted and process parameters. The degree of oxidation of process products, determined by the concentration of Cu_2O in the slag and the oxygen content of copper, depends mainly on the lead content of the concentrate. The degree of oxidation of the concentrate components is used to achieve the required concentration of Pb in the copper blister. The content of the main components of flash smelting slag can vary within quite wide limits [3,4]: 10–16% Cu, 2.5–4% Pb, 4–6% Fe, 32–36% SiO_2 . Structural studies of liquid flash smelting slag show that copper forms a separate phase, whose chemical composition is similar to cuprous oxide. In a liquid slag from a flash smelting furnace, all of the cuprous oxide forms a homogeneous solution with the remaining oxide components of the silicate slag [5]. Due to the high copper content in the slag, the decopperization process is carried out. Therefore, reduction of cuprous oxide and other metals, mainly lead and iron, is carried out in a liquid state in an electric arc furnace in the presence of coke covering the surface of the slag bath and an alkaline technological additive, which is primarily calcium carbonate.

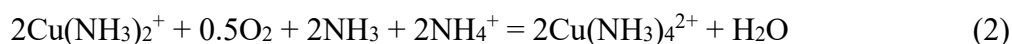
An alternative to the currently used method of processing flash smelting slag could be its crushing, leaching under appropriate conditions, and then extraction of valuable components from the leaching solution. There is a significant number of literature reports on leaching of copper oxide ores in ammonia solutions [6–8], processing of flotation waste in ammonia solutions [9], recovery of copper from waste of electrical and electronic equipment (WEEE) [10,11] or extraction of copper from spent catalyst [12–14]. Hydrometallurgical processing of flash smelting slag was the subject of several papers [15–17], and the results obtained there indicated the possibility of a wider application of the methods presented there. Copper has the greatest value among the main components of flash smelting slag. Therefore, from the economic point of view, the highest degree of extraction of copper from this material at the lowest process costs should be obtained. On the other hand, lead has an oxidized form in flash smelting slag and from both an economic and ecological point of view it must be removed from the processed slag so that its content in the leaching residue is low enough to meet this ecological requirement.

In this work it was decided to analyze the possibility of using ammonium acetate solutions for hydrometallurgical processing of flash smelting slag and extraction of the most important elements from it. Ammonia leaching is an effective way to recover metals that form stable ammonia complexes (Cu, Ni, Co, Zn, Ag). This process is selective, metals such as iron or lead do not pass into the solution. Ammonia leaching can be used for both oxide and sulfide materials [18]. An important advantage of ammonia leaching is that copper forms soluble complex ions, while iron is

precipitated as hydroxide $\text{Fe}(\text{OH})_3$. Thanks to this, selective separation of metals is possible already in the first stages of processing of polymetallic raw materials. The dissolution of copper compounds in ammonia solution can be described by the following equations [19]:



In the presence of oxygen, another reaction may occur:



The resulting complex ions of divalent copper $\text{Cu}(\text{NH}_3)_4^{2+}$ can oxidize metallic copper according to the reaction:



Therefore, if the copper-bearing material that undergoes ammonia leaching contains both Cu_2O and copper in the form of metal or copper alloys, it should be assumed that the process of transferring copper to the solution will occur the more effectively the better the access of oxygen to the Eq 2. The separation of metals from solutions after ammonia leaching can be carried out, for example, by pressure reduction with hydrogen gas [20] or by using liquid ion exchangers [21].

2. Materials and methods

2.1. Material

Slag from the Głogów II smelter was used in the tests. This material was obtained in crushed form and therefore a sieve analysis of the slag was made. Its results are presented in Figure 1.

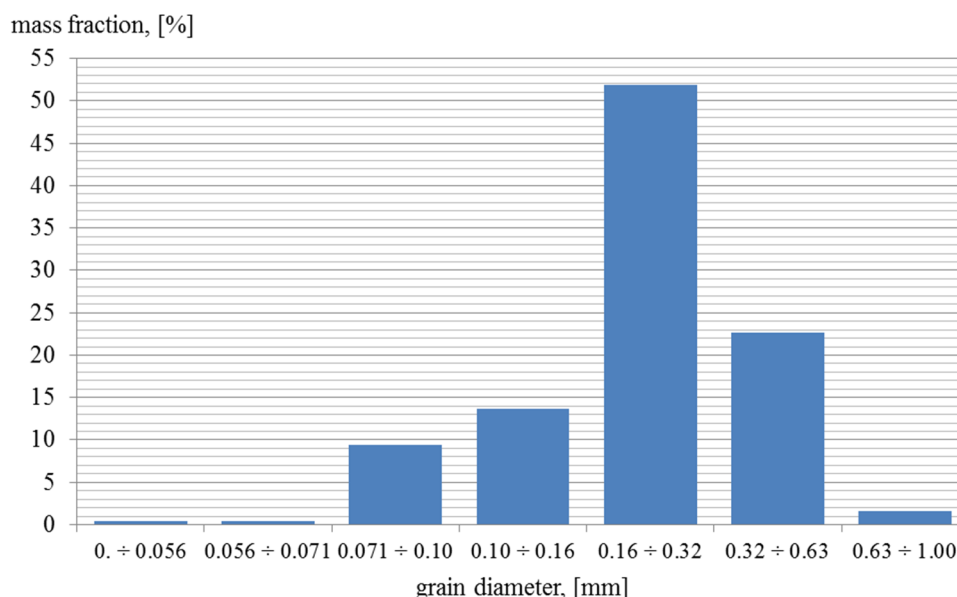


Figure 1. The results of the sieve analysis of flash smelting slag.

Further tests that were carried out on the flash smelting slag sample were performed by scanning microscopy (Figure 2), EDS analysis of the slag (Figure 3) and identification of the slag phase composition made by XRD (Figure 4).

