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Study of the Phase Composition and Kinetics of the Metal Leaching Process Using Electrohydropulse Discharge on the Waste from the Zhezkazgan Concentrating Plants

This work involved conducting X-ray phase analysis and energy-dispersive spectroscopy (EDS), for the first time, as well as examining the kinetics of metal extraction processes from the tailings of the Zhezkazgan Concentration Plant (ZhCP) before and after their treatment. Based on the results of X-ray diffraction results, the main diffraction peaks have been found to corresponded to silicon-containing or silicate minerals, as well as phases characteristic of Cu_2S, $CuFe_2S_3$, and ZnS. Following electrohydropulse discharge leaching, the silicates (SiO_2) were partially destroyed, releasing metal oxides. The elemental composition of the tailings was analyzed using electron probe X-ray microanalysis. The EDS analysis revealed the presence of 13 elements, the most intense lines of which were silicon, oxygen, and aluminum, while the remaining elements were found in minor quantities. The activation energy was determined graphically using the Arrhenius equation based on the rate constants (ln*K*) at various temperatures and exposure times to electrohydropulse discharge. To determine the kinetics of the leaching process, a high-temperature thermometer was installed on the reactor to record the temperature of the slurry, including the solution reaction mixture. During the hydropulse discharge process, the temperature of the reaction medium rose to 60 °C over 30 minutes.

Keywords: hydropulse discharge, pulp, laboratory cell, ammonium bifluoride, tailings samples, metals, acid, copper

Introduction

The metallurgical industry produces large quantities of waste, including tailings and process residues that present significant environmental and economic challenges. Efficiently recycling and repurposing of these materials is essential in order to reduce ecological impact and recover valuable components. Without proper management, these industrial by-products can contribute to soil and water contamination, disrupting ecosystems, and leading to the loss of potentially useful raw materials. Processing metallurgical tailings and waste using physical and chemical methods has attached considerable attention due to growing environmental concerns and the potential for resource recovery. Metallurgical processes produce a various types of waste, such as slags, dusts, and sludges, which can be repurposed using innovative recycling techniques.

Metallurgical waste, such as slag and tailings, are often contains valuable metals and minerals. For instance, that mining and metallurgical waste can be effectively repurposed as construction materials, emphasizing the importance of physical beneficiation processes alongside hydrometallurgical and pyrometallurgical methods for recycling these materials [1, 2]. The evaluation of sulphation baking and autogenous leaching of Turkish metallurgical slag flotation tailings further illustrates the potential of these wastes as valuable raw materials, particularly in the context of declining ore grades and increasing metal prices [3]. These findings align with those of other authors, who discuss the transformation of hazardous metallurgical waste into resources and showcase various harmless treatment and utilization methods [4, 5].

The utilization of metallurgical waste in construction materials is also a prominent theme in the literature. For example, the use of metallurgical waste slag as a cementitious material, which not only contributes to sustainable construction practices and addresses the environmental impact of waste disposal [6]. Similarly, other research emphasizes the role of metallurgical wastes in promoting sustainability within the steel industry, highlighting the diverse types of waste generated and their potential applications [7, 8].

Environmental considerations are paramount in the processing of metallurgical wastes. A review emphasizes the marketability of blast furnace and steelmaking slag, which can be utilized in various applications, thereby reducing landfill waste and promoting resource conservation [9]. Furthermore, the environmental impacts of copper slag are discussed with the highlighting of the need for effective management strategies to mitigate the risks associated with waste accumulation [10].

Chemical methods for processing metallurgical waste are also crucial for recovering resources. For instance, metals can be recovered from waste-derived copper-lead electrocatalysts, demonstrating the potential to convert waste into valuable products through chemical processes [11, 12]. Another study explores the extraction of valuable metals such as lead, zinc, and copper from metallurgical waste using chloride distillation, showcasing the effectiveness of chemical approaches in waste management [13, 14]. The importance of understanding the properties of metallurgical waste in order to determine suitable recycling methods is also emphasized, as are the challenges that arise from insufficient knowledge of waste characteristics during the recycling process [15, 16].

