

Selective separation and recovery of copper from industrial waste by leaching

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Abstract

Efficient management of limited natural metal resources and minimizing environmental impact is crucial in modern industrial production. This study focuses on the selective separation and recovery of copper from industrial waste through hydrometallurgical and electrolytic methods. Industrial metal waste, particularly from electronics and manufacturing, contains valuable materials that, if properly processed, can serve as secondary raw materials. The research investigates various technological approaches for metal recycling, emphasizing hydrometallurgical leaching and electrolysis as effective recovery methods.

The experimental part explores the processing of copper, brass, and bronze waste through sulfuric acid leaching, enhanced with hydrogen peroxide as an oxidizing agent, followed by electrolysis. The efficiency of copper recovery was assessed by atomic absorption spectrometry, scanning electron microscopy, and X-ray fluorescence spectrometry. Results demonstrate that leaching at elevated temperatures (60°C) significantly improves metal dissolution rates, leading to higher recovery yields.

Electrolytic processing of the leachate successfully produced high-purity copper, confirming the viability of this method for sustainable metal recycling. The findings highlight the environmental and economic benefits of recovering metals from industrial waste, reducing the reliance on primary raw materials, and minimizing waste generation. Further optimization of process parameters, including temperature, pH, and electrolyte composition, could enhance recovery efficiency and metal purity. This study contributes to the development of sustainable recycling strategies, promoting circular economy principles and reducing the environmental footprint of metal production.

Keywords

Copper recovery, hydrometallurgy, electrolysis, industrial waste, metal recycling, environmental sustainability.



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Introduction

Today, it is increasingly important to manage limited natural metal resources efficiently and to minimise the environmental impact of mining. This issue is particularly discussed in the context of industrial production, where large amounts of waste containing valuable materials remain unused. Industrial waste containing metals is often perceived as a burden on the environment; however, if properly treated, it can serve as a valuable basis for secondary raw materials. In the current climate of high demands for sustainable development and waste reduction, the recycling of non-ferrous metals is not only economically viable but also crucial for environmental protection.

As the production and increased reliance on non-ferrous metals, such as copper, aluminium, and zinc, increase, so does the amount of waste containing these elements. The use of metals is central to many industrial applications, including electronics, construction, and transportation (Sliva et al., 2019). This paper gives particular attention to recycling options for non-ferrous metals, which encompass a range of technological approaches, from mechanical to chemical and biological methods.

Finally, the work focuses on methods for analyzing industrial copper (and metal) wastes in general, utilizing techniques such as atomic absorption spectrometry, X-ray fluorescence spectrometry, scanning electron microscopy, and other relevant methods. Some of these methods were subsequently applied to the characterization of copper matrices in the practical part of the paper (Khan et al., 2022).

The experimental part of the work focuses on hydrometallurgical and electrolytic methods of copper waste processing. Hydrometallurgy was chosen because of its capability to extract metals efficiently, even from low-grade ores and wastes, which makes it ideal for treating industrial wastes. Electrolysis subsequently enables the production of metal with high purity, which is essential if the material is to be further utilized in industry (Sliva et al., 2019).

Metal is one of the best recyclable materials as it can be recycled repeatedly without losing its quality. Recycling metals and their alloys is an effective way to reduce the ecological consequences of mining and conserve the natural resources of primary raw materials. With increasing technological innovations, processing methods are constantly improving, increasing the efficiency of recycling and expanding the possibilities for further use of recycled materials. Metal recycling helps to reduce waste and promote the production of new metal products in a more sustainable and environmentally responsible way.

Recycling of metal waste is an essential strategy to address issues related to material availability, material efficiency, reduction of primary raw material consumption, energy efficiency, and environmental protection. Metal recycling helps to conserve natural resources, reduce waste, and promote the production of new metal products in a more sustainable and environmentally friendly way (Iannicelli-Zubiani et al., 2017; Samuelsson et al., 2014).

Treatment and recovery of industrial waste containing copper and copper alloys

In the context of industrial applications and research, it is important to promote innovation and the advancement of technologies that enable the efficient treatment and recycling of waste comprising copper and its alloys. Due to its high thermal and electrical conductivity, as well as its high corrosion resistance makes copper suitable for numerous uses, such as electrical and electronic equipment (for instance, tubes utilized in televisions, computer monitors, and audio/video amplification systems), automobiles (radiators, brakes and wiring), construction, telecommunications, energy, machinery and equipment manufacturing (Khan et al., 2022; Samuelsson et al., 2014).

