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NON-CONVENTIONAL HYDROMETALLURGICAL SEPARATION OF IRON, ZINC, AND COPPER FROM COPPER INDUSTRY WASTE



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Abstract. *In this study, the possibilities of efficiently separating iron, zinc, and copper from copper smelting slag, which is a waste of the copper industry, using a non-conventional hydrometallurgical method were investigated. The recycling of such wastes is important not only for the recovery of valuable metals but also for reducing their negative impact on the environment. In order to eliminate technological problems that arise during the filtration stage after sulfuric acid leaching of copper smelting slag, the research was carried out in three stages. First, the slag sample was selectively leached with sulfuric acid; in the second stage, the obtained solution was dried; and in the final stage, the resulting solid metal sulfates were selectively leached with water.During the sulfuric acid leaching process, the effects of acid concentration, leaching time, temperature, and stirring speed on the transfer of metals into the solution were comprehensively investigated. The experimental results showed that an acid concentration of 2 M, a leaching time of 120 minutes, a temperature of 90 °C, and a stirring speed of 300 rpm are optimal conditions for metal dissolution. Under these conditions, the degree of metal dissolution into the solution was 89.5% for Cu, 88.7% for Zn, and 59.2% for Fe. The solution obtained after selective leaching was dried at 150 °C for 90 minutes to obtain solid metal sulfates. In the next stage, this product was selectively leached with water, and it was determined that a leaching time of 40 minutes and a temperature of 40 °C are the most favorable conditions for the transfer of metals into the solution.*

Keywords: *Copper smelting slag; selective leaching; sulfuric acid leaching; metal sulfates; water leaching; non-ferrous metals; hydrometallurgy; extraction efficiency.*

НЕТРАДИЦИОННОЕ ГИДРОМЕТАЛЛУРГИЧЕСКОЕ РАЗДЕЛЕНИЕ ЖЕЛЕЗА, ЦИНКА И МЕДИ ИЗ ОТХОДОВ МЕДНОЙ ПРОМЫШЛЕННОСТИ

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Аннотация. *В данной работе исследованы возможности эффективного извлечения железа, цинка и меди из медеплавильного шлака, являющегося отходом медной промышленности, с использованием нетрадиционного гидрометаллургического метода. Переработка подобных отходов имеет важное значение не только для возврата ценных металлов, но и для снижения их негативного воздействия на окружающую среду. С целью устранения технологических проблем, возникающих на стадии фильтрации после сернокислотного выщелачивания медеплавильного шлака, исследования проводились в три этапа. На первом этапе образец шлака подвергался селективному выщелачиванию серной кислотой, на втором этапе полученный раствор высушивался, а на заключительном этапе образовавшиеся твердые сульфаты металлов селективно выщелачивались водой. В процессе сернокислотного выщелачивания комплексно изучено влияние концентрации кислоты, продолжительности процесса, температуры и скорости перемешивания на степень перехода металлов в раствор. Экспериментальные результаты показали, что концентрация кислоты 2 М, продолжительность выщелачивания 120 минут, температура 90 °C и скорость перемешивания 300 об/мин являются оптимальными условиями для растворения металлов. При этих условиях степень растворения металла в растворе составила 89,5% для Cu, 88,7% для Zn и 59,2% для Fe. Раствор, полученный после селективного выщелачивания, высушивали при температуре 150 °C в течение 90 минут с получением твердых сульфатов металлов. На следующем этапе данный продукт селективно выщелачивали водой, при этом установлено, что продолжительность процесса 40 минут и температура 40 °C являются наиболее благоприятными условиями для перехода металлов в раствор.*

Ключевые слова: *медеплавильный шлак; селективное выщелачивание; сернокислотное выщелачивание; сульфаты металлов; водное выщелачивание; цветные металлы; гидрометаллургия; эффективность извлечения.*