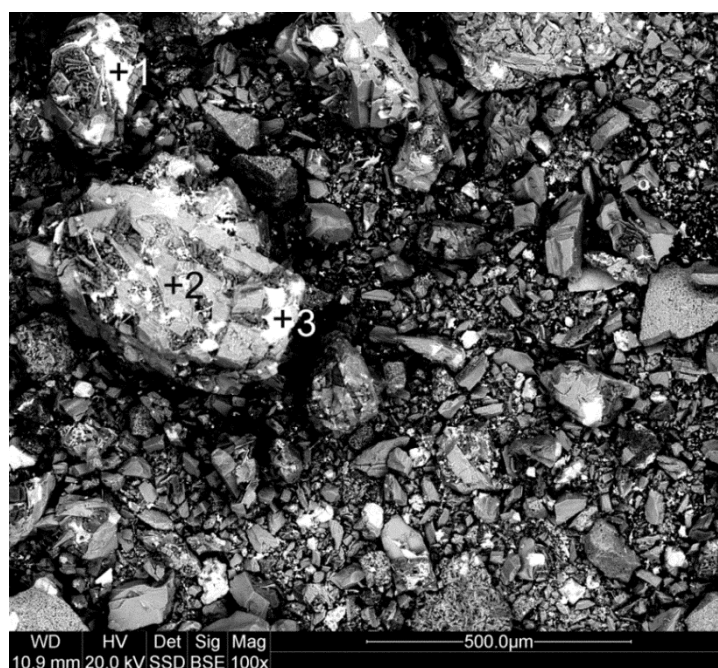


Figure 2. The microscopic image of flash smelting slag sample.

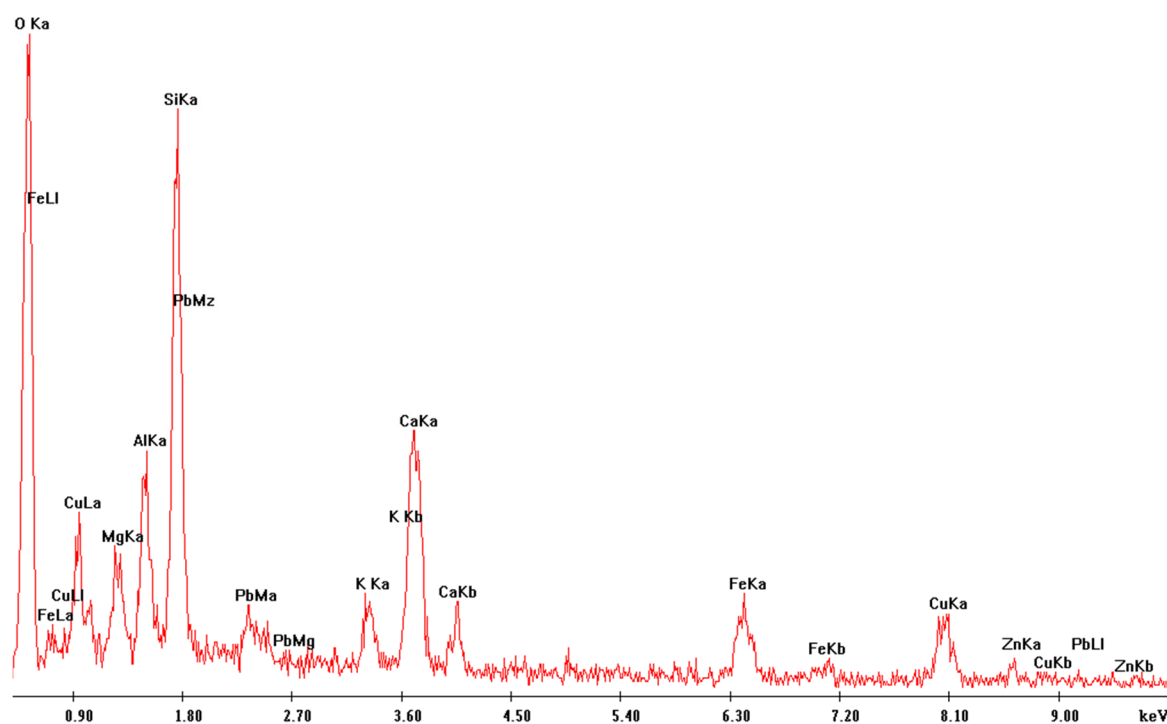


Figure 3. EDS analysis of flash smelting slag sample.