Significant progress in electrical and electronic technologies leads to the frequent replacement of existing products with upgraded models that offer enhanced capabilities and broader functionalities. As a consequence, discarded equipment has become one of the waste categories with the highest growth rate worldwide (Khan et al., 2022). Electronic waste (e-waste) consists of a diverse range of discarded electrical and electronic equipment, irrespective of whether it is actively disposed of or kept in storage within households. This category is commonly divided by size and function, with large equipment including items such as televisions, desktop computers, and washing machines, while small equipment comprises devices like mobile phones, laptops, and tablets. The classification facilitates systematic assessment of e-waste production and management (Brožová et al., 2021). Irrespective of their dimensions, the majority of a printed circuit board (PCB) is an integral component of electronic devices, which serves as a key component due to its concentration of high-value metals such as palladium, silver, gold, and copper. The recovery of these materials not only presents considerable economic benefits but also reduces the environmental consequences of primary metal extraction (Yust et al., 2022; Sarfo et al., 2017). Furthermore, printed circuit boards also contain hazardous metals, such as lead and mercury, which pose significant environmental and health risks if not properly managed during disposal and recycling processes (Gunaratne et al., 2022). Other studies (Quijada-Maldonado et al., 2020; Iannicelli-Zubiani et al., 2017) have also reported the presence of additional hazardous elements in PCBs, including cadmium, arsenic, chromium, and antimony. The total composition of printed circuit boards has been analyzed by many researchers (Rai et al., 2021; Gómez et al., 2023) and is presented in Table 1. PCBs usually contain 40% metals, 30% plastics, and 30% other materials, such as ceramics (Xiao et al., 2013; Yu et al., 2020). Proper management and recycling of e-waste

contribute to environmental protection, while also helping to protect natural resources (Goldstein et al., 2017; Fomchenko et al., 2020; Martynková et al., 2022).

The primary techniques employed for recycling electronic waste include pyrometallurgical processes (Samuelsson et al., 2014; Kaksonen et al., 2020), hydrometallurgical (Cocchiara et al., 2019; Drápala et al., 2022), as well as biohydrometallurgical (Schueler et al., 2021; Ye et al., 2017) in combination with mechanical pre-treatment (Quoc et al., 2020). In contrast, hydrometallurgical techniques provide higher operational precision and process predictability, coupled with straightforward parameter control. They typically demand lower capital expenditure and energy consumption, while exhibiting a reduced environmental footprint relative to thermal metallurgy processes (Kaksonen et al., 2020; Martynková et al., 2022). In recent developments, advanced methodologies for recovering metals from e-waste have been implemented, encompassing chelation-based separation processes, the use of ionic liquids (ILs), hybrid extraction systems, green adsorption techniques, and other innovative recovery strategies aimed at enhancing efficiency and sustainability (Rai et al., 2021). Common agents employed for the purpose include H_2SO_4 , HNO_3 , HCl , and aqua regia, often in combination with oxidizing agents such as O_2 , Cl_2 , and H_2O_2 (Adetunji et al., 2023; Bendikiene et al., 2019). In addition to these oxidants, ozone has emerged as a widely used leaching agent due to its strong reactivity with metals, and it has been applied not only in e-waste processing but also in the recovery of metals extracted from primary ores.

Tab. 1 The composition of the printed circuit board (Adetunji et al., 2023; Bendikiene et al., 2019).

Materials and their contents	Metals (to 40%)	Cu (6.9-22%), Al (2-14.2%), Pb (1.35-6.3%), Zn (1.4-20.5%), Ni (0.28-2%), Fe (1.4-20.5%), Sn (1-4%), Sb (0.4-20ppm), Au (20-1000ppm), Ag (20-2000ppm), Pd (10-50ppm), Ge (20ppm), As (10ppm), Ti (200ppm), In (20ppm), Ta (200ppm), Co (200ppm), Se (20ppm), Ga (10ppm)
	Ceramics (to 30%)	SiO_2 (15-41.86%), Al_2O_3 (6-30%), CaO (6-30%), titans and mica (3-30%)
	Plastics (to 30%)	Polyethylene (9.9%), polypropylene (4.8%), polyesters (4.8%), epoxies (4.8%), polyvinyl chloride (2.4%), polytetrafluoroethane (2.4%), nylon (0.9%)

Ozone is a colorless gas with an intense odor noticeable at minimal concentrations of 0.02 ppm to 0.05 ppm. Ozone has a density of 2.144 g/L, a boiling point of -111.9°C , and a melting point of -193.0°C (Kaksonen et al., 2020). Literature sources indicate that ozone exhibits a higher oxidizing strength than molecular oxygen (Turan et al., 2024). A comparative overview is presented in Table 2 (Kaksonen et al., 2020).

Tab.2 Comparative analysis in relation to the oxidation potential of various chosen reagents (Kaksonen et al., 2020).