MIS SANOATI CHIQINDILARIDAN TEMIR, RUX VA MISNI NOAN'ANAVIY GIDROMETALLURGIK AJRATIB OLISH

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Annotasiya. *Ushbu tadqiqotda mis sanoati chiqindisi hisoblangan mis eritish shlagi tarkibidan temir, rux va mis metallarini noan'anaviy gidrometallurgik usulda samarali ajratib olish imkoniyatlari o'rganildi. Mazkur chiqindilarni qayta ishlash nafaqat qimmatli metallarni qayta tiklash, balki atrof-muhitga salbiy ta'sirni kamaytirish nuqtayi nazaridan ham dolzarb hisoblanadi. Sulfat kislotada tanlab eritish jarayonidan so'ng filtrlash bosqichida yuzaga keladigan texnologik muammolarni bartaraf etish maqsadida tadqiqot ishlari uch bosqichda olib borildi. Dastlab, shlak namunasi sulfat kislotada tanlab eritildi, keyingi bosqichda olingan eritma quritildi va oxirgi bosqichda hosil bo'lgan qattiq metall sulfatlari suvda tanlab eritildi. Sulfat kislotada tanlab eritish jarayonida kislota konsentratsiyasi, jarayon davomiyligi, harorat va aralashtirish tezligining metallarning eritmaga o'tish darajasiga ta'siri kompleks ravishda o'rganildi. Tajriba natijalari shuni ko'rsatdiki, 2 M kislota konsentratsiyasi, 120 minut davomiylik, 90 °C harorat va 300 ayl/min aralashtirish tezligi metallarning eritmaga o'tishi uchun optimal sharoitlar hisoblanadi. Ushbu sharoitlarda metallning eritmaga erish darajasi Cu uchun 89,5%, Zn uchun 88,7% va Fe uchun 59,2% ni tashkil etdi. Tanlab eritish natijasida olingan eritma 150 °C haroratda 90 minut davomida quritilib, qattiq metall sulfatlari olindi. Keyingi bosqichda ushbu mahsulot suvda tanlab eritilib, 40 minut davomiylik va 40 °C harorat metallarning eritmaga o'tishi uchun eng maqbul sharoit ekanligi aniqlandi.*

Kalit so'zlar: *mis eritish shlagi; tanlab eritish; sulfat kislotada eritish; metall sulfatlari; suvda eritish; rangli metallar; gidrometallurgiya; ekstraksiya samaradorligi.*

Introduction

Copper slag is a by-product formed in the pyrometallurgical processing of copper, consisting mainly of fayalite (Fe_2SiO_4), magnetite (Fe_3O_4), and minor amounts of valuable metallic elements such as copper, iron, zinc, cobalt, and nickel [1,2]. In addition, some copper slags may contain harmful elements such as lead and arsenic [3]. It is considered an important hazardous solid waste that can be utilized for secondary purposes. According to statistics, while the annual global copper production volume is 23 million tons, the annual volume of copper slag waste has exceeded 70 million tons [4]. Currently, due to the lack of effective and safe processing and neutralization measures, the majority of copper slags are accumulating around smelting plants. Only 20% are being used in the production of high-value products, including cement additives, fillers, road surface slag, abrasives, cutting tools, inert aggregates, roofing granules, glass, and tiles [5]. This situation not only leads to the wastage of high-value resources such as iron, cobalt, copper, lead, and silicon but also poses a serious threat to ecological safety, human health, and sustainable development. The heavy metal elements in copper slag can disperse into the surrounding soil, groundwater, and open water bodies, causing significant environmental hazards [6]. Currently, there are three main methods for recovering valuable metals from copper slag: froth flotation, pyrometallurgy, and hydrometallurgy. In the flotation process, the surface property differences between valuable and gangue minerals are exploited to recover copper and cobalt. This process utilizes various reagents, such as frothers, modifiers, and depressants. However, the efficiency of flotation and the grade of the copper concentrate depend on the thermal history (cooling rate) of the slag, which can lead to significant losses of residual copper and cobalt during the process [7,14]. Hydrometallurgy primarily involves transferring valuable metallic elements from copper slag into a liquid phase. This process is carried out using alkaline (strong base) or acidic solutions. Subsequently, the separation and enrichment of various valuable metals present in the liquid phase are performed, ultimately leading to their recovery in the form of metals or other compounds [8]. The selective leaching method using inorganic acids has attracted significant attention from researchers due to its high separation efficiency. Although some researchers have conducted systematic studies on the selective leaching of copper slag using alkaline or organic acids, the separation efficiency has not reached the desired level. For example, Sari et al. [9] investigated the leaching behavior of metals from copper slag using hydrogen peroxide and an organic acid (acetic acid) under atmospheric pressure. Under optimal conditions, the separation efficiency for copper, zinc, and iron reached 98.04%, 28.8%, and 2%, respectively. Meshram et al. [10] studied the selective leaching of metals from copper slag using citric acid, achieving recovery rates of 99.0% for copper, 89.2% for nickel, 94% for cobalt, and 99.2% for iron under optimized conditions. Acids such as HCl and HNO_3 release highly toxic gases like Cl_2 and NO_x during the selective leaching process due to their inherent instability. These gases not only lead to environmental pollution but also pose serious risks to human health, as they can disperse through the air and cause damage to the respiratory system and other organs [11].