A comprehensive understanding of the characteristics of metallurgical waste informs suitable recycling methods and highlights the need to advance processing technologies. While established methods for handling depleted ore and mining waste, particularly tailings from processing plants, provide a foundation, further innovation is necessary to improve the recovery of resources.

In hydrometallurgical industries, efficient leaching techniques, such as those utilizing ammonium salts, have proven effective for extracting copper from processing plant waste [17]. Fluorine-containing components are widely used as additives and fluxes in the processing of various ores, in the separation of some rare and rare-earth metals, as well as in electrolysis. Ammonium hydrodifluoride is an effective reagent for processing mineral raw materials containing non-ferrous and noble metals resistant to simple fluorination. It facilitates the separation of valuable components by converting raw materials into a mixture of simple and complex fluorides. Up to 90 % of the ammonium fluoride used in fluorination can be recovered for NH_4HF_2 regeneration, supporting a closed technological cycle.

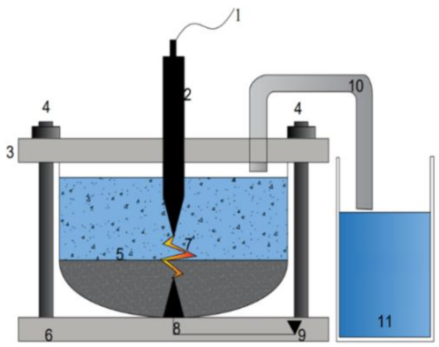
The regeneration of NH_4HF_2 enables a closed, environmentally friendly production cycle. Ammonium hydrodifluoride can process polymetallic raw materials effectively at temperatures below 200 °C, and the resulting fluorination by-products ensure environmental safety [18]. Copper leaching from chalcopyrite has been demonstrated using aqueous ammonia and glycine, with ceramic chips aiding particle breakdown [19]. NH_4HF_2 facilitates the extraction of valuable metals, including gold and silver, using closed-loop bifluoride processes. Compared to chlorine metallurgy, these methods operate at lower temperatures with greater environmental safety and simpler reagent regeneration. Ammonium bifluoride is a promising reagent for processing rare metal and technogenic raw materials. Studies confirm its effectiveness in isolating rare earth elements and calcium fluoride CaF_2 [20, 21].

Experimental

Effective extraction of valuable metals from metallurgical waste requires optimized treatment methods that facilitate the breakdown of mineral structures and promote metal solubility. In this study, electrohydropulse (EHP) processing was applied to waste samples from the Zhezgazgan concentrating plant to assess its efficiency in metal recovery. The process involved high-voltage discharge in a liquid medium to induce structural disruption and enhance leaching. For each experiment, 300 g of tailing samples (stale ZhCP No. 1.2, current ZhCP No. 1.2, and stale ZhCP No. 3) were placed in a stainless steel reaction cell with a total volume of 1 liter. To establish the required reaction conditions, 500 ml of water was added along with 15 ml of H_3PO_4, adjusting the medium's acidity to pH 1–2. Additionally, 4 g of NH_4HF_2 was introduced as an activator to promote the decomposition of silicate structures and enhance metal release. The prepared pulp was then subjected to an electrohydropulse discharge for 30 minutes to facilitate the transfer of metals into solution. Following treatment, the solution was filtered to remove solid residues.

The electrohydropulse discharge was generated using a high-voltage charged capacitor, which was discharged into water within a controlled laboratory cell [22]. The electrohydropulse treatment setup included a system capable of accumulating and releasing high-voltage energy, generating powerful pulsed discharges. The anode had a conical shape with an insulated tip, while the cathode was a cylindrical reaction cell with a central protrusion at its base. The energy stored in the system allowed for effective disruption of mineral structures. The parameters of the high voltage were optimized to ensure a stable and efficient process. The operating parameters were as follows: switch operating voltage — 30 kV; pulse frequency — 0.6 Hz; pulse energy — 200 J; voltage amplitude — 30 kV; voltage rise rate — 0.005–5 μs; type of electric discharge — spark discharge in liquid; pulse duration — 100 μs.

The reaction medium was acidified to enhance the efficiency of metal leaching. A schematic of the experimental setup is shown in Figure 1.