Reagents	Fluorine (F_2)	Ozone (O_3)	Hydrogen Peroxide (H_2O_2)	Chlorine (Cl_2)	Oxygen (O_2)	Bromine (Br_2)	Iodine (I_2)
Redox potential	2.87 V	2.07 V	1.78 V	1.36 V	1.23 V	1.09 V	0.54 V

Ozone is formed naturally by the ultraviolet rays of the sun and lightning strikes. Due to the fact that this gas turns back into oxygen within a relatively short period, it cannot be stored and delivered in a cylinder. Ozone can therefore be generated in situ by an ozone generator. Artificial ozone production is carried out using ultraviolet rays (UV light) or corona discharge (Brožová et al., 2021). Both methods use the principle on which ozone is formed in nature. In practice, corona discharge is more commonly used to produce ozone due to its numerous advantages. This method is more economically efficient, and ozone-generating elements are also more durable. Atmospheric air or pure oxygen can be a feed gas. Higher ozone concentrations can be achieved when pure oxygen is used as the source gas, for example, from oxygen cylinders (Habibi et al., 2020).

The application of ozone functioning as an oxidizing agent represents a viable alternative in the process of leaching metal-bearing concentrates and waste streams, including those containing precious metals. Its primary environmental advantage lies in the fact that the sole by-product of the reaction is molecular oxygen, thereby minimizing secondary pollution (Habibi et al., 2020):



Ozone has been employed as a high-potential oxidizing agent in the hydrometallurgical processing of a wide spectrum of sulfide ores and secondary raw materials (Rosado et al., 2021; Bendikiene et al., 2019). In the oxidative leaching of stibnite concentrate in hydrochloric acid media, an antimony recovery efficiency of 99% was achieved (Adetunji et al., 2023). Zhao et al. (2019) demonstrated its application in the leaching of tetrahedrite in HCl

solutions, attaining a maximum copper dissolution of 25% in 0.5 M HCl and approximately 18% antimony dissolution in 0.75 M HCl. For pyrrargyrite, Haque et al. (2024) utilized H₂SO₄ with ozone addition, resulting in an 80% silver dissolution rate, with process efficiency strongly dependent on ozone concentration.

In precious metal recovery, ozone has been integrated as a pre-oxidation step prior to chlorate(I) leaching (Samuelsson et al., 2014) and before conventional cyanidation (Zhao et al., 2019), enhancing gold liberation. Additionally, its efficacy has been confirmed in the oxidative treatment of chalcopyrite (Samuelsson et al., 2014) and in H₂SO₄ leaching systems (Brožová et al., 2021). Keber et al. (2020) reported 94.3% Sb extraction with minimal Fe dissolution (2.3%) from antimony sulfide ore containing pyrite using 4.5 M HCl with ozone.

Beyond primary ore processing, ozone has been applied to secondary resources, including silver-bearing waste, where the addition of 0.1 M H₂SO₄ with ozone facilitated efficient silver recovery (Zhao et al., 2019).

Materials and methods

Within the practical part, hydrometallurgical and subsequent electrometallurgical processing (electrolysis) of copper, brass, bronze, and copper-copper metal waste, namely sheet metal scraps, wire residues, transformer coils, ropes, and other materials from the manufacturing industry, was carried out. The aim was to obtain copper metal with a certain level of purity that can serve as a secondary source of raw materials in the foundry industry, with the possibility of its further processing and production of a specific product (Fig. 1).