Sulfuric acid has been favorably received by researchers as a leaching agent for metal recovery from copper slag. However, this method still presents certain unresolved challenges. If some stable compounds or soluble phases within the slag are encapsulated within insoluble minerals, it can reduce the recovery efficiency of metals such as iron, cobalt, copper, and zinc [12,15]. The process can be optimized by adjusting various parameters, such as the leaching system, acid concentration, temperature, and time. However, the selective leaching process using sulfuric acid becomes more prone to silica gel formation when the H_2SO_4 concentration, leaching temperature, and duration are increased [13,16]. To address the aforementioned issues, this study proposes a combined method of "sulfuric acid pretreatment + drying + water leaching" for recovering valuable components from copper slag. Using single-factor experiments, the effects of pretreatment conditions—temperature, acid concentration, liquid–solid ratio, and time—as well as drying temperature and duration on the selective leaching efficiency of non-ferrous metals from the slag were investigated in detail. Comparisons were made with the conventional sulfuric acid selective leaching process, and the filtration characteristics of the leaching solution were analyzed. As a result, the proposed combined process demonstrated the potential for efficient, safe, and energy-saving processing of copper slag, offering a novel and promising method for its recovery.

Materials and methods

Materials. In this study, copper slag obtained from the copper industry was used as the sample material. The samples designated for research were first crushed, ground, and prepared for chemical analysis. To determine the chemical composition of the samples, a Shimadzu EDX-7000 energy-dispersive X-ray fluorescence spectrometer was utilized. The obtained data are presented in Table 1.

Initially, a jaw crusher (Дробилка щековая ДЩ 80×150, NPK "Механобр-техника" Russia) was used to reduce the size of the slag. Subsequently, to further grind the material and reduce the particle size to less than 2 mm, a roller crusher (Валковая дробилка ДВГ 200×125, Russia) was employed.

Table 1

Chemical composition of the copper slag

Elements	Fe	Zn	Cu	Si	Al	K	Mg	Ni	Mn
%	37,9	0,74	0,86	16,4	5,31	0,43	0,25	0,07	0,5

High-purity chemical reagents were used to conduct the experimental work. A 96% sulfuric acid (H_2SO_4) solution was employed to transfer the metallic components from the slag into the leachate. Sodium hydroxide (NaOH) was used to regulate the solution's pH, while hydrogen peroxide (H_2O_2) was utilized to enhance the oxidation process. All solutions used in the experiments were prepared using distilled water.

