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Electrochemical Behaviour of Copper Powder Recovery by Electrodeposition Process from Industrial Brass Waste

Shahad Moqbel Abdulsattr†, F. F. Sayyid, and Sami I. Jafar Al-Rubaiey

Department of Production Engineering, and Metallurgy, University of Technology-Iraq, P.O. Box: 10001, Baghdad- Iraq

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The process of recovering copper from industrial brass waste is a significant focus of scientific research, especially as the volume of this waste increases daily due to the frequent use of brass in various industries. This research aims to study the electrochemical behavior of recovering copper in the form of an ultra-fine, high-purity powder using the electrochemical deposition method. Rectangular samples of industrial brass waste, measuring 12 cm in length, 2.5 cm in width, and 1.5 mm in thickness, were used. The electrochemical deposition method involved recovering copper powder using a chemical solution consisting of 1M H2SO4 and CuSO4·5H2O. Different voltages were applied, specifically 0.8, 0.9, and 1 volt, for a constant time period of 30 minutes. The results showed that high purity reached 99.2% at an applied voltage of 0.9 volts, while the maximum deposit of copper powder was 2.5 g at an applied voltage of 1 volt. An ultra-fine copper powder was obtained with a grain size of 4.3 micrometers. The electrochemical behavior also indicated the possibility of precipitating copper and zinc oxides, along with some impurities, during the electrostatic deposition process from industrial brass waste.

Keywords: *Brass, Electrochemical, Tafel technique, Copper powder*

1. Introduction

The demand for copper is constantly increasing due to its important role in power generation and industrial engineering. As naturally occurring copper deposits decline, the recovery and reuse of copper from secondary sources such as scrap metal will become increasingly important. Electrochemical restoration strategies provide a sustainable and green way to get rid of lead from waste substances [1,2].

Brass turned into once an alloy made of copper and zinc. Because of its practical mechanical houses, resistance to corrosion, and appealing look, brass may be very useful in many one-of-a-kind applications, inclusive of plumbing, electric additives, and musical contraptions [3].

Brass alloys can be divided into three primary groups according to their composition and properties, as shown by the copper-zinc phase diagram in Fig. 1. [4,5]:

1. Single-section brass (α brass): constitutes a single-phase α stable answer and has a zinc attention of much less than 36%. This kind is frequently hired within the manufacturing of ammunition cartridge instances, bolts, screws, and pins.

2. Duplex brass, or α β brass, has a zinc content of 36% to 45 %. It can be used for numerous fittings, sprinkler heads, and tap handles.

3. Single phase brass, generally called β copper, is an alloy made normally of copper and zinc Compared to α copper which has a different phase composition and generally lower zinc content, it has a higher zinc content and β-phase crystal structure Beta copper usually contains 60–70% copper and 30–40% zinc in the contents of the. Although its ductility is very low, its composition contributes to its unique properties such as strength and toughness, superior to α copper and machinability. These are designed for specific purposes in the commercial and industrial sectors.

Fig. 1 shows the instant impact of zinc-containing coatings at the coloration and appearance of copper. Copper yellow is typically extra yellow in color and carries less zinc. Copper yellow is greater red in color and consists of extra zinc.

The presence of certain metal ions, such as zinc, in the electrolyte can hinder the electrodeposition of copper. This can lead to preferential deposition of zinc, reducing the efficiency