Refined Hydrometallurgical Processing Flowchart

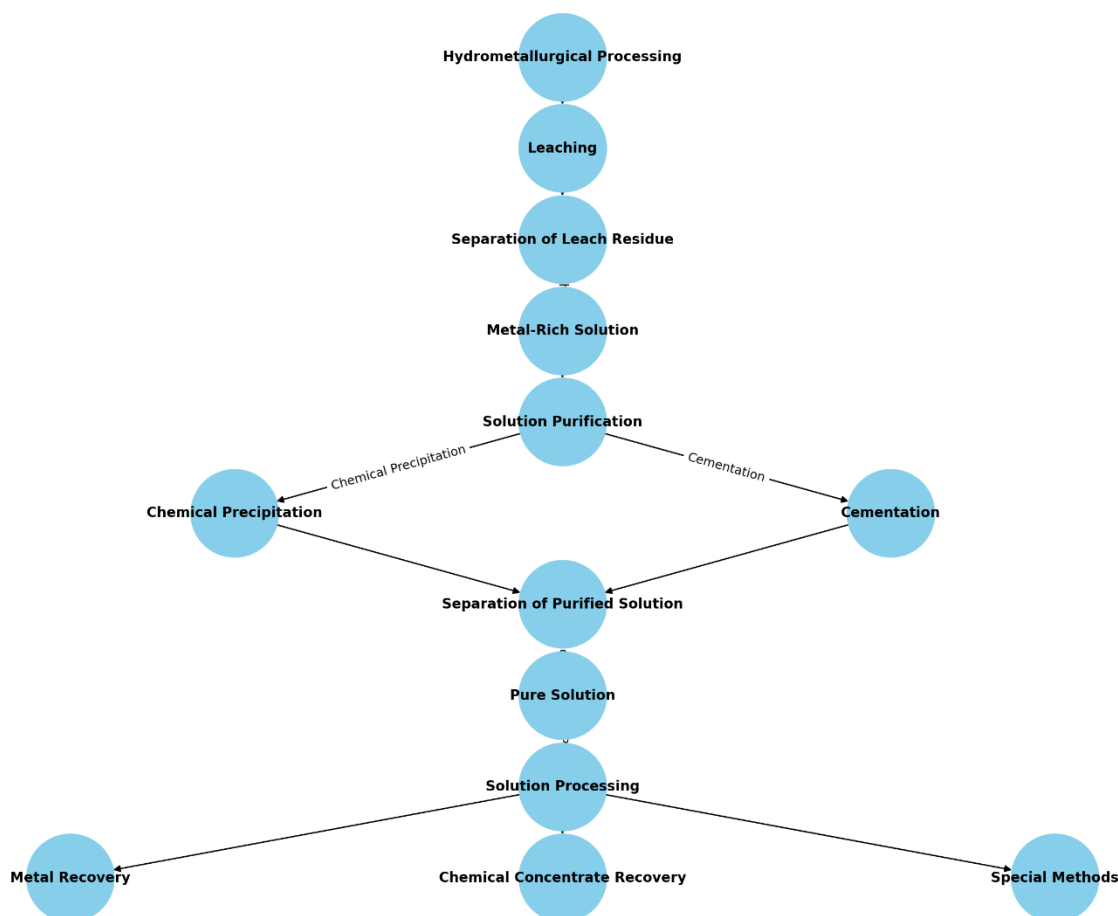


Fig. 1 Flowchart of Hydrometallurgical Processing of Ores

In Fig. 2, two samples of copper waste are photographed, which were used under different temperature conditions. One of them was further used for leaching at 20 °C (sample 1), and the other at 60 °C (sample 2).



Fig. 2 Copper waste: (1) Sample 1 (leaching temperature 20 °C) and (2) Sample 2 (leaching temperature 60 °C).

In terms of material characterization, scanning electron microscopy (SEM) images were taken of the copper waste and both copper samples after the electrolysis process. Electron images were obtained using a STEM microscope JEOL JSM-7610F Plus (JEOL Ltd., Tokyo, Japan) at the Department of Nanotechnology Centre, Centre of Energy and Environmental Technologies, VŠB-TU Ostrava. Two detection modes, namely SEI and COMPO, with an accelerating voltage of 15 kV, were used to acquire the images.

Elemental analysis was performed on both the input copper waste and copper samples obtained by electrolytic processing. The elemental composition was measured using a separate XRF spectrometer and also using an EDS microanalyzer within the SEM/EDS. Atomic absorption spectrometry (AAS) was performed on the leachate obtained after copper leaching before and after electrolysis.

The SEM image in Fig. 3, which was taken in the dark field, shows the surface of a copper sheet cut off. The surface of the copper sheet exhibits a distinct contrast between smoother areas, which may represent regions where the sheet has been treated with a protective coating, and other areas with a rougher texture, possibly caused by oxidation processes, corrosion, or mechanical wear.

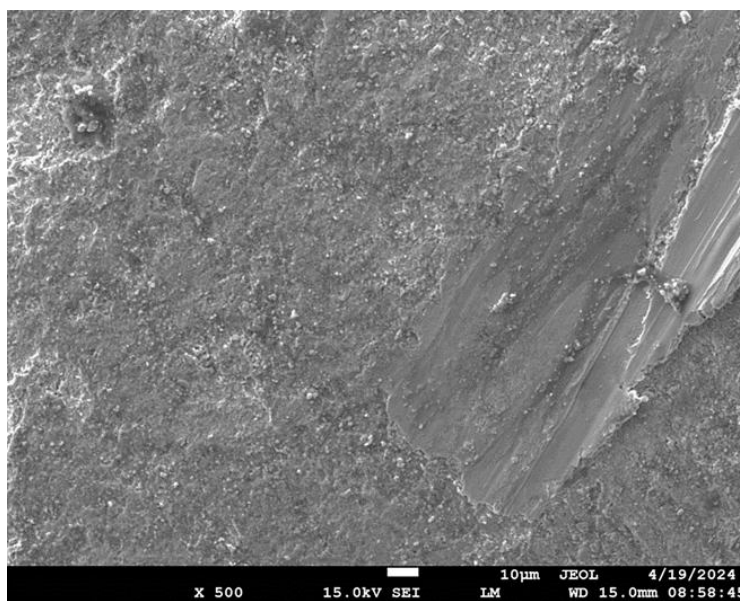


Fig. 3 SEM image of the surface of the copper sheet used in the hydrometallurgical processing, shown in secondary electron mode at 500x magnification (scale bar 10 μm).