Methods. Selective Leaching (sulfiric acid). To determine the influence of key process parameters on the leaching of metals from the slag, selective leaching experiments were conducted. During the experiments, sulfuric acid concentration (1.4–2.4 M), leaching time (90–140 minutes), temperature (70–120 °C), and agitation speed (100–350 rpm) were sequentially varied and studied. All experiments were carried out in a 1000 ml beaker using a laboratory magnetic stirrer. The temperature was continuously monitored throughout the process. An MS7-H550-S model stirrer (DLAB, Beijing, China) was used for the experiments.

Selective Leaching (water). After the selective leaching process in sulfuric acid, the resulting suspension was dried by heating without prior filtration. This yielded solid metal sulfates. In the subsequent stage, this solid product was leached in plain water to study the selective leaching process in an aqueous environment. During the water leaching experiments, the focus was on key process parameters: leaching duration (30–80 minutes), temperature (30–80 °C), and the solid-to-liquid ratio (S/L), which were sequentially varied. All experiments were conducted under laboratory conditions.

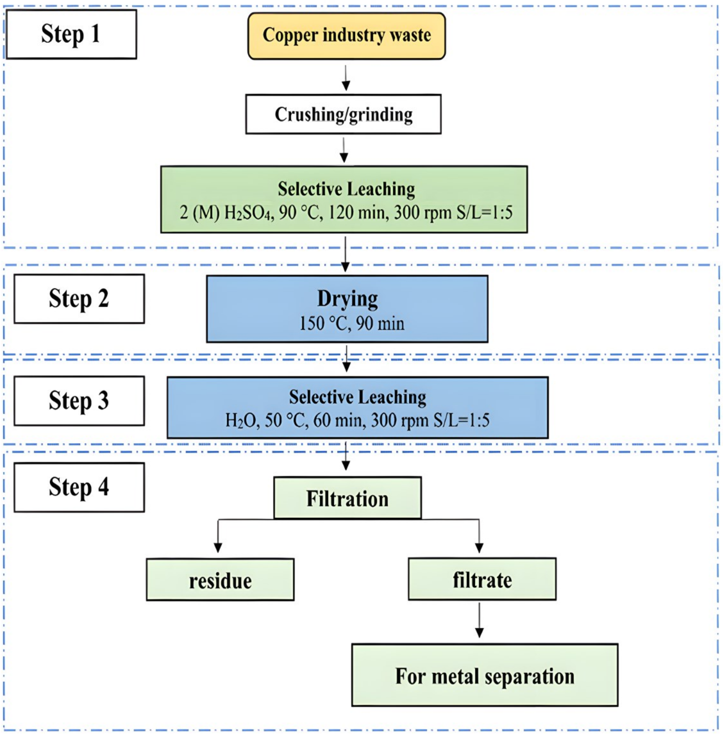


Fig. 1. The flow chart of the recovery process

A simplified process flow diagram illustrating the overall sequence of operations performed in this study is presented in Figure 1. This diagram provides a clear and comprehensive visual representation of the experimental procedure.

Results and discussion

Determining optimal parameters for selective dissolution in sulfuric acid. To begin the experimental work, six samples of 100 g each were weighed using a balance. Each sample was processed in sulfuric acid solutions with different concentrations: 1.4, 1.6, 1.8, 2.0, 2.2, and 2.4 M. In the first stage, research was conducted in order to convert the metal compounds contained in the sample into sulfate form.