†Corresponding author: pme.22.07@grad.uotechnology.edu.iq

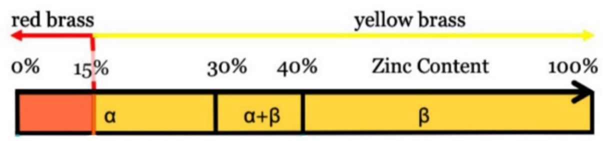


Fig. 1. General trends for brass alloys regarding copper and zinc content

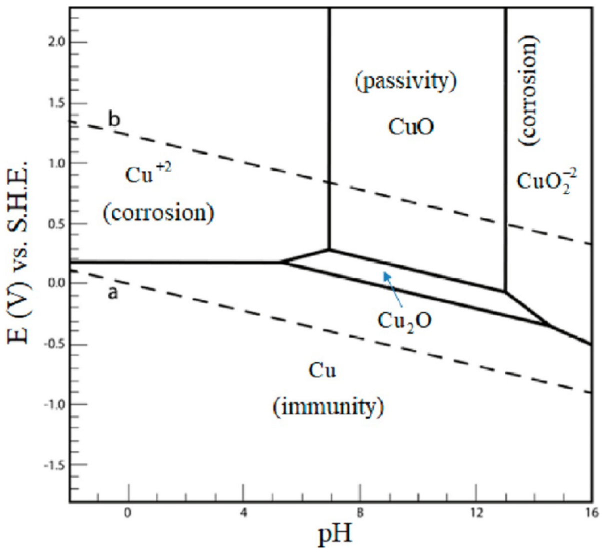


Fig. 2. Pourbaix Diagram for Copper [6]

and purity of copper recovery. To ensure high-quality copper deposition, it is essential to carefully control the voltage and solution composition. The impurities present in brass waste can also negatively impact the copper recovery process, emphasizing the need for meticulous control of deposition parameters and conditions to achieve high-purity copper.

Numerous factors effect the electrochemical traits of copper and zinc. Pourbaix diagrams can be used to observe the precise electrochemical behaviors that each metal shows. The stability zones of numerous copper species underneath diverse pH and capacity (Eh) settings are proven in Fig. 2[6].

Fig. 2 shows that the copper is in a state of corrosion due to the fact $Cu2^{+}$ ions predominate at low pH and excessive capacity. CuO (copper oxide) seems at even better pH stages, even as CuO (cuprous oxide) forms as pH rises. Metallic copper is stable and impervious to corrosion at low pH and occasional ability, offering vital insights into how copper behaves in diverse environmental settings.

For the motive of comprehending and measuring the electrochemical conduct of metals and alloys, electrochemical strategies like Tafel evaluation are vital. Tafel plots, which can be produced by means of measuring polarization, offer data approximately the kinetics of both cathodic and anodic reactions in addition to the formation or dissolution of oxides in one of a kind electrochemical setting. These techniques are especially useful for learning complex alloys, which includes brass, in which numerous electrochemical reactions may take region straight away.

In the research (2024) [7] had introduced several innovative approaches to copper recovery. explored the use of camel saliva alpha-amylase to synthesize copper nanoparticles, highlighting its eco-friendly nature, although challenges such as complex biological 3, scalability issues, and potential impurities remain. Chen et al. (2023) [8] achieved a copper recovery efficiency of 97.11% with 98.47% purity by employing an NH3·H2O–NH4Cl slurry electrolysis system for processing reclaimed copper smelting fly ash. Liu et al. (2022) [9] attained 99.1% Faraday efficiency in copper deposition from waste printed circuit boards using a pH-neutral ethylene glycol electrolyte. Additionally, leaching techniques have shown promise: Mokhlis et al. (2021) [10] reported a 92.5% copper recovery efficiency from printed circuit boards using glycine as a leaching agent, while Mingxin et al. (2021) [11] demonstrated a 99% recovery rate from acidified sediments in gold processing using thiosulfate leaching.

The observe underscores the importance of understanding electrochemical conduct to enhance recuperation strategies and foster sustainable practices within the metallurgical enterprise. The primary objective is to analyze the electrochemical behavior of brass alloys to perceive most suitable voltage degrees for the electrochemical deposition of copper powder. This research goals to establish situations for acquiring high-purity, quality-sized copper powder from commercial brass waste, contributing to more sustainable useful resource control practices.