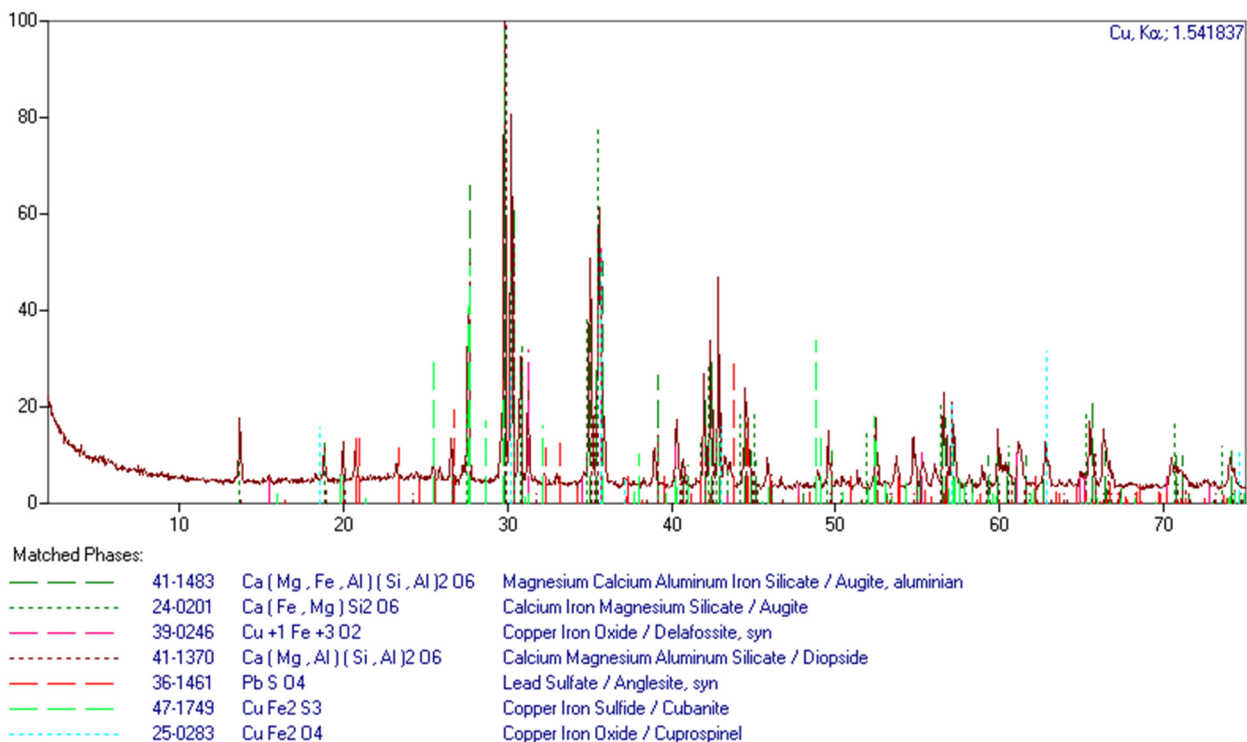


Figure 4. XRD pattern of flash smelting slag sample.

XRF analysis allowed to obtain an approximate chemical composition of the slag sample. It was determined that the following elements were found in the tested material: 11.30% Cu, 5.31% Pb, 6.83% Fe, 13.63% Si, 8.78% Ca, 5.83% Al, 3.67% Mg, 2.73% Zn and 2.03% K. Because of the above chemical composition was determined without reference materials and should be considered as an approximation only, it was decided to perform additional tests of Cu and Pb content in the slag. These elements are crucial for the research carried out in this work, therefore, precise information on the content of these metals in the slag was necessary. Copper and lead concentrations were carried out using AAS method (PerkinElmer atomic absorption spectrometer 3110), obtaining the following result: 12.43% Cu and 3.03% Pb.

Using electron microprobe (EMP) analysis, the contents of elements from characteristic points (1, 2, 3) of the analyzed field presented in Figure 2 were examined. The results in the form of EDS diagrams are presented in Figure 5, and the approximate contents of the elements are presented in Table 1.

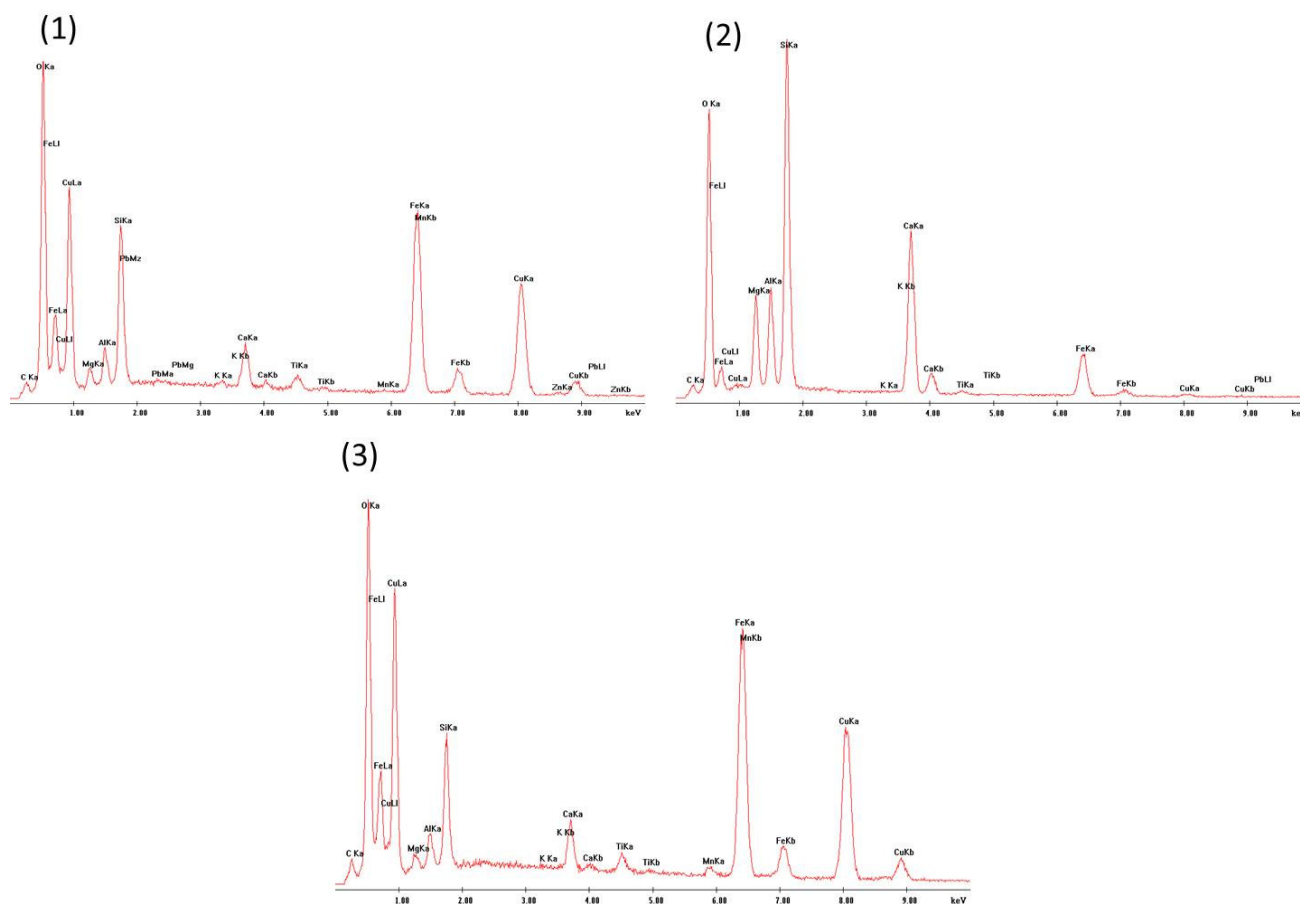


Figure 5. EDS analysis of flash smelting slag samples—(points 1, 2 and 3 seen in Figure 2).

