Journal of Chemical Technology and Metallurgy, 60, 5, 2025, 861-868

STUDY OF THE LEACHING PROCESS FOR COPPER FROM THE PRODUCTION WASTE OF THE BALKHASH COPPER SMELTER WITH THE ADDITION OF AN OXIDIZING AGENT

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Received 28 November 2024
Accepted 10 April 2025
DOI: 10.59957/jctm.v60.i5.2025.17

ABSTRACT

The use of metallurgical slags as secondary raw materials is a pressing issue today. The most rational way to get rid of the negative impact of such objects on the environment is the organisation of heap-leaching sulphuric acid solution. The disadvantages of this process (high temperature, duration of dissolution and concentration of the sulphuric acid) are related to the known high chemical stability of metallic copper and the resulting delayed dissolution.

The study aims to improve the process of slag leaching at the Balkhash Metallurgical Plant. The main idea is that the addition of an oxidizing agent - hydrogen peroxide - to sulphuric acid by analogy with uranium leaching will reduce the duration of dissolution and concentration of sulphuric acid, as well as will not require heating. Modern physical and chemical analytical methods were used to identify the products: the composition of metallurgical slag and post-treated waste was investigated by X-ray spectral analysis on the RLM-21 device, and the phase composition was analysed on the D8 Advance device (Bruker). Processing of the obtained diffractogram data and calculation of interplanar distances were carried out using EVA software. Sample interpretation and phase search were carried out using the Search/Match programme with the PDF-2 Powder Diffractometry Database. This paper presents the first results on the effect of hydrogen peroxide on the leaching of copper. The extraction of copper from the test material is analogous to uranium leaching where the addition of an oxidant in the form of 10 % hydrogen peroxide accelerates the leaching process. We increased the copper recovery to 50 % in 63 days of leaching. Oxygen is known to interact with copper in slag only when heated. In our experiment, the sample was oxidised with 10 % hydrogen peroxide solution and leached with 20 % sulphuric acid solution. In our opinion, oxygen interacts with copper through the formation of monosulphuric and nadsulphuric acids, which are stronger oxidants than hydrogen peroxide. Therefore, the process was carried out without additional heating.

Keywords: metallurgical slag, copper, secondary raw materials, recovery, heap leaching, sulfuric acid, hydrogen peroxide.

INTRODUCTION

As a result of the depletion of reserves, there is an increasing focus on the waste materials produced during the thermal treatment of concentrate in the mining and smelting production industry. The smelting of copper-containing concentrates results in the formation of converter and waste metallurgical slags during the copper manufacturing process at the Jezkazgan and Balkhash Copper Smelters. Converter slags are returned to smelting, and dump slags are stored in a specially designated area - a slag dump according to technology [1]. The enterprises have landfills and areas for the disposal of production waste. Basically, due to the lack of effective approaches for the treatment of such slags in many countries, they are simply stored, which leads to huge environmental risks as well as the waste of many valuable resources [2 - 5, 6]. Therefore, there is an urgent need for the development of new effective methods of copper smelting slag recycling.

Long-term storage of this type of waste harms the environment in the region and is the main source of soil pollution due to the spontaneous leaching of copper, zinc, lead, arsenic and other heavy metals from them. A monitoring study of the soil composition in the Balkhash region shows that the content of SiO_2 and Al_2O_3 in the soil is below the norm [7]. The content of CaO and Fe (in the form of Fe_3O_4) exceeds the norm, S, As, and such heavy metals as Zn, Cu, Cd, Pb, Mo is also present in free form. S, As, Pb, Zn are particularly toxic among them, while the components - As, Pb, Zn are not soil components.

Today, the use of metallurgical slags as secondary raw materials is relevant. The most rational way to get rid of their negative impact on the environment is to organize heap leaching (HL).

A literature review showed that heap leaching is the standard method for processing copper-containing waste from old landfills [8]. Copper leaching technology uses various reagents, including mine water [9].

One of the reagents available in the processing of copper ores and wastes is sulphuric acid. The influence of different concentrations of sulphuric acid on the leaching process has been studied by many scientists, a high concentration of acid leads to the formation of sulphur, which blocks the reactive surface of the material, and a low concentration leads to the formation of soluble sulphates [10 - 14]. A sharp decrease in acidity leads to glandular hydrolysis with the formation of a dense crust on the surface of the material.