1 — wire connected to a spark gap; *2, 8* — electrodes; *3, 6* — retaining plates made of textolite; *4* — anchor bolts; *5* — pulp; *7* — plasma cord; *9* — grounding; *10* — gas outlet tube; *11* — glass with a solution

Figure 1. Scheme of a laboratory cell for studying the effect of electrohydropulse discharge

Dried tailing samples — stale ZhCP No. 1.2, current ZhCP No. 1.2, and stale ZhCP No. 3, both before and after treatment — were analyzed using X-ray phase analysis conducted on a powder diffractometer. This investigation revealed the phase composition and structural changes resulting from electrohydropulse treatment. This method provided a comprehensive evaluation of the mineralogical transformations and the efficiency of the processing technique. In order to understand how the metals are distributed in the samples, X-ray phase studies were carried out using a powder diffractometer D8 AdvanceEco, Brucker. Recording mode Bragg-Brentano geometry, angular range from 15 to 75°, with a step of 0.03°, spectrum acquisition time 2 s X-rays were generated using a copper tube with a wavelength Cu-kλ = 1.5406 Å. DiffracEVA v.4.2 Decryption Software, PDF-2 Matching Phase Search Database (2016). The weight ratio of the phases was assessed using the standard equation (1), which is based on determining the values of the integral intensities and estimating their contributions.

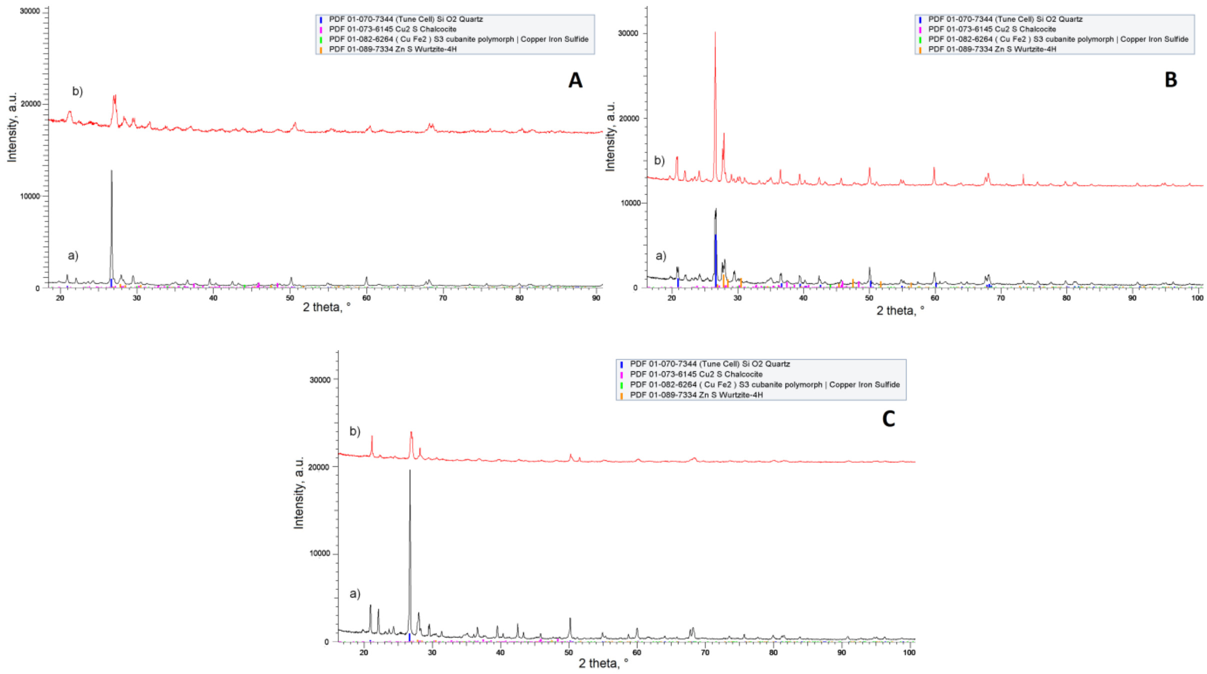
$$V_{\text{admixture}} = \frac{RI_{\text{phase}}}{I_{\text{admixture}} + RI_{phase}}, \tag{1}$$

where I_{phase} is the average integrated intensity of the main phase of the diffraction line, $I_{\text{admixture}}$ is the average integrated intensity of the additional phase, R is the structural coefficient equal to 1.45.