2. Experimental Work and Producer

2.1 Materials

2.1.1 Industrial Brass Waste

In this research, industrial brass waste was used in the form of plates 50 × 10 cm and 0.15 cm The plates were brought from local market. Copper and zinc are the two principal constituents of brass waste, although some small trace elements may also be present. Table 1 shows the chemical analysis of the industrial brass waste used in this work.

The spaceman of Brass waste plates was prepared by cutting the plate into dimensions 12 × 2.5 cm with thickness of 1.5 mm. All the spacemen were cleaned and drilled, polished. The polishing was carried out with silicon carbide paper grit size of 800 for the spacemen's and of 1000 for the cathode plates. Tafel Technique was carried out using a spaceman of rectangular area with 1 cm².

Table 1. Chemical Composition of Industrial Brass Waste

Continent	Percentages %
Cu	60.30
Zn	32.66
Others	6.45

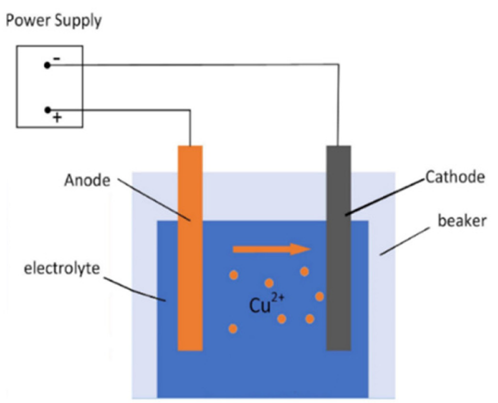
2.1.2. Electrolyte Solution

The electrolyte solution contains of 1 M $CuSO_4 \cdot 5H_2O$ and H_2SO_4. The pH of the solution was 2.5. All experimental were done at home temperature

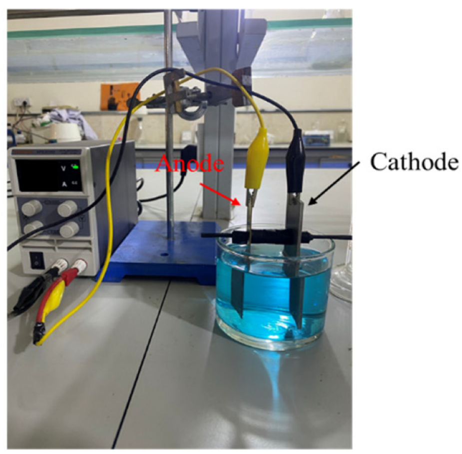
2.1.3. Electrodeposition of Copper Powder

The electrodeposition cell for the electrochemical behavior in the experiments were consistent of a three-electrodes. The working electrode was the spaceman of brass waste brass which used as the anode electrode. Platinum electrode was employed as the auxiliary electrode. A saturated calomel reference electrode [SCE] was used for potential measurement. In a copper recovery process. The anode was made of industrial brass waste plate and cathode was stainless steel 316 plate in our experimental electrochemical cell. Fig. 3 shows Schematic diagram of the electrodeposition cell.