One drawback of copper (HL) is its low reactivity compared to other forms of metallic copper. It is a well-established fact that copper is insoluble in non-oxidizing acids. The solubility of copper in sulfuric acid is contingent upon the acid concentration; it remains insoluble in dilute sulfuric acid. Sulfuric acid is a common solvent for heap leaching of copper ores and wastes. Moreover, it is the cheapest of all known solvents. During leaching ores, an increase in acid concentration helps to increase copper extraction and acid consumption depending on the chemical and mineralogical composition of the ores. However, when acidity is increased oxidation of sulphides occurs with the formation of elemental sulphur, which screens the reaction surface of the sulphides. As the acid concentration in the solution decreases, the process proceeds with the formation of soluble sulphates. With a sharp decrease in the acidity of the solution, iron salts are hydrolysed to form a dense crust on the surface of ores and copper minerals.

The high chemical stability of the metallic medium and the resulting slow dissolution, as well as high temperature and concentration of sulfuric acid are a continuation of the process [15, 16]. However, despite this, in the production of copper sulphate, as well as in the processing of secondary raw materials, for example, cement additives, the process of leaching metallic copper in solutions of sulfuric acids is also carried out.

The above-mentioned disadvantages of copper acid treatment with sulfuric acid, namely the concentration of the acid and the duration of leaching, do not allow the use of this technology for a large volume of processing solid waste from the Balkhash copper smelting plant (BMZ).

The above-mentioned disadvantages of copper conversion with sulfuric acid, i.e. temperature, and especially the acid concentration and the duration of leaching, do not allow the use of this technology for a large processing amount of solid waste in the Balkhash Copper Smelter. The same technology is used in the nuclear industry to extract uranium and other valuable components at the uranium mining enterprises of the mining plant Rostom [17]. Much attention has been paid to the improvement of HL processes for the extraction of uranium and other valuable components from various types of uranium ores in the works of such nuclear industry specialists as Shatalov [18], Kuznetsov's work [19] investigated the HL technology for uranium with sulfuric acid from uranium ores using oxidizing agents, including hydrogen peroxide. Good results were obtained in uranium extraction. This study suggested the possibility of using this HL method for copper leaching.

The purpose of this study is to improve the leaching process of metallurgical slag from the Balkhash Metallurgical Plant**.** The main idea is that adding an oxidizing agent, hydrogen peroxide, to sulfuric acid, by analogy with uranium leaching will reduce the temperature, reduce the duration of dissolution and the concentration of sulfuric acid [20]. The following problems were solved for this purpose: the copper content in the slag, the effect of the oxidizing agent and the size of the material on the leaching performance, as well as the modes of sulfuric acid leaching based on the concentration of the sulfuric acid solution were determined. The copper content in the solutions obtained after leaching was determined.

EXPERIMENTAL

Research methods

A range of analytical methods were used in this work. Modern physical and chemical analysis methods were utilized to identify products: The composition of metallurgical slag and after-processed waste was studied by X-ray spectral analysis on an RLM-21 device, and the phase composition was determined on a D8 Advance device (Bruker), α-Cu, tube voltage 40 kV, current 40 mA. The obtained data of diffractograms were obtained, and interplanar distances were calculated with the use of EVA software. Sample interpretation and phase search were performed by the Search/match program with the use of the PDF-2 Powder Diffractometry Database. The surface morphology of the original and processed materials was studied using a MIRA-3 scanning electron microscope (TESCAN). A scanning electron microscope (SEM) is equipped with a system of detectors that record various signals resulting from the interaction of an electron beam with the surface of a sample. The secondary electron detector enables one to take images with topographic contrast. Meanwhile, the X-Act X-ray dispersive microanalysis system (Oxford Instruments) makes it possible to locally determine the elemental composition on the surface of the sample. The copper content in the liquid phase was studied using a Varian AA140 atomic absorption device

The main components of the slag iron 75 % of which passes into the slag in the form of FeO, and 25 % in the form of Fe_3O_4, copper, zinc oxide, calcium oxide and silicon oxide [21]. This metallurgical slag is obtained by processing copper concentrate in a furnace. Pure copper melts at 1084^{o}C, and in the presence of impurities, at a lower temperature. Impurities of volatile metals and oxides - metallic zinc, arsenic and antimony trioxide - are removed by heating copper until it melts. Copper oxidizes to cuprous oxide when it is melted. This oxide is stable above 1100^{o}C. Copper (I) oxide accumulates on the surface of molten copper in solid (up to 1200^{o}C) and liquid (above 1235^{o}C from and partially dissolves in copper, and then it interacts with impurities, for example: $Cu_2O + Fe = FeO + 2Cu$.

As the dissolved copper oxide is consumed, new quantities move from the surface into the solution, and the copper undergoes further oxidation.