As mentioned above, the copper waste was of a heterogeneous nature; however, SEM/EDS microanalysis was performed prior to leaching, specifically on the copper sheet whose surface is visible in the electron image (Fig. 3) and also on the wire. The elemental composition, as shown in the EDS spectra, indicates that both the sheet (Fig. 4) and the wire (Fig. 5) contain the highest proportion of copper, specifically 53.5 wt.% and 67.8 wt.%, respectively. Furthermore, carbon (35.1 wt. % and 28.4 wt. %) and oxygen (10. 6 wt. % and 3.7 wt. %) are present in high levels. The remaining elements (iron, titanium, aluminium, and silicon) are only present in trace amounts ranging from 0.1 to 0.2 wt. %.

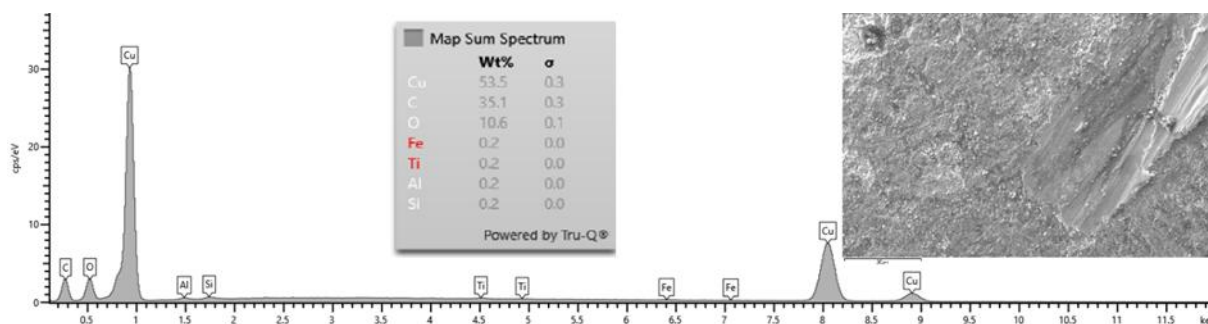


Fig. 4 Elemental composition of the copper sheet, which showed a content of 53.5 wt.% Cu, 35.1 wt.% C, 10.6 wt.% O and 0.2 wt.% Fe, Ti, Al and Si.

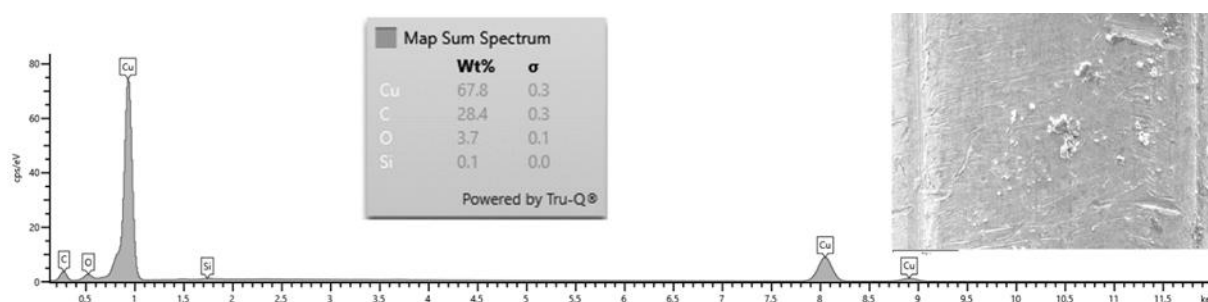


Fig. 5 Elemental composition of the copper wire, which showed a content of 67.8 wt.% Cu, 28.4 wt.% C, 3.7 wt.% O and 0.1 wt.% Si.

Chemicals used

Sulphuric acid (96% H_2SO_4 ; Ing. Petr Švec - PENTA s.r.o., Prague, Czech Republic, CAS: 7664-93-9) was used in a dilute state (20% solution) as a leaching agent (solution) to extract copper ions. Hydrogen peroxide in its original state (30% H_2O_2 ; VWR International S.A.S., Briare, France, CAS: 7722-84-1) was used as the oxidizing agent, where the oxidizing agent helps to oxidize copper from its solid state to a form that is soluble in the leaching solution used. Nitric acid (65% HNO_3 ; Ing. Petr Švec - PENTA s.r.o., Prague, Czech Republic, CAS: 7697-37-2) was used in its original undiluted state at the end of the leaching process, when a few drops were added to each sample for stabilization. In addition, a binder ($\text{C}_{38}\text{H}_{76}\text{N}_{2}\text{O}_4$; Hoechst wax C micropowder tabletting agent for XRF analysis, Darmstadt, Germany, CAS: 110-30-5) was added to the copper powder in the preparation of the tablet for XRF analysis.