In order to study the thermodynamic aspects of the leaching process, a setup was arranged to precise monitoring temperature changes. A high-temperature thermometer integrated into the reactor enabled the temperature increase of the pulp and reaction mixture solution to be recorded. During the electrohydropulse treatment, which lasted 30 minutes, the temperature of the reaction medium gradually increased to 60 °C, indicating the active progression of chemical processes. Furthermore, the activation energy of the reactions was determined using a graphical method based on the Arrhenius equation, which enabled a quantitative evaluation of the kinetic parameters.

Results and Discussion

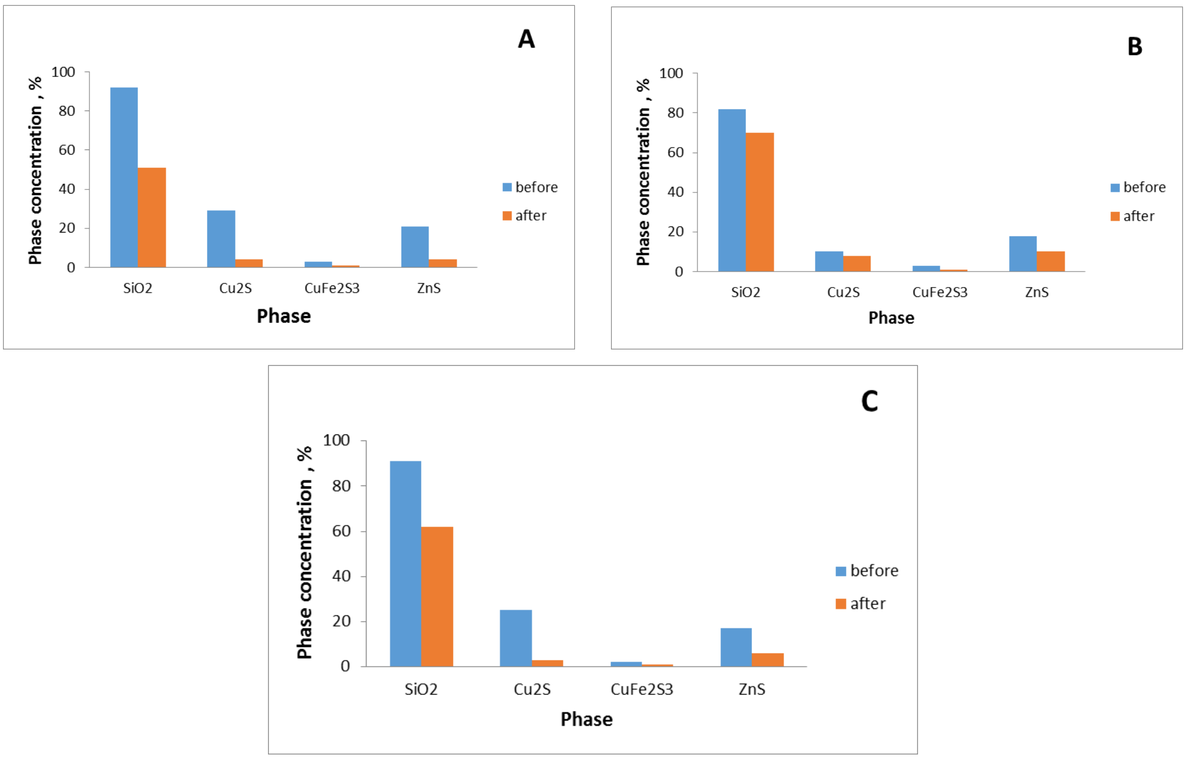
Figure 2 shows the results of X-ray diffraction of the studied samples before and after leaching of ZhCP No. 1, 2, 3. According to the results of X-ray diffraction, it was found the main reflections, with the highest intensity, are characteristic of silicon dioxide (SiO_2 — quartz) appropriate to most silicon-containing or silicate rocks. Diffraction reflections typical to the Cu_2S, $CuFe_2S_3$ and ZnS phases have also been established. No reflections characteristic of $CaSiO_3$ phases were found. The determination of the phase composition, as well as the concentration of the phases, was carried out using the equation (1).