The resulting oxides of iron, calcium and other metals are insoluble in copper and turn into slag, which floats to the surface of the metal. Due to the interaction of copper (I) oxide with some oxides (for example, with iron oxide with the formation of copper ferrite), part of it also passes into slag, and the Cu_2O content in it reaches 30 %. In this case, the resulting starting material has the following chemical composition: Cu - 0.72 %, Fe - 38.6 %, Fe_3O_4 - 14.7 %, SiO_2 - 28.7 %, CaO - 2.4 %, Pb - 0.3 %, Zn- 3.6 % and others 10.98 %.

Depending on the physical and mechanical properties of the material, two modes of heap leaching are possible: infiltration and filtration [22]. Porous and fractured materials are most favourable for filtration leaching since they have good filtration properties and sometimes. They are leached without crushing, since filtration properties are characterized by porosity, permeability and the rate of penetration of the solution into pieces of material, therefore, the surface of pieces of slag material was studied using a Tescan Mira-3 scanning electron microscope to determine pores and cracks. The study shows that the surface of the original component is uneven, there are numerous cracks and pores (Fig. 1). The filtration mode was used in this case. It ensures the complete filling of the void of the material under study.

The elemental composition of the sample surface was analysed by electron microscope (Fig. 2).

The obtained results of analysis on the Tescan Mira 3 scanning electron microscope show that the copper content is very low, and large quantities of oxygen, silicon and iron are present. The D8 Advance apparatus (Bruker) was used to analyse the phase analysis of the feed material to determine the state of copper and other associated elements bound in the slag. It was found

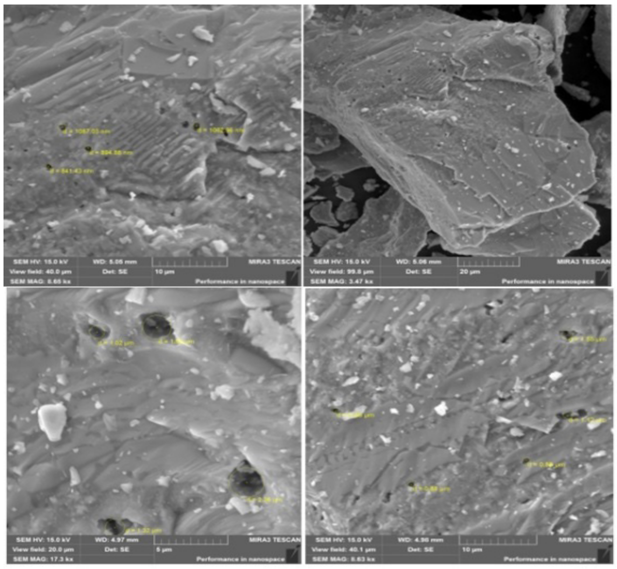

Fig. 1. Electron micrographs of slag sample.

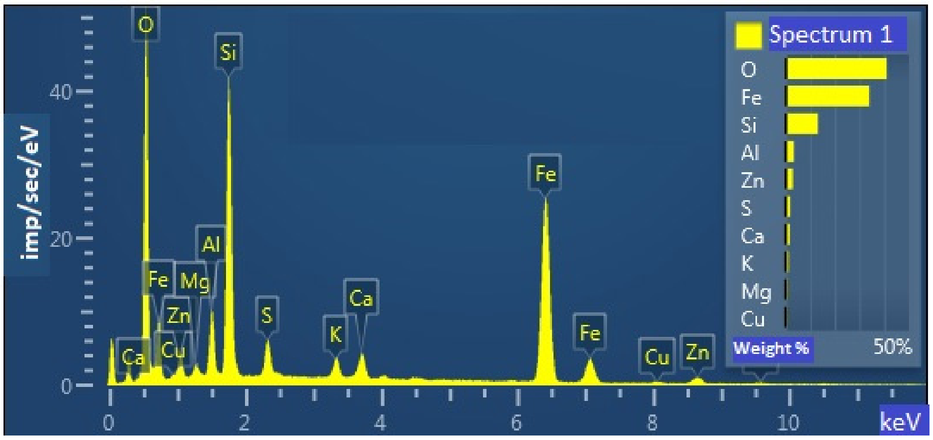

Fig. 2. Elemental composition of the sample surface.

that the initial sample contained calcium, magnesium and iron silicates $Mg_{0.26}Fe_{1.74}(SiO_4)$, as well as small amounts of calcium, magnesium ferrites $(Mg_{0.22}Fe_{0.78})(Al_{0.4}Mg_{0.78}Fe_{0.82}Cu_{0.65})O_4$ (Tab.1). Iron is also present in the form of magnesite. Previously, the characterisation of slag and the distribution of elements in it has been studied by researchers using semi-quantitative X-ray phase analysis [6, 23, 24].