Table 1. Chemical composition of flash smelting slag in three selected points (based on EDS analysis).

Element	Content%		
	Point 1	Point 2	Point 3
Mg	1.38	5.47	0.90
Al	2.06	5.85	1.74
Zn	0.98	-	-
Mn	0.27	-	0.95
Si	7.65	18.41	5.13
Ca	2.24	12.46	2.01
Fe	24.86	9.24	27.76
Cu	29.89	0.99	35.70
Pb	2.49	2.88	-

2.2. Method

The test stand (Figure 6) consisted of a water bath in which a 300 mL beaker for leaching solution was placed. An external thermometer was used to measure the temperature of the solution in the beaker and verify the temperature in relation to the set in the bath. A mechanical stirrer was placed in the beaker to ensure leached material movement in the entire volume of the solution. After

pouring the right amount of ammonium acetate solution of the specified concentration into the beaker, the solution was heated to the assumed temperature. When it was reached, the stirrer was switched on and the rotational speed constant for all tests was 250 rpm. The appropriate amount of slag was introduced into the solution. After leaching, all suspension was filtered on a Büchner funnel, and the resulting solid material was washed with distilled water. Sludge obtained after leaching was analyzed for Cu and Pb content by AAS method. The leaching efficiency was determined on the basis of equation:

$$\eta_m = \left(\frac{m_m^0 - m_m}{m_m^0} \right) \cdot 100\% \quad (4)$$

where m_m^0 is the mass of metal in the sample before the process and m_m is the mass of metal in the sample after the process.

Leaching selectivity was determined for each test by calculating the ratio of copper concentration to lead concentration in the leaching solution:

$$\alpha = \frac{c_{Cu}}{c_{Pb}} = \frac{c_{Cu}^0}{c_{Pb}^0} \cdot \frac{\eta_{Cu}}{\eta_{Pb}} = 4.102 \cdot \frac{\eta_{Cu}}{\eta_{Pb}} \quad (5)$$

where c_{Cu} is the copper concentration in solution after leaching, c_{Pb} is the lead concentration in solution after leaching, c_{Cu}^0 is the copper concentration in flash smelting slag (12.43%), c_{Pb}^0 is the lead concentration in flash smelting slag (3.03%), η_{Cu} is the leaching efficiency of copper, and η_{Pb} is the leaching efficiency of lead.

This coefficient indicates how selectively the process of copper leaching from slag is in relation to lead. The higher α coefficient, the more copper is in the solution after leaching in relation to lead.

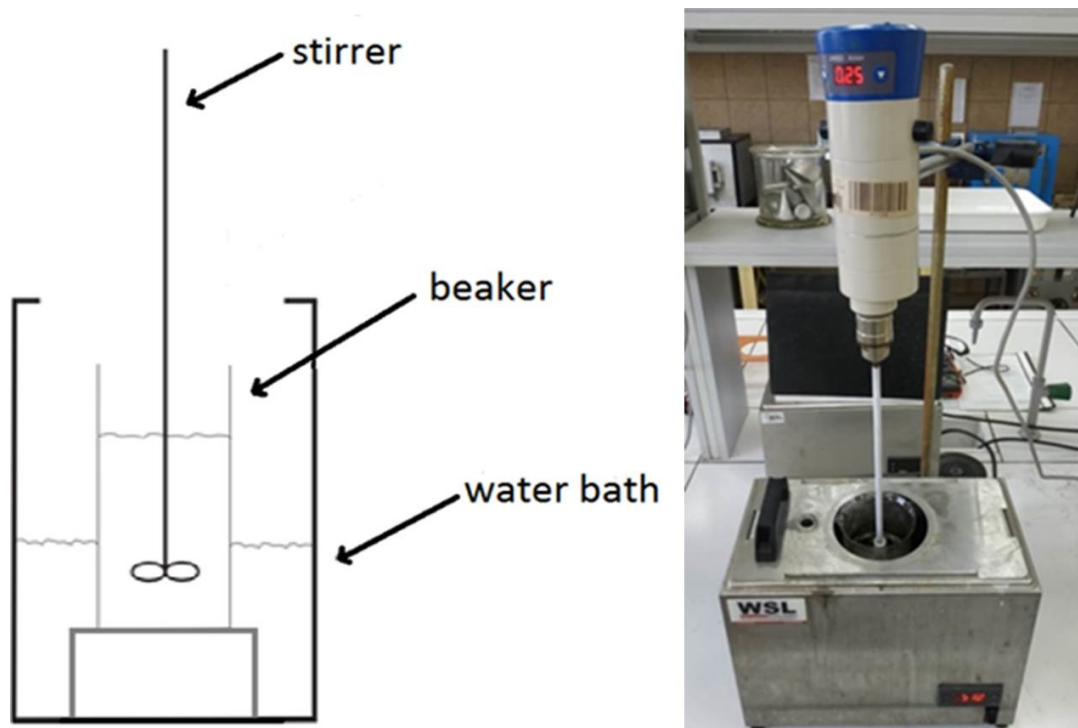


Figure 6. Scheme of the research set for leaching of flash smelting slag.

3. Results

3.1. Effect of ammonium acetate concentration and time

Research on leaching of many materials indicates a number of parameters affecting this process. The leaching time and the concentration of the leaching solution are the parameters that most significantly affect the speed and efficiency of transferring elements from solid to liquid phase. Therefore, it was decided to first examine how copper and lead leaching efficiency and selectivity depend on the time and concentration of the ammonium acetate solution. The tests were carried out at a set temperature of 70 °C and a liquid to solid ratio (l/s) of 10. Samples for analysis for Cu and Pb content were taken after 30, 60 and 90 min. The results of the research are presented in Figure 7.