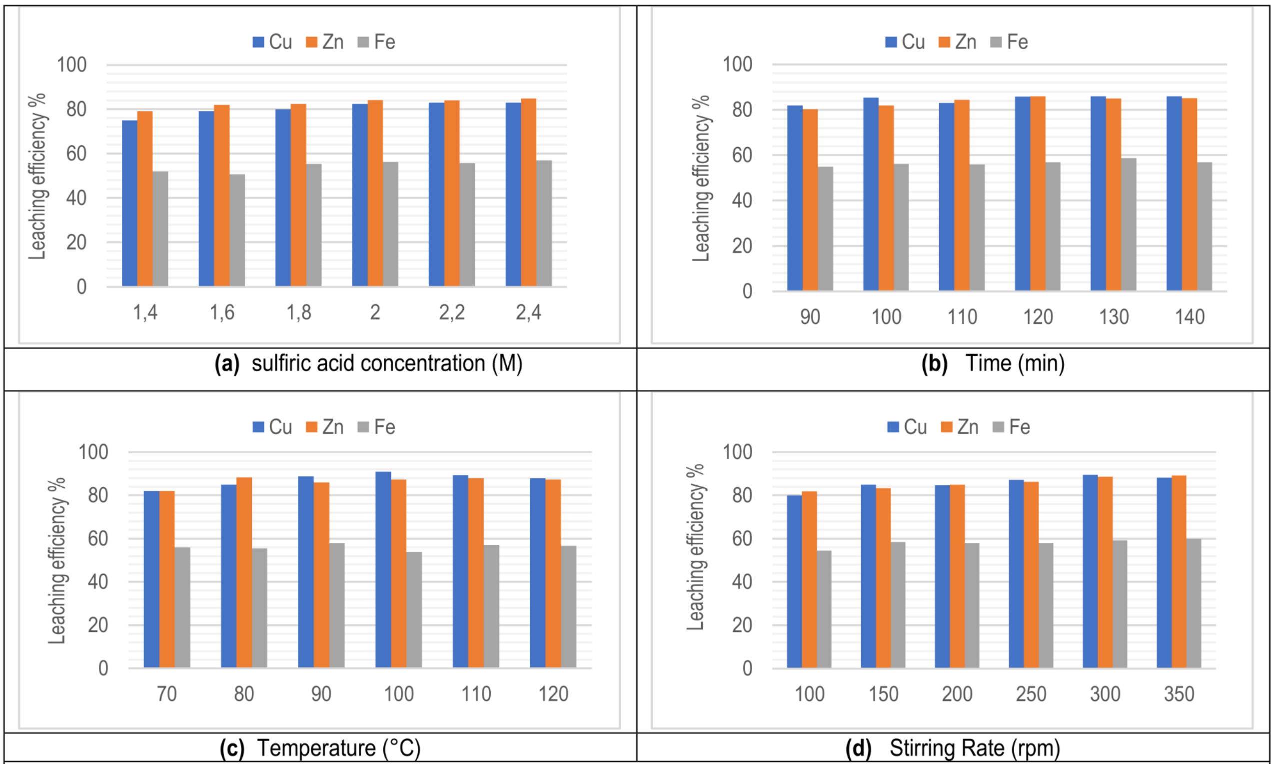
In this case, initially, experiments were carried out to determine the optimal value of the solution concentration. The sulfuric acid concentration was varied in the range of 1.4–2.4 M, while the other process parameters were kept constant: leaching time of 100 minutes, temperature of 80 °C, stirring speed of 300 rpm, and a solid-to-liquid phase ratio of 1:5. Through this sequence of experiments, the effect of sulfuric acid concentration on the degree of m tal transfer into the solution was systematically evaluated. The obtained results served as a basis for analyzing the process efficiency and determining the optimal operating conditions. In particular, at a sulfuric acid concentration of 1.4 M, the degrees of metal transfer into the solution were 75% for copper, 79.1% for zinc, and 52% for iron. When the acid concentration was increased to 2 M, the metal transfer efficiencies increased to 82.5% for Cu, 84.2% for Zn, and 56.3% for Fe, respectively. However, further increases in sulfuric acid concentration did not have a significant effect on the degree of metal transfer into the solution. The experimental results are presented graphically in Figure 2a.