The phases of calcium, magnesium and iron silicates, calcium, magnesium and copper ferrites present are almost insoluble in hydrochloric acid, but soluble in concentrated sulfuric acid. Two experiments were carried out to leach copper from industrial waste.

In experiment 1, the material was subjected to HL using the traditional method, where the test material was leached with a 20 % sulfuric acid solution

In experiment 2, the material was leached with an oxidizer in analogy to uranium leaching. The studied material was oxidized with 10 % hydrogen peroxide solution and leached with 20 % sulfuric acid solution. Experiments 1 and 2 were conducted in parallel.

A solid and liquid sample of the material was analysed during the experiment. The phase composition of the solid sample was studied. The results of the analysis showed that the initial phases decreased after treatment of the material under study with hydrogen peroxide and sulfuric acid (Fig. 4), and were divided into silicate, ferrite phases, and sulphate phases

Table 1. Phase analysis of the initial sample.

Compound Name	Formula	S-Q
Fayalite	$Mg_{0.26}Fe_{1.74}(SiO_4)$	60.4 %
Magnetite, magnesian, syn	$(Mg_{0.22}Fe_{0.78})\ (Al_{0.4}Mg_{0.78}Fe_{0.82}Cu_{0.65})O_4$	39.6 %

Table 2. Phase analysis of depleted sample.

Compound Name	Formula	S-Q
Fayalite	$Mg_{0.26}Fe_{1.74}(SiO_4)$	31.7 %
Magnetite, magnesian, syn	$(Mg_{0.22}Fe_{0.78})(Al_{0.4}Mg_{0.78}Fe_{0.82}Cu_{0.65})O_4$	27.7 %
Gypsum, syn	$CaSO_4{\cdot}2H_2O$	17.4 %
Jarosite, hydronian, syn	$(K,H_3O)Fe_3(SO_4)_2(OH)_6$	12.5 %
Rozenite	$FeSO_4{\cdot}4H_2O$	10.7 %

were also formed with the following composition: $Mg_{0.26}Fe_{1.74}(SiO_4)$, $(Mg_{0.22}Fe_{0.78})(Al_{0.4}Mg_{0.78}Fe_{0.82}Cu_{0.65})O_4$, $CaSO_4{\cdot}2H_2O$, $(K,H_3O)Fe_3(SO_4)_2(OH)_6$, $FeSO_4{\cdot}4H_2O$ (Table 2).

RESULTS AND DISCUSSION

The copper content in the solution was determined 4 weeks after loading. The analysis results show that the copper content in the solution increases. A comparison of the results of experiments 1 and 2 showed that the solubility of copper in experiment 1 increased moderately. The dissolution of the copper oxidized solution increases not uniformly in experiment 2. The copper content did not increase noticeably in the beginning, later the content began to increase intensively.

Based on the diagram, there was a significant surge in the rise of copper content in the solution starting from the seventh week of leaching, as it is shown in Table 2. Approximately 30 % of the copper underwent dissolution in Experiment 2. As a result, hydrogen peroxide has a beneficial impact on the leaching process, enabling to accelerate it.

Copper is known to be insoluble in non-oxidising acids. The solubility of copper in sulphuric acid depends on the concentration of the acid, it is not soluble in dilute sulphuric acid.

The oxidising power of the acid can therefore be increased by hydrogen peroxide, which will increase the yield of copper in the solution. It is necessary to consider the order of mixing of sulphuric acid and hydrogen peroxide. It was found that the introduction of hydrogen peroxide into sulphuric acid is effective, and then the conversion of hydrogen peroxide into monosulphuric and persulphuric acids will be complete [25, 26].

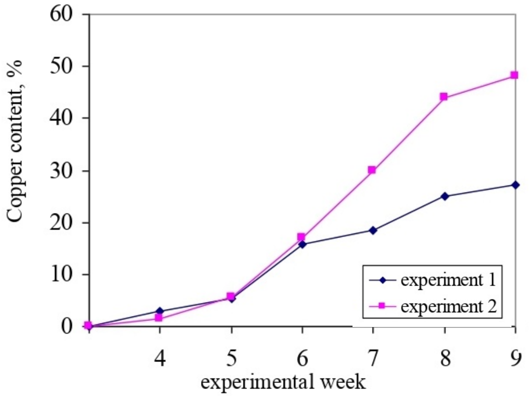

Fig. 3. Effect of oxidizing agent on the leaching process of copper into solution.