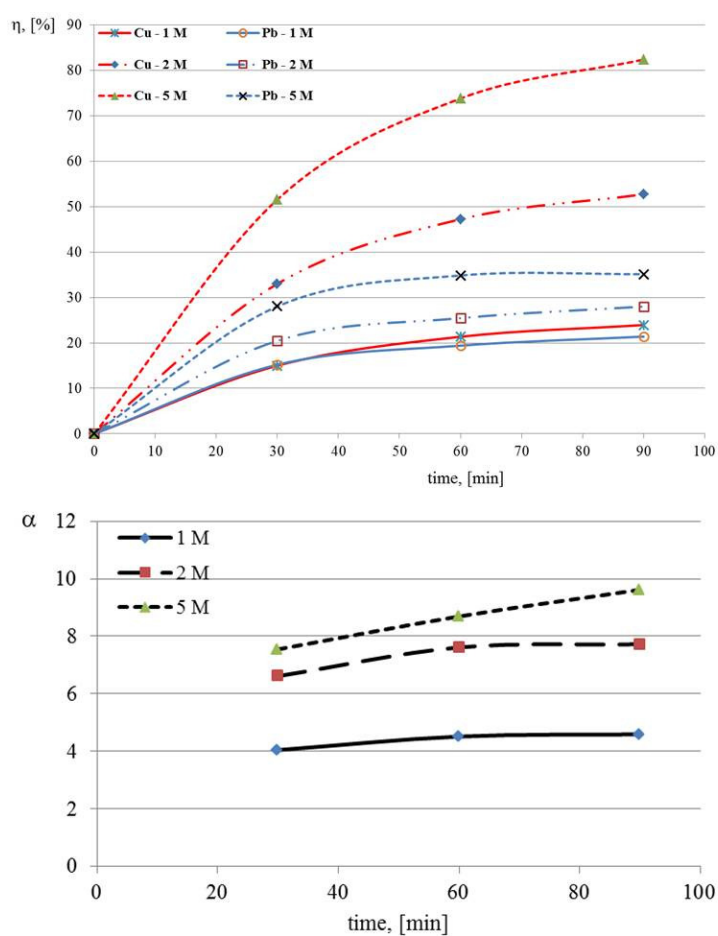


Figure 7. Leaching efficiency of copper η_{Cu} and lead η_{Pb} as well as leaching selectivity α vs process time for various concentration of the ammonium acetate solution ($T = 70\text{ }^{\circ}\text{C}$, $l/s = 10$).

3.2. Effect of leaching temperature

The effect of temperature on the leaching process was examined for the time at which the highest copper leaching efficiency was noted in the previous test stage, and for two concentrations of

ammonium acetate solutions at which the copper leaching efficiency exceeded 50%. Therefore, it was assumed that leaching would take place over 90 min, the ammonium acetate solution concentrations were 2 and 5 mol/dm³, and the l/s ratio was still 10. The temperature was changed from 50 °C by 70 °C to 90 °C. The results obtained in the form of the chart of copper and lead leaching efficiency and selectivity vs temperature are presented in Figure 8.

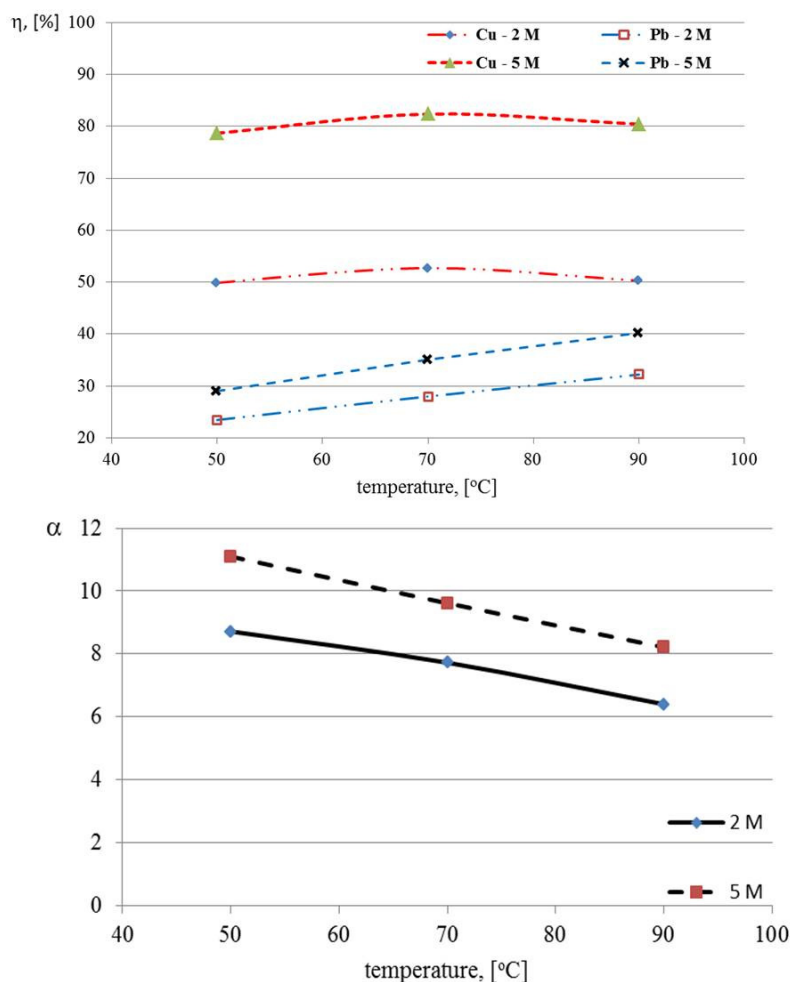


Figure 8. Leaching efficiency of copper η_{Cu} and lead η_{Pb} as well as leaching selectivity α vs temperature for various concentration of the ammonium acetate solution ($t = 90$ min, $l/s = 10$).

3.3. Effect of solid/liquid ratio

The last leaching parameter analyzed in these studies was l/s ratio. The previous stages showed that the highest copper leaching effects and high leaching selectivity were obtained at the highest concentration of ammonium acetate solution (5 mol/dm³), within 90 min and at 70 °C. Therefore, with these parameters, this part of the research was carried out, changing the ratio l/s from 5 by 10 to 20. The obtained results are shown in Figure 9.