After determining the optimal concentration of sulfuric acid, a series of experiments was conducted to study the time parameter, which is one of the important factors affecting the process. In each experiment, the solution concentration was maintained at 2 M, the selective leaching time was varied in the range of 90 –140 minutes, the temperature was kept at 80 °C, the stirring speed was 300 rpm, and the solid-to-liquid phase ratio was maintained at 1:5. Through these studies, the effect of selective leaching duration on the degree of metal transfer into the solution was systematically analyzed. The obtained results served as an important basis for evaluating the process efficiency and determining the optimal time parameter. In particular, when the selective leaching duration was 90 minutes, the degrees of metal transfer into the solution were 82% for Cu, 80.3% for Zn, and 55% for Fe. When the leaching time was extended to 120 minutes, these values increased to 85.9% for Cu, 86% for Zn, and 57% for Fe, respectively. The experimental results obtained are presented graphically in Figure 2b.

After determining the optimal duration for the selective leaching process, in the next stage the effect of temperature, which is one of the important factors significantly influencing the process, was studied. For this purpose, a series of experiments was conducted in which the temperature was varied in the range of

70–120 °C. The remaining technological parameters were kept constant: solution concentration of 2 M, selective leaching time of 120 minutes, stirring speed of 300 rpm, and a solid-to-liquid phase ratio of 1:5. Under these conditions, the effect of process temperature on the degree of metal transfer into the solution was evaluated. According to the research results, when the temperature was increased to 90 °C, the degrees of metal transfer into the solution were 88.8% for Cu, 86% for Zn, and 58% for Fe. However, further increases in temperature did not have a significant effect on the degree of metal transfer into the solution, indicating that a temperature around 90 °C is optimal for the process. The obtained experimental results are presented graphically in Figure 2c.

In the next stage of the process, the effect of the solution stirring speed on the efficiency of selective leaching was evaluated. These experiments were carried out based on the optimal technological conditions determined in the previous stages. According to the research results, when the solution stirring speed was 300 rpm, the degrees of metal transfer into the solution reached the highest values, namely 89.5% for Cu, 88.7% for Zn, and 59.2% for Fe. Based on the results obtained during the experiments, Figure 2d was constructed in graphical form.



(a) sulfiric acid concentration (M) | **(b)** Time (min)

(c) Temperature (°C) | **(d)** Stirring Rate (rpm)

Fig. **2**. Effects of **(a)** sulfiric acid concentration, **(b)** time, **(c)** temperature, **(d)** stirring rate.

№1 №2 №3 №4

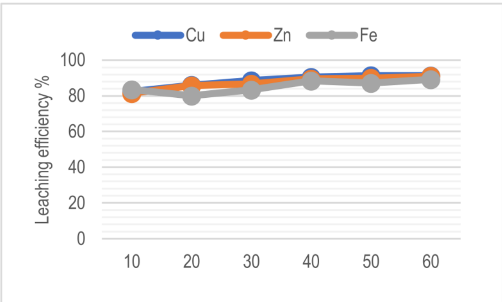
Fig. 3. Samples of solid metal sulfates obtained by drying

After the process was fully completed, the resulting solution was dried under the effect of high temperature in order to obtain solid metal sulfates. In this process, the solution was maintained at a temperature of 150 °C for 90 minutes, ensuring complete evaporation of the water in the liquid phase. As a result, solid metal sulfates suitable for further research in subsequent stages were obtained. The appearance of the metal sulfates obtained under different technological conditions is presented in Figure 3.

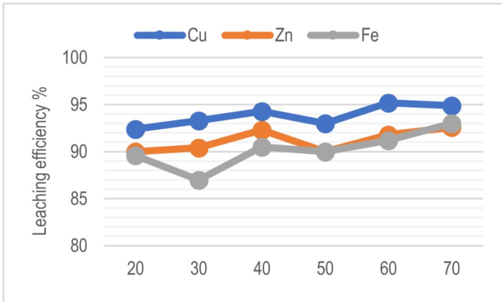
Determining optimal parameters for selective dissolution in water. In order to evaluate the possibilities of efficient separation of solid metal sulfates in subsequent stages, a selective leaching technology using distilled water was applied. For this purpose, several samples weighing 100 g were prepared from solid metal sulfates obtained as a result of selective leaching with sulfuric acid followed by drying. To determine the kinetic characteristics of the selective leaching process, the leaching duration was selected as the main variable parameter and was successively varied in the time range from 10 to 60 minutes. The experiments were carried out at a temperature of 30 °C. The research was conducted consistently under the specified conditions, and the obtained experimental results made it possible to analyze the course of the process. In particular, when the duration of selective leaching in water was 40 minutes, the degrees of metal transfer into the solution were 90.4% for Cu, 89.4% for Zn, and 88.4% for Fe. These results are presented graphically in Figure 4a.