In experiment 2, we add sulphuric acid to a solution of hydrogen peroxide and then some of the hydrogen peroxide decomposes to form oxygen. Oxygen is known to interact with copper in slag only when heated. In this work, the leaching process is carried out at room temperature. No heat release was observed during the experiment, so there is no need for high temperatures during the leaching process. In this system, we believe that oxygen is necessary to enhance the oxidising property of the acid. Therefore, we added 10 % hydrogen peroxide solution to the aqueous solution containing the material.

Before testing, the starting material was saturated with water for 10 days. According to technological data, it is known that 30 % of copper is in the form of copper (I) oxide, while the balance is in the metallic condition.

Copper (I) oxide does not react with water. It dissociates to a very small extent:

$$Cu_2O + H_2O \leftrightarrow 2Cu^+ + 2OH^- \quad (1)$$

$$Cu_2O + 3H_2SO_4 = 2CuSO_4 + SO_2\uparrow + 3H_2O \quad (2)$$

Besides, copper (I) oxide in aqueous solutions is slowly oxidized by oxygen to copper (II) hydroxide.

$$2Cu_2O + 4H_2O + O_2 = 4Cu(OH)_2\downarrow \quad (3)$$

$$Cu(OH)_2 + 2H_2SO_4 = CuSO_4 + 2H_2O \quad (4)$$

Hydrogen gas formation is possible in the system because the slag composition contains 38.7 % iron in the metallic state, which forms hydrogen gas when interacting with sulphuric acid. Hydrogen hurts the properties of copper [27]. In the leach solution, hydrogen interacts with cuprous oxide to form water vapour:

$$Cu_2O + H_2 = 2Cu + H_2O \quad (5)$$

Large water molecules remain in the volume of the copper and accumulate in the smallest pores, and as the temperature rises, the water pressure increases and bubbles are formed. As the vapour cools, the water molecules cause deformation of the sample at the boundaries, and the copper becomes brittle.

In Experiment 2, the size of the leached sample was much reduced, when comparing the transformation of the material samples before and after the leaching experience, there were some changes (Fig. 4), which favoured the rapid process flow.

The changes in the samples may be due to the occurrence of hydrogen sickness of copper, which leads to embrittlement of the metal [28].

In the samples studied, the metals are present in both oxide and metallic forms. Therefore, the formation of hydrogen and oxygen in the system is more likely when leached by sulphuric acid. Then, in experiment 2, it is possible to simultaneously proceed with the decomposition of hydrogen peroxide with the formation of oxygen, as well as the release of hydrogen gas in the interaction of Zn, Fe, and Pb with acid. This leads to the reduction of the sample size and proves the efficiency of the use of hydrogen peroxide.

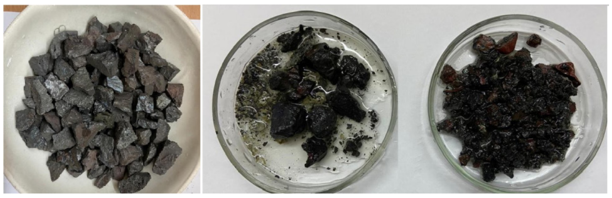

Fig. 4. Study samples before (a) and after (b) leaching.

CONCLUSIONS

Thus, these studies demonstrate the possibility of improving the process of leaching copper from the waste slag of the Balkhash metallurgical plant. The results of the experiments show that the extraction of copper from the test material by analogy with uranium leaching with the addition of an oxidant in the form of 10 % hydrogen peroxide accelerates the leaching process. We were able to increase the degree of copper extraction up to 50 % for 63 days of leaching, whereas, under production conditions, it takes much longer.

The scanning electron microscope examination of the surface of the slag pieces showed that the number of pores in the oxidised sample decreased and the size of the sample pieces became much smaller. Therefore, the level of copper ions in the solution became higher in Experiment 2.

According to the results of the experiment, it can be concluded that the addition of hydrogen peroxide to the solution in sulphuric acid increased the transition of copper into the solution due to the interaction of copper with monosulphuric and supersulphuric acids, which have a high oxidising power at room temperature.

Acknowledgements

This research has been/was/is funded by the Science Committee of the Ministry of Science and Higher Education of the Republic of Kazakhstan (Grant No. AP22683272).

Authors' contributions: *A.O.: Conceptualization, Experiments, Methodology; G.K.: Conceptualization, Methodology; A.K.: X-ray spectral analysis; T.S.: Conceptualization, Writing - original draft; M.S.: Methodology; G.O.: Investigation, Validation.*

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