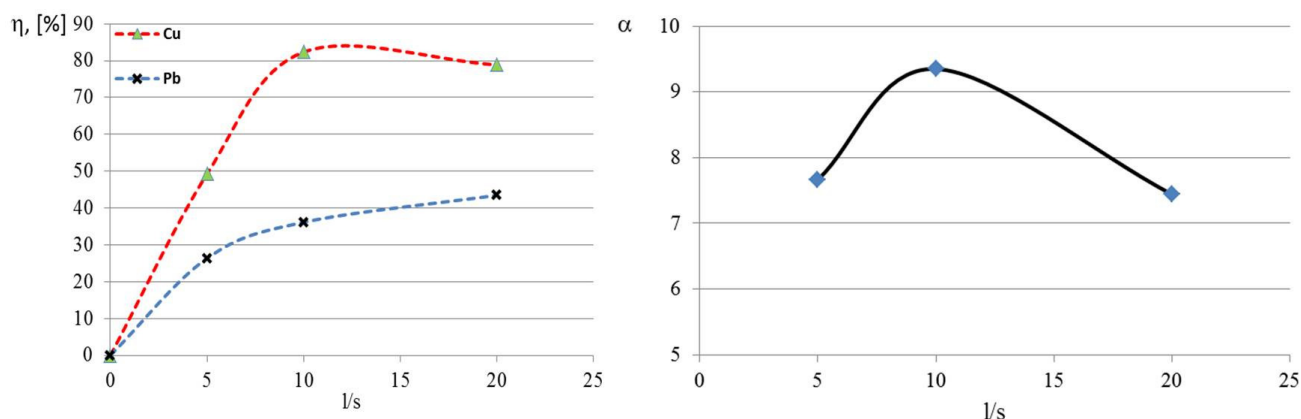


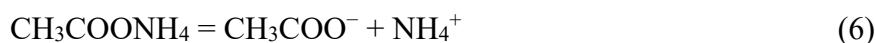
Figure 9. Leaching efficiency of copper η_{Cu} and lead η_{Pb} as well as leaching selectivity α vs l/s ratio (5 mol/dm³ CH₃COONH₄, T = 70 °C, t = 90 min).

4. Discussion

Flash smelting slag used in the tests was characterized by a fairly high degree of comminution (Figure 1). More than 75% of its mass was made of grains in the 0–0.32 mm diameter range, and over 50% in the 0.16–0.32 mm range. It can be assumed that such a fine material ensured an efficient leaching process. XRD analysis (Figure 4) indicated that copper in the slag is in a complex form with iron and oxygen (CuFeO₂, CuFe₂O₄) and with iron and sulfur (CuFe₂S₃). A slightly more complicated situation can occur in the case of lead compounds contained in leached material. XRD analysis identified lead in slag in the form of PbSO₄ sulfate. This is probably not the only lead compound found in the analyzed material. A large amount of silicon in the slag (~13.63%) may indicate that the slag contains some amounts of lead silicates, which due to their amorphous nature cannot be identified in the XRD analysis. An interesting observation is also that flash smelting slag contains lead in the form of PbSO₄, not PbO. Contrary to appearances, this is quite likely and results from the oxidation sequence of PbS lead sulfide during smelting copper concentrate under highly oxidizing conditions at high temperature [22]. This sequence is PbS → PbSO₄ → PbSO₄·PbO → PbSO₄·2PbO → PbSO₄·4PbO → PbO.

EMP analysis of the slag sample showed that we are dealing with a material with a fairly large variation in chemical composition even in a field with a very limited surface (Figures 2 and 5). In the immediate vicinity there are areas with a high concentration of Cu, Fe and Pb (point 1) or Cu and Fe (point 3) and places rich mainly in slag-forming components (point 2).

This study carried out tests using aqueous ammonium acetate solutions. In the case of salts whose cation and anion are derived from weak electrolytes in aqueous solutions, both ions formed as a result of dissociation are hydrolysed:



In the case of copper and its compounds, when ammonia and NH_4^+ ions appear in an aqueous solution, the possibility of Eqs 1–3 appears, which leads to copper being dissolved. The probability of spontaneous leaching of copper in the presence of ammonia can be confirmed by the E-pH diagram of the Cu– NH_3 – H_2O system [23]. It is presented in Figure 10.

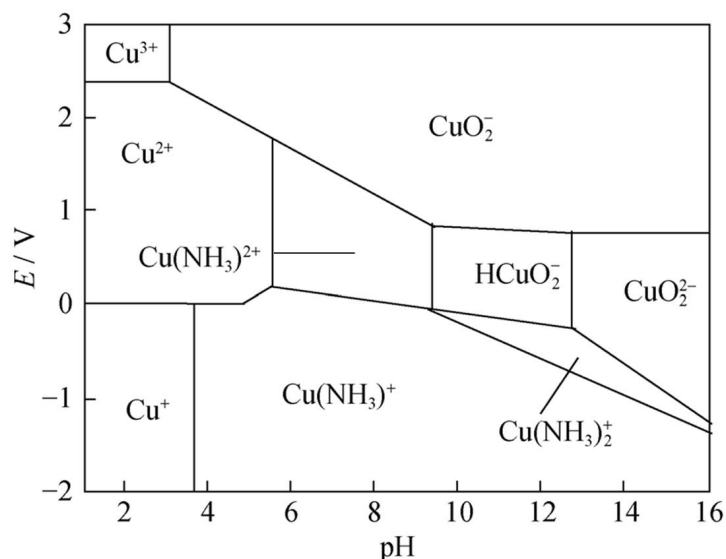


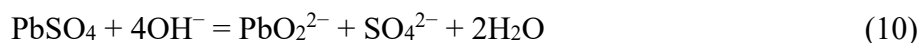
Figure 10. E-pH diagram for Cu– NH_3 – H_2O system.