Copper Powder Produced by electrodeposition. The



A)Schematic electrodeposition cell setup



B)Real electrodeposition cell setup

Fig. 3. Electrodeposition cell setup

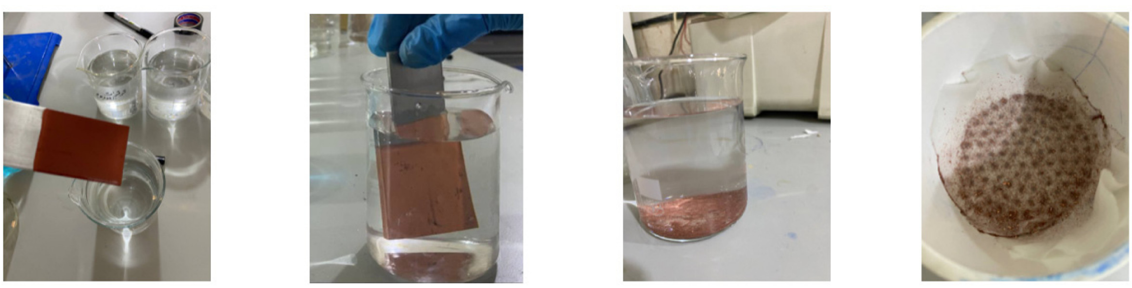


Fig. 4. Stages of powder production by the electrodeposition process

Fig. 4 illustrates the stages of powder production. This stages involve: the formation of copper ions, then metal ions migrate to the cathode where they are reduced to the metallic atom. The atoms deposit either on the cathode surface or in the solution. At a later stage, the powder in collected and several analyses where done.

2.2 Analytical Techniques

In this work, techniques such as XRF, SEM-EDS, and XRD were used to establish the quality of copper recovery. These characterizations gave enough evidence of the purity, morphology, and crystallinity of the copper. They also provided important information related to impurities, structural features, and possible applications that enriched the knowledge about the process of electrochemical deposition and its outcome.

2.2.1 X-ray Fluorescence (XRF) Analysis

The chemical composition of the produced copper powders was analyzed using X-ray fluorescence (XRF) with an XEPOS model from Germany, type 76004814, which operates at a maximum power of approximately 100 VA, in the Laboratory of the Department of Applied Sciences. XRF is used to determine the elemental composition of the recovered copper. It provides quantitative data on the purity of the copper by measuring the intensity of fluorescent X-rays emitted from the sample when it is excited by a primary X-ray source.

2.2.2 Particle Size Analysis

Dynamic Light Scattering (DLS) describes a technique that measures the particle size and distribution of submicron particulate systems. Probe sonication was shown to be the preferred method for dispersing metal, ethanol that was added to the DLS device, Model 90 Plus, and included powder that had been evenly distributed to prevent particle agglomeration that had collected in the bottom of the storage cans. In the Nanotechnology and Advance Materials Research Unit, precision is usually within ± 1%.

2.2.3 Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDX)

Surface morphology, including highly rough surfaces, was characterized by an FEI scanning electron microscope model INSPECT S-50, Model 1450. The mentioned SEM was put into service for imaging at resolutions reaching the lower orders of nanometers and was accelerated within a voltage range starting from 200 up to 30,000 V. The SEM analyses were carried out at the SEM Laboratory of the Department of Applied Sciences. The elemental composition of samples was also done by Energy Dispersive X-ray Spectroscopy EDX BRUKER model. This enables the identification and quantification of element compositions in very small areas of the sample down to the limit of several cubic micrometers, investigation By AlKhora Company. SEM provides high-resolution images of the surface morphology of the deposited copper, while EDS allows for elemental analysis of the sample. This combination helps in assessing the uniformity and structure of the copper powder. SEM-EDS can reveal the particle size distribution, shape, and surface characteristics of the copper powder, which are critical for understanding its performance in applications. It can also detect the presence of any surface contaminants or secondary phases that may affect the quality of the copper.

2.2.4 Particle Characterization by X-ray Diffraction (XRD)

Copper powders were characterized by X-ray diffraction analysis using an X-ray Diffractometer, Type XRD-6000 SHIMADZU-made Japan. The ray target was Cu Kα1 with a wavelength of 1.54060A. Voltage supplied was 40.0kV and a current of 30.0mA. Scanning was effected by step 0.0500 deg, measuring the range 40-800 two theta. XRD is used to analyze the crystalline structure of the deposited copper. It identifies the phases present in the sample and assesses the crystallinity and purity of the copper.XRD can provide information on the crystallite size and the degree of crystallinity, which are important for determining the mechanical properties of the copper. It can also help in identifying any unwanted phases or compounds that may have formed during the deposition process.

2.3 Mass measurement

The digital weighing scale is designed for precisely weighing any valuable item. It supports up to 500 grams of maximum weight and gives precision up to 10 milligrams or 0.01 grams. In the given weighing scale, the size of the weighing platform is 10 cm by 10 cm in size.