Hydrometallurgical processing of copper waste

Hydrometallurgical treatment of copper waste was carried out as follows. Copper waste weighing 250 g (for each sample) was first weighed using an OHAUS Navigator XT NVT641/2 portable weigher (OHAUS Europe GmbH, Nänikon, Switzerland) and then transferred to a beaker. The beaker with copper waste was added with 1,000 mL of a 20% H_2SO_4 leaching solution, as shown in Fig. 6.



Fig. 6 Copper waste (samples 1 and 2) mixed with a leached 20% H_2SO_4 solution at the input.

After 2 hours of leaching, 50 mL of a 30% H_2O_2 was added to both samples as an oxidizing agent to accelerate the reaction, and the leaching solution turned blue. The colouration of both samples is visible in Fig. 7, where sample 1 appears significantly darker than sample 2 in the photograph. However, this difference is due to the different structure of the copper waste.



Fig. 7 Samples 1 and 2 after addition of the oxidizing agent (H_2O_2).

Table 3 shows the sample designations and summarises information on the conditions under which the copper waste was leached using leaching and oxidising agents. The hydrometallurgical treatment of each sample was carried out under different temperature conditions, with sample 1 leached at 20 °C and sample 2 heated to 60 °C. The leaching time in both cases was 8 hours.

After 8 hours, the leaching was stopped, and a few drops of a 65% HNO_3 were added to stabilize the samples. Subsequently, filtration was performed to separate the leachate from the leachate.

Tab. 3 Identification of the samples and the basic conditions under which the leaching was carried out.

	Sample 1	Sample 2
Amount of copper waste [g]	250	250
Leaching solution (20% H_2SO_4) [ml]	1 000	1 000
Leaching temperature [°C]	20	60
Leaching time [hours]	8	8
Oxidizing agent (30% H_2O_2) [ml]	50	50

Filtration of samples 1 and 2 produced a clear leachate from which samples were collected for AAS analysis (Table 4).

Tab. 4 Analysis of leachates of samples 1 and 2 from copper waste by the AAS method before electrolysis.

Chemical element	Fe [$\text{mg}\cdot\text{l}^{-1}$]	Cu [$\text{mg}\cdot\text{l}^{-1}$]
Sample 1	211.0	22 870.0
Sample 2	229.9	27 260.0

Electrolysis of leachates

Fig. 8 shows the instrumentation arrangement for electrolysis. The leachate (electrolyte) was transferred to the electrolyzer, and two stainless steel, well-cleaned electrodes were immersed in the electrolyzer and connected to the power supply and multimeters using clamps and jumper cables. One electrode is positive (the anode) and the other is negative (the cathode). During electrolysis, chemical reactions occurred at both electrodes, with oxidation at the anode and reduction at the cathode.

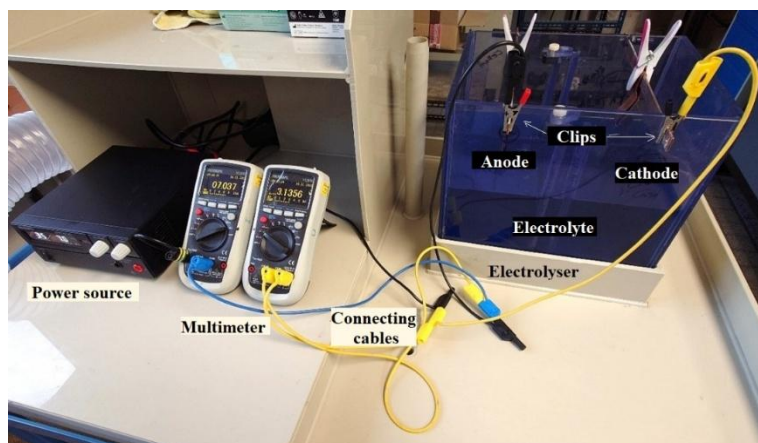


Fig. 8 Experimental setup for electrolysis.