Considering that the E-pH diagram refers to the state of equilibrium, it can be said with a high degree of certainty that the process of transferring copper from the crushed slag to the solution will take place at de-energized conditions in the pH range close to 7, i.e. to that obtained after dissolving ammonium acetate in water.

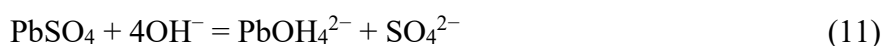
Lead sulfate reacts with ammonium acetate according to the reaction:



As a consequence, lead goes into solution in the form of soluble lead acetate [24]. The presence of hydroxide anions resulting from the Eq 7 may affect the process of conducting lead to the solution [25]:

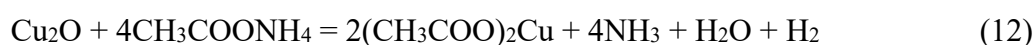


or

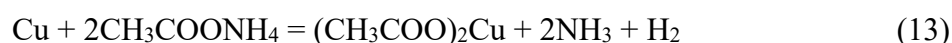


Theoretically, therefore, there may be a situation in which lead is in solution both in the form of the Pb^{2+} cation and the PbO_2^{2-} (PbOH_4^{2-}) anion.

Cuprous oxide can react with ammonium acetate according to the reaction:



In the case of metallic copper, the following reaction can occur:



Previous studies [15] indicate that the above reactions can occur during leaching of flash smelting slag rather in limited range, and the transition of copper to the solution will be provided primarily by Eqs 1–3.

The tests carried out on flash smelting slag leaching in ammonium acetate solutions indicate a significant effect of this reagent concentration on the leaching efficiency of both copper and lead (Figure 7). The highest numerical value is obtained for a solution with a concentration of 5 mol/dm³. The relationship between process time and leaching efficiency is also noticeable. It can therefore be concluded that the higher the leaching solution concentration and the longer the process time, the higher copper and lead leaching efficiency. The leaching selectivity α determined for a time of 90 min and for a solution with a concentration of 5 mol/dm³ is approaching the value of 10. This means that after leaching in the solution, the concentration of copper is almost 10 times higher than the concentration of lead. The leaching tests performed showed a small influence of temperature on copper leaching efficiency and a directly proportional relationship between lead leaching efficiency and temperature in the tested range (Figure 8). Therefore, from the point of view of copper leaching efficiency, there is no significant difference between the process carried out at 50, 70 or 90 °C. However, from the point of view of the selectivity of leaching or energy consumption in this process, the process should be carried out at the lowest possible temperature, i.e. in relation to the tests carried out at a temperature equal to 50 °C. Under these conditions, the factor α exceeds the value of 11 for an ammonium acetate solution with a concentration of 5 mol/dm³. The 1/s ratio of 10 is optimal from the point of view of copper leaching efficiency and high value of the coefficient α (Figure 9). Summing up this part of experimental research, it can be stated that the optimal leaching conditions of flash smelting slag in ammonium acetate solution are as follows: CH₃COONH₄ concentration is the 5 mol/dm³, time is the 90 min and temperature is the 50 or 70 °C, 1/s = 10. In the above conditions up to about 80% Cu and 35% Pb contained in the slag passes into solution, and the concentration of copper in the solution after the process is almost 10 times higher than the concentration of lead.

The reaction that occurs between the slag particles and the ammonium acetate solution during leaching is a typical example of a heterogeneous liquid-solid reaction. Kinetic analyzes of this type of reaction are generally performed according to non-catalytic heterogeneous reaction models. In this work, it was decided to analyze and apply to the obtained results two models found in the literature. First of them based on the interface transfer and diffusion across the product layer [26]. Equation of this model is given as follows:

$$\frac{1}{3} \ln(1-x) - [1 - (1-x)^{-1/3}] = k_1 t \quad (14)$$

where: x is the mass of metal in solution/mass of metal in the original slag sample, t is the process time and k_1 is the speed constant.

Among the non-catalytic heterogeneous reaction models, the most common model applied to leaching reactions is the shrinking core model. The integrated rate equations derived for the shrinking core model have been described in detailed in the literature [27,28], and they have been applied to many leaching processes [29,30]. The kinetic equation has the following form:

$$1 - 3(1-x)^{2/3} + 2(1-x) = k_2 t \quad (15)$$

Leaching performance results as a function of time for three leaching solution concentrations were used to calculate the speed constants according to the two models presented above. Table 2

presents the results of calculating the speed constants k_1 and k_2 and square of the correlation coefficients R^2 . Figure 11 shows the graphs of model Eqs 14 and 15 relative to experimental points obtained during the research.

Table 2. Speed constants k_1 and k_2 and R^2 leaching reaction of copper and lead depending on the concentration of leaching solution obtained for the models used.

	CH ₃ COONH ₄ concentration (mol/dm ³)	$\frac{1}{3} \ln(1-x) - [1 - (1-x)^{-1/3}]$ k_1	R^2	$1 - 3(1-x)^{2/3} + 2(1-x)$ k_2	R^2
Cu	1	4.99E-05	0.9881	2.53E-04	0.9829
	2	3.81E-04	0.9900	1.45E-03	0.9872
	5	2.09E-03	0.9479	4.61E-03	0.9921
Pb	1	3.98E-05	0.9581	2.06E-04	0.9451
	2	7.51E-05	0.9531	3.68E-04	0.9336
	5	1.44E-04	0.8473	6.54E-04	0.8214