A — ZhCP No. 1.2 stale; *B* — ZhCP No. 1.2 current; *C* — ZhCP No. 3 stale

Figure 2. X-Ray diffraction patterns of ZhCP tailings before (*a*) and after leaching (*b*)

As a result of treatment with ammonium bifluoride, the X-ray spectra show that SiO_2 silicates were partially destroyed and released the metal oxides, which is clearly visible in terms of the height of the peaks. Additionally, the spectra show that the amount of metals decreases in those particles of impurities of metal oxides after treatment with HPD.



A — ZhCP No. 1.2 stale; *B* — ZhCP No. 1.2 current; *C* — ZhCP No. 3 stale

Figure 3. Results of changes in the phase composition

The presented diagrams (Figure 3) show that in the dried sediment after leaching, a decrease in the content of phases characteristic of the ZhCP 1,2 (A) SiO_2 from 94 % to 50 %, Cu_2S from 30 % to 5 % and $CuFe_2S$ from 5.1 % to 0.3 %. ZnS from 18.7 % to 4.1 %. is observed. The diagram also shows a depletion of the metals content in the sediment after leaching, which characterizes their transition into solution.

One method of studying sediment composition is electron probe X-ray spectral microanalysis (EP-XRSMA). In this study, we present the results obtained using a JXA8200 microanalyser (JEOL, Japan) in the X-ray methods of analysis laboratory.

Based on phase composition data, samples of the ZhCP and KCP tails were subjected to X-ray spectral EMF analysis. Thirteen elements (Cu, Fe, Si, Al, Mn, Cr, Mg, Ca, Ba, O, Na, K and S) were recorded in the energy dispersive spectra of the studied sample sections prior to HPD treatment (Figure 4).

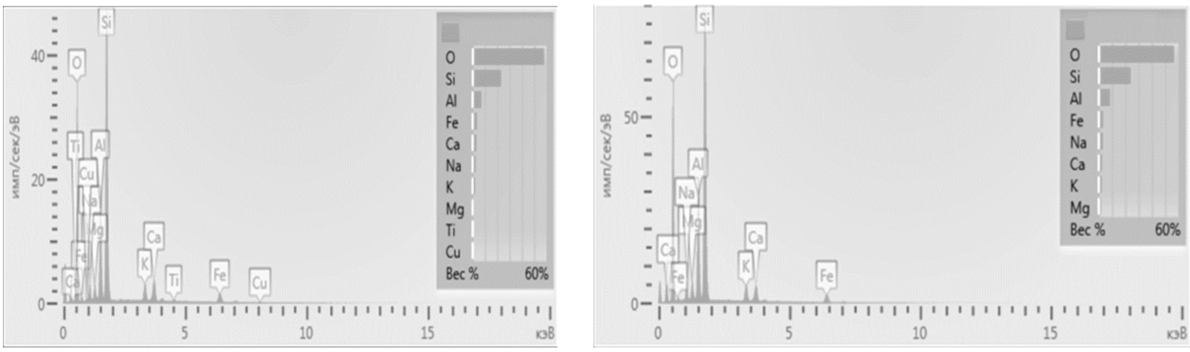


Figure 4. X-ray spectral EMF analysis of the dried sediment before and after HPD ZhCP No. 1.2 Current

The spectra showed the presence of silicon, oxygen and aluminium in varying intensities. The remaining elements are present in small quantities.

The spectrum before HPD (Fig. 5) shows the intensities of the lines of sulphur, oxygen, magnesium, iron, copper and zinc, as well as a significant number of other elements.