After the electrolysis was completed, electrolyte samples (samples 1 and 2) were taken for AAS analysis to either confirm or refute that there was a loss of copper in the leachate, resulting in copper leaching at the cathode. Table 5 shows the amount of copper remaining in the electrolyte after 2 hours of electrolysis. Comparing the resulting AAS values measured in the samples before and after electrolysis (see Table 4), it was shown that there was a significant reduction in the copper content of the leachate of both samples (before electrolysis: sample 1 - 22 870.0 mg·l⁻¹; sample 2 - 27 260.0 mg·l⁻¹; after electrolysis: sample 1 - 6 640.0 mg·l⁻¹; sample 2 - 7 790.0 mg·l⁻¹). In addition to the AAS analysis, the fact that copper precipitation occurred on the cathode surface is confirmed by Fig. 8, which shows a cathode covered with powdered copper.

Tab. 5 Electrolyte analysis (samples 1 and 2) by the AAS method after electrolysis.

Chemical element	Cu [mg·l ⁻¹]
Sample 1	6 640.0
Sample 2	7 790.0

The cathode surface covered with copper powder is shown in Fig. 9.



Fig. 9 Cathode surface covered with powdered copper that was expelled during electrolysis (sample 1).



Fig. 10 Copper after electrolysis (sample 1) analysed by SEM and SEM/EDS.

In Fig. 10, the copper after electrolysis for sample 1 is photographed and subjected to SEM/EDS elemental analysis, and electron images were also taken using SEM.

The EDS spectrum (Fig. 11) shows the elemental composition of copper from sample 1. As in the case of copper from sample 2, the microanalysis revealed the highest concentration of copper at 64.3 wt.%, along with trace amounts of sulfur (1.2 wt.%). Compared to copper from sample 2, oxygen (20.1 wt.%) and carbon (14.4 wt.%) were also present, whereas chlorine and iron were absent (see information in Fig. 11).

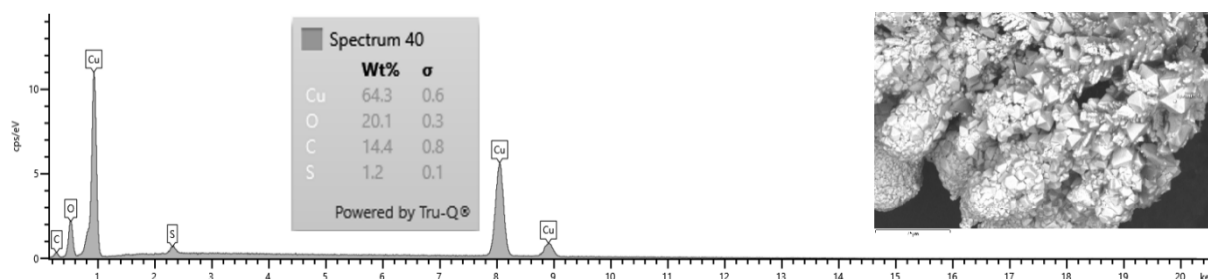


Fig. 11 Elemental composition of powdered copper after electrolysis from sample 1, which showed a content of 64.3 wt.%, Cu, 20.1 wt.% O, 14.4 wt.% C and 1.2 wt.% S.

Copper after electrolysis (samples 1 and 2) was further analysed by SEM. The microstructures of the powdered copper (sample 2) can be seen in the images in Fig. 12, which were taken in secondary electron mode. The electron image in Fig. 12(a) shows clusters of copper particles that appear as compact aggregates with a fine porous structure. The particles are of different sizes and shapes. The image in Fig. 12(b) was taken at 2,000x magnification, and a more detailed structure of the particles can be observed. We can see that some particles are of well-defined geometric shapes with flat surfaces and sharp edges, indicating crystalline growth. These characteristics may be the result of the electrolysis process and the conditions under which the copper was electrolytically prepared.

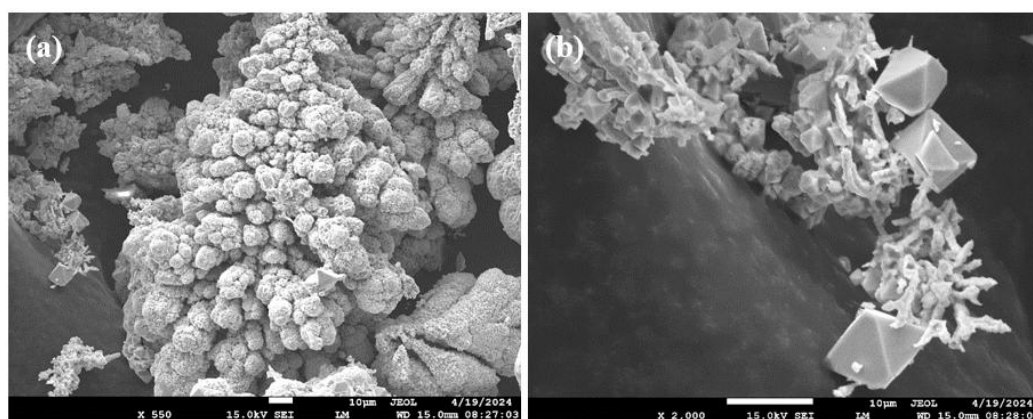


Fig. 12 SEM images of powdered copper after electrolysis (sample 2) in secondary electron mode at magnifications of (a) 550x and (b) 2000x (scale bar on images 10 µm).