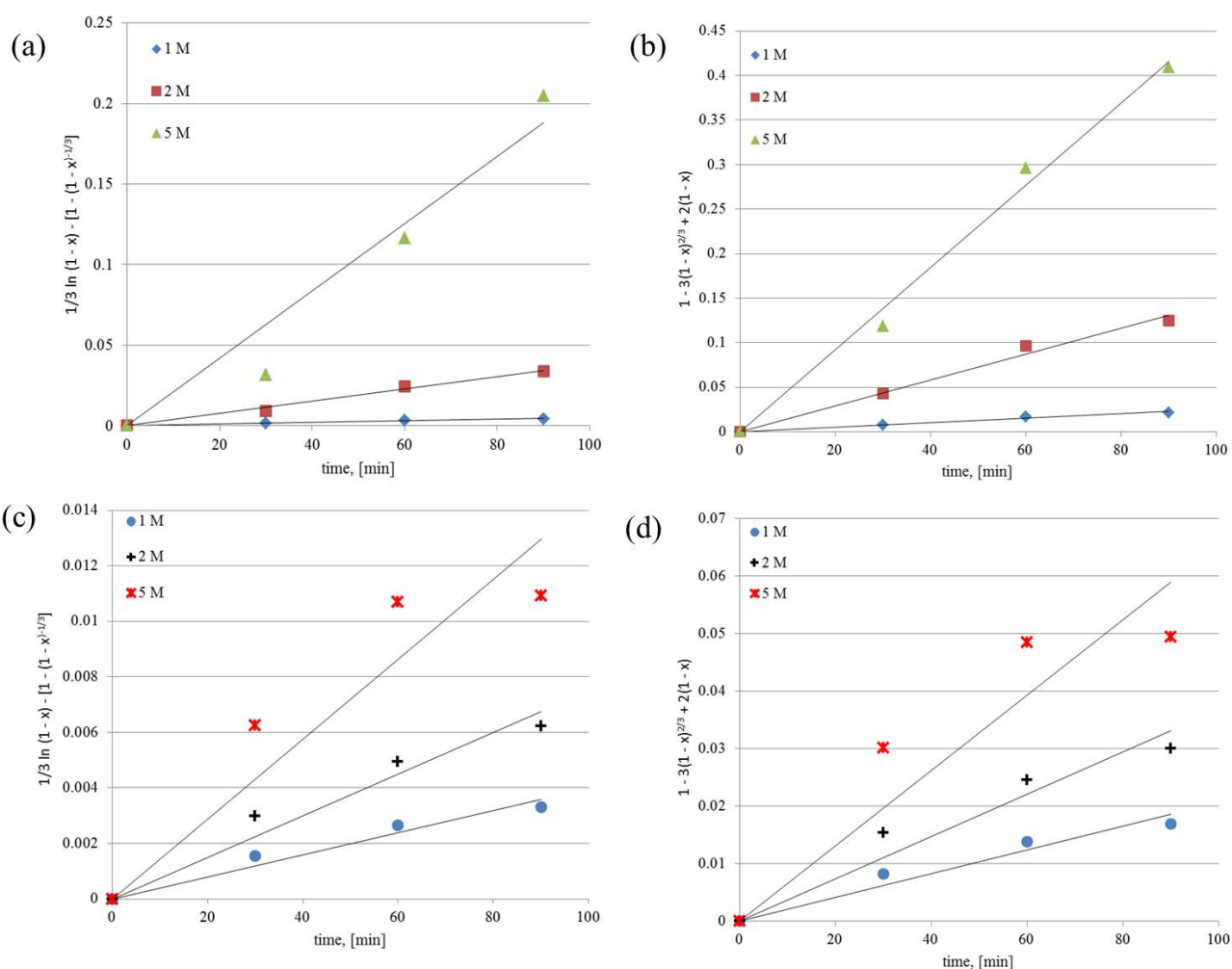


Figure 11. The course of model curves 14 and 15 relative to the experimental points obtained during the tests. (a) and (b) Cu leaching, (c) and (d) Pb leaching.

Fair equation of the model equations with experimental points was obtained in the case of copper leaching. The square of the correlation coefficient for the model described by Eq 14 varied from 0.9479 to 0.99, and for the model according to Eq 15 from 0.9829 to 0.9921. In the case of lead leaching, lower values of this indicator appear. In the first model, R^2 varies from 0.8473 to 0.9581, and in the second model from 0.8214 to 0.9451. A smaller correlation may result from the aforementioned situation in which the lead from the slag goes into solution entering into different chemical reactions which leads to the existence of lead in the solution both in the form of a cation as well as in the anionic form. Both kinetic equations were used to determine copper and lead leaching efficiency for process time extrapolated to 120 min. The following numerical values were obtained for the model according to Eq 14: $\eta_{Cu} = 85.12\%$, $\eta_{Pb} = 36.43\%$, $\alpha = 9.59$. For the model according to Eq 15, the corresponding values were: $\eta_{Cu} = 89.93\%$, $\eta_{Pb} = 44.51\%$, $\alpha = 8.29$.

The square of the correlation coefficient in relation to copper for the model according to Eq 15 at a concentration of 5 mol/dm^3 was 0.9921. It can be said with certainty that the Cu leaching efficiency after 120 min will be around 89.93%. It can be seen that the relatively small extension of the leaching time from 90 to 120 min will result, according to the results of the models used, of an increase in the leaching efficiency of both copper and lead. As a consequence of the calculations made, it can be stated that by extending the duration of the process, copper leaching efficiency of at least 90% can be obtained without great difficulty.

5. Conclusions

In the present work slag leaching was carried out using ammonium acetate solutions due to the selective action of this reagent. The influence of process time, temperature, leaching solution concentration and l/s ratio on copper and lead leaching efficiency as well as the selectivity of this process were investigated. The concentration of the ammonium acetate solution clearly influences the leaching efficiency of Cu and Pb. In the studied temperature range, no significant effect of this parameter on copper leaching efficiency was noted. It was different in relation to lead leaching efficiency—an increase in the leaching efficiency of this element was noted along with the increase of process temperature. The tests carried out as part of this work allowed the determination of optimal, from the point of view of copper leaching, process parameters: solution concentration is the 5 mol/dm^3 , time is the 90 min, temperature is the $50 \text{ }^\circ\text{C}$ or $70 \text{ }^\circ\text{C}$, $l/s = 10$. The use of two kinetic models for description of the obtained experimental data allowed determination of copper and lead speed constants. Extrapolation of experimental points in accordance with the obtained model equations led to the thesis about the possibility of obtaining 90% efficiency of the copper leaching process by extending the process time to about 120 min.

Conflict of interest

The author declares no conflict of interest.

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