After studies on the duration of the selective leaching process determined that the optimal time was 40 minutes, in the next stage the effect of temperature, which is one of the important factors influencing the process, was investigated. For this purpose, experiments were carried out in the temperature range from 20 to 70 °C. In each experiment, the selective leaching duration was strictly fixed at 40 minutes, and the other technological parameters were kept constant. Based on the results of the conducted experiments, the effect of temperature on the dissolution process of metal sulfates in water was evaluated. It was found that at a temperature of 40 °C, the degrees of metal transfer into the solution for copper, zinc, and iron were 94.3%, 92.3%, and 90.5%, respectively. Increasing the temperature to higher values did not have a nearly significant effect on the degree of metal dissolution. The obtained experimental results are presented graphically in Figure 4b.

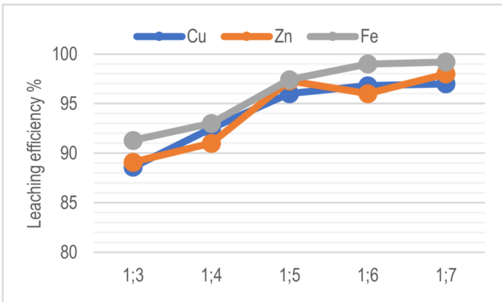
After determining the optimal temperature, the effect of another important technological factor influencing the selective leaching process—the solid-to-liquid phase ratio (S/L)—was investigated. In this stage, the experiments were conducted for a duration of 60 minutes at a temperature of 50 °C. The solid-to-liquid phase ratio was successively varied, and its effect on the degree of dissolution of metal sulfates in water and the overall process efficiency was evaluated. As a result of the experiments, the role of the S/L ratio in the selective leaching process was determined, making it possible to establish the optimal ratio. The obtained experimental results are summarized in Figure 4c and served as a basis for further analysis.



(a) Time (min)



(b) Temperature (°C)



(c) Solid-to-Liquid Ratio

Fig. 4. Effects of **(a)** time, **(b)** temperature and **(c)** solid-to-liquid ratio.

Conclusion. This study investigated the possibilities of extracting metals from copper industry waste, with the aim of eliminating problems that arise during filtration in the sulfuric acid dissolution process. The experiments were conducted in three stages: first, selective leaching in sulfuric acid, then drying the solution, and finally, selective dissolution of the resulting solid metal sulfates in water. Additionally, the main technological parameters influencing the process were systematically evaluated.

The effects of acid concentration, time, temperature, and stirring speed on the degree of metal dissolution during selective leaching with sulfuric acid were determined. Research results established that optimal conditions for the process are a sulfuric acid concentration of 2 M, a duration of 120 minutes, a temperature of 90°C, and a stirring speed of 300 rpm. Under these conditions, the degree of metal dissolution into the solution was 89.5% for Cu, 88.7% for Zn, and 59.2% for Fe.

After selective dissolution, the obta ined suspension was dried at a temperature of 150°C for 90 minutes without filtration, resulting in solid metal sulfates. In the next stage, the selective dissolution properties of this product in water were studied. Experiments in an aqueous medium revealed that a duration of 40 minutes and a temperature of 40°C were the optimal conditions for the transition of metals into solution. At this stage, recovery rates of 96% for Cu, 97.3% for Zn, and 97.4% for Fe were achieved.

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