The images below, in Fig. 13, show powdered copper after electrolysis from sample 1, captured by SEM in backscattered electron mode. The image (Fig. 13(a)) at 500x magnification shows particles that tend to clump together. The surface of the particles in this image is finer compared to the image in Fig. 12(a), which captures copper obtained from sample 2. The higher porosity of the particles in the image could be the result of chemical leaching, which removed some of the constituents from the input copper material. The electron image shown in Fig. 13(b) clearly shows a distinct morphological structure. Similar to sample 2, copper particles with distinct sharp edges can be observed.

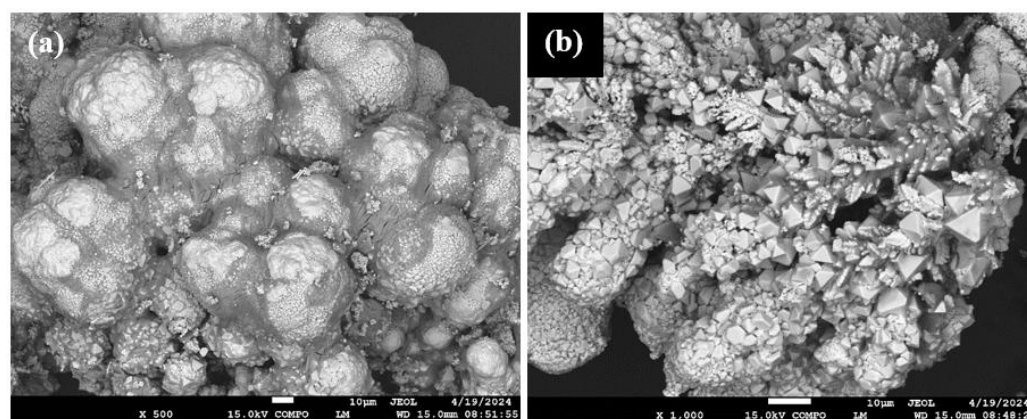


Fig. 13 STEM images of powdered copper after electrolysis (sample 1) in the back-reflected electron mode at magnifications of (a) 500x and (b) 1,000x (scale bars on the images 10 µm).

Comparing these images from Fig. 13 with the previous ones (Fig. 12), it is clear that the electrolysis of the leachates (samples 1 and 2) yielded powdered copper with only slight differences in structure. While the formation of more clumping particles with relatively smooth surfaces and less defined crystals can be observed in sample 2, the electrolysis of sample 1 produced more distinct crystalline structures that appear to be more structured and have more porosity. The different particle structure may affect the physicochemical properties of the material, such as electrical conductivity, which is important for their potential industrial applications.

The copper after electrolysis from samples 1 and 2 was further processed, i.e., it was smelted and subsequently transformed.

Comparison of results and discussion

The above experimental results show that the transition of copper from the waste material proceeded efficiently during leaching in H_2SO_4 . It was found that the leaching process was more efficient at the elevated temperature of 60 °C, which could be due to the increased kinetics of chemical reactions at higher temperatures. This result aligns with information reported in the literature, which confirms that temperature can significantly impact the rate and efficiency of copper leaching from solid materials (Cocchiara et al., 2019; Drápala et al., 2022).

The next step in the experiment involved treating the leachate through electrolysis to obtain powdered copper as a secondary raw material. This process of converting waste into useful material is key to the circular economy and is an important step for improving the sustainability of material resources. Furthermore, it was demonstrated how copper, in the form of secondary raw material, can be processed and subsequently utilized. In this particular case, the recovered copper was subjected to further processing, namely smelting and forming.

Given these results, it is clear that optimizing the leaching and electrolysis conditions, such as controlling temperature, pH, and electrolyte composition, can lead to better quality of the recovered copper. Furthermore, research should be carried out into the possibility of separating impurities during electrolysis, which could positively influence the results in relation to the purity and properties of the final product. This approach could not only increase the quality and financial value of the recycled copper but could also contribute to a more sustainable processing of the materials. The above suggestions may be the subject of further research.

Further research in metal-bearing waste recycling is likely to focus on the development of metal-bearing material processing technologies that can be used to recover high-purity elemental metals from secondary raw materials. The result could be a system of metal management in the industrial production process that is both environmentally friendly and capable of using all metal-bearing waste.

Data availability

The data that support the findings of this study are openly available in [Zenodo] at <https://doi.org/10.5281/zenodo.14921588> (see Last Reference).

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