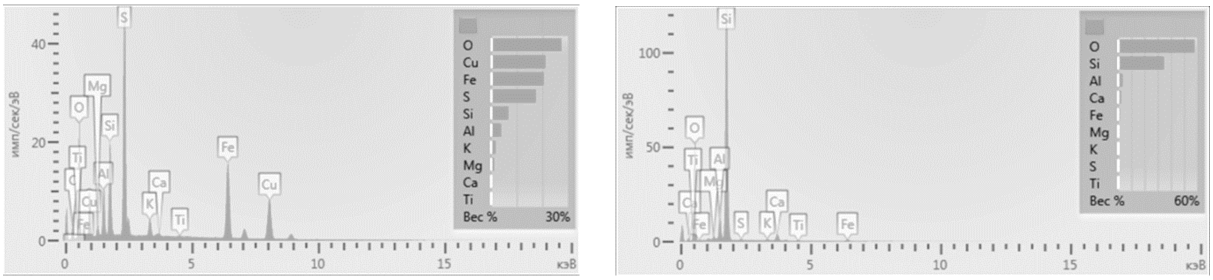


Figure 5. X-ray spectral EMF analysis of the dried sediment before and after HPD ZhCP No. 1.2 stale

The spectrum after HPD (Fig. 5) shows the intensity of the silicon, aluminium and oxygen lines, with the remaining elements present in small amounts. The spectrum (Fig. 6) before HPD treatment shows the intensities of the copper, carbon, oxygen and sulfur lines.

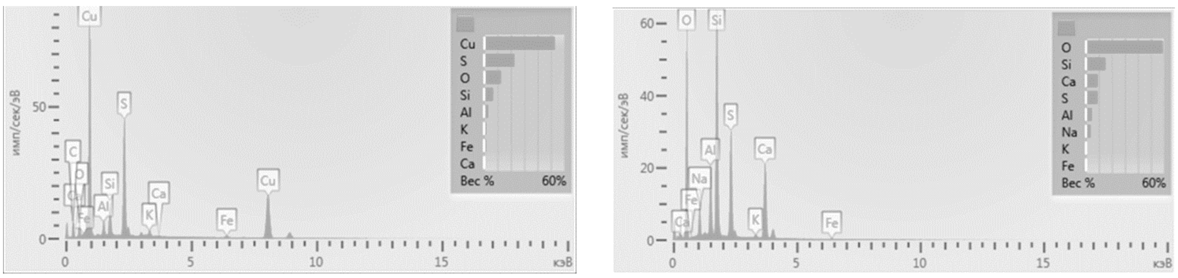


Figure 6. X-ray spectral EMF analysis of the dried sediment before and after HPD ZhCP No. 3 stale

The EMF spectra show that the concentration of copper and other elements decreases after the leaching and processing of HPD. The elements detected in the spectra transition into solution. These elements are all in solution in the form of complexes, which must be precipitated using reagents to keep the copper ions in solution. The process by which copper transitions into solution is complex and staged, beginning with the opening of the silicate surface under the influence of ammonium hydrodifluoride. [23]:

$$SiO_2 + 3NH_4HF_2 \rightarrow (NH_4)_2SiF_6 + NH_3 + 2H_2O \quad (2)$$

The remaining sediment consists of complex conglomerates of particles of various sizes and compositions. The concentrations of all the studied elements (Fe, Zn, Pb, Cu, S, Si, Al, Na, Mg, K, Ca, Ti, Cr and Mn) fluctuate significantly. Spectra obtained from different particles at different points all contain lines of carbon and oxygen. Rather than forming compounds, these elements form mechanical mixtures bonded in the form of complexes containing oxygen and carbon.

The experiment results on duration of the experiments in minutes and on the temperature dependence have been processed mathematically to obtain the rate constant of copper leaching. This rate constant has been converted the logarithmic form in order to determine the activation energy using the Arrhenius equation.

Table 2

Copper leaching rate constants at different temperatures

Copper leaching rate constants	Temperature, °C				
	36	42	48	54	60
K	0,05204	0,05973	0,06353	0,066439	0,073857
1/*T*	0,003236	0,003174	0,003115	0,003058	0,003003
ln *K*	–2,9556	–2,8178	–2,7562	–2,7114	–2,6056

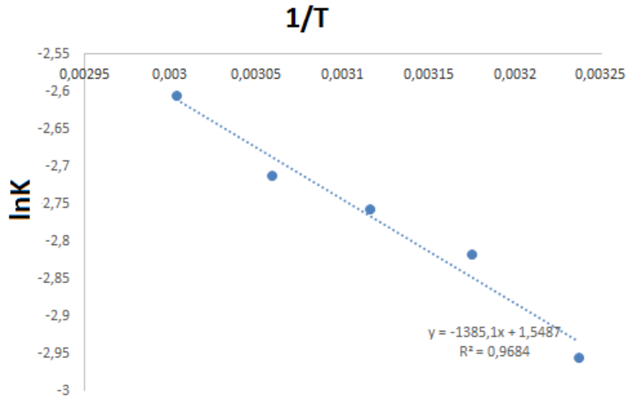


Figure 7. Rate constant versus reciprocal temperature

The activation energy is determined using a graphical method. Knowing the rate constants (ln*K*) at different temperatures from the HPD time can be used to calculate the activation energy (E_A). The Arrhenius function was built using the table data (Fig. 7), according to which the activation energy was determined. The activation energy was calculated using the Arrhenius equation:

$$\ln K = A_0 e^{-E_A/RT}, \quad (3)$$

where A_0 is the pre-exponential factor (does not depend on temperature); R is the universal gas constant; $R = 8.314$ J/(K*mol); K — rate constant s^{-1}, E_A — activation energy, $E_A = 1385.1*8.314 = 11515.72$ J/mol = = 11.52 kJ/mol.

The process of copper leaching from the tailings of the ZhCP proceeds in a mixed region with a predominance of diffusion factors, as evidenced by the obtained value of the activation energy. When extracting copper from the tailings of the ZhCP, it is necessary to carry out intensive mixing of the feedstock for the best extraction.

Conclusions

For the first time, the tailings of the Zhezkazgan Concentrating Plant were studied using X-ray phase analysis and energy-dispersive spectroscopy (EDS) analysis, as well as the kinetics of the leaching process. The conducted research allowed us to determine changes in the phase composition of the minerals in the tailings, which occurred as a result of electrohydropulse treatment. The applied method provided a comprehensive assessment of mineralogical changes and the effectiveness of the treatment.

Based on the obtained data, the following conclusions were made:

The combined application of electrohydropulse discharge and the ammonium biftrate activator demonstrated the destruction of silicon-containing rocks.

Energy-dispersive spectroscopy (EDS) revealed the presence of thirteen elements, the most intense lines being those of silicon, oxygen, aluminium and various non-ferrous metals. Following electrohydropulse discharge (EGIR) treatment, a significant decrease in copper concentration in the tailings minerals was observed, indicating a transition of copper ions into solution.

This study investigated the kinetics of copper leaching at low temperatures (up to 60 °C) over a 30-minute treatment period. Graphical analysis based on the Arrhenius equation was used to determine the activation energy value from the Zhezkazgan Concentrating Plant tailings, which amounted to 11.52 kJ/mol. It was established that this process occurs in the diffusion region and can be effectively carried out at relatively low temperatures.

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Author Contributions

The manuscript was written through contributions of all authors. All authors have given approval to the final version of the manuscript. CRediT: **Askhat Sakenovich Borsynbayev** conceptualization, investigation, data curation, investigation, validation, visualization, writing-review & editing; **Yedige Suindikovich Mustafin** conceptualization, methodology, data curation, supervision; **Khylysh Beysenovich Omarov** conceptualization, methodology, data curation, supervision; **Aigul Akizhanovna Muratbekova** writing-review & editing, resources; **Dauletkhan Asanovich Kaykenov** formal analysis, validation, data curation; **Daniyar Tleuzhanovich Sadyrbekov** formal analysis, data curation; **Asanali Anuarovich Aynabayev** formal analysis, investigation, data curation; **Abylaikhan Nurlanuly Bolatbay** data curation, formal analysis; **Milana Aleksandrovna Turovets** data curation, writing — Review & Editing.

Conflicts of Interest

The authors declare no conflict of interest.

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