3. Results and Discussion

3.1. Electrochemical Behaviour of Brass Waste

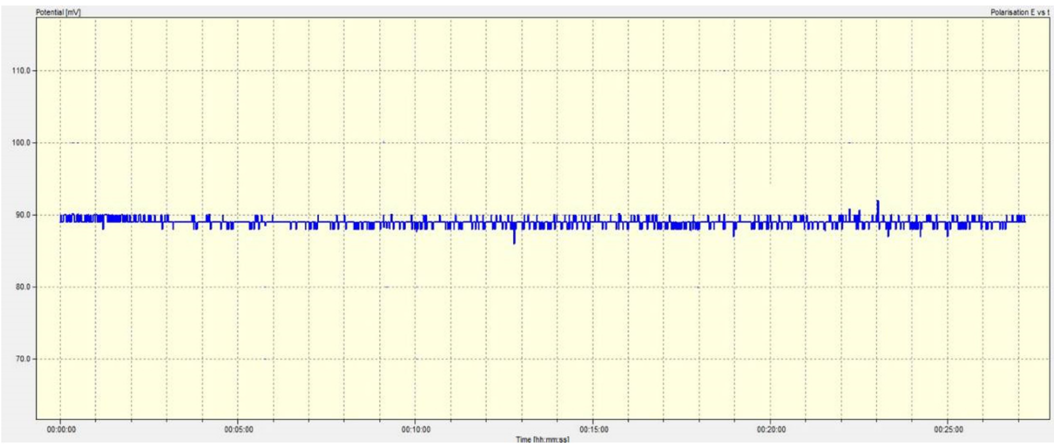
The electrochemical behaviour of brass waste was evaluated using Open Circuit Potential (OCP) and Tafel analysis. As shown in the Fig. 5 and Table 2.

Fig. 5 displays OCP and the Tafel plot for industrial brass waste The main parameters obtained are outlined in Table 2.

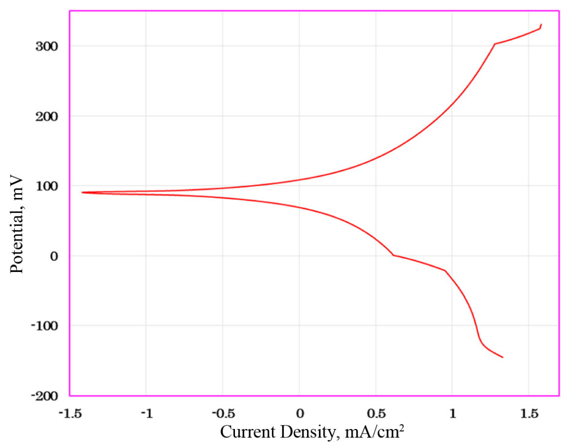
The stable OCP at 89 mV suggests that brass maintains a consistent electrochemical state, which is crucial for understanding its stability in practical applications.

Tafel analysis provides an effective means of elucidating the kinetics of the anodic and cathodic reactions by their overpotential-current density relationship. Such a method will reveal several key features:

1. Kinetic Parameters: The anodic Tafel slope, β anodic, and cathodic Tafel slope, $\beta_{cathodic}$, characterize the sensitivity of reaction rates with voltage. For instance, a



a) Open circuit potential (O.C.P) of brass waste specimens after 30 minutes in 1 M $CuSO_4{\cdot}5H_2O$ and H_2SO_4



b) Tafel extrapolation results for industrial brass waste

Fig. 5. Electrochemical Analysis of Brass Waste: a) Open Circuit Potential and b) Tafel Extrapolation Results

Table 2. Tafel Extrapolation Parameters for Brass Sample Analysis

Sample	OCP mV	$E_{Corr.}$ mV	$I_{Corr.}$ mA	β_{Anodic} mV/Dec.	$\beta_{Cathodic}$ mV/Dec.	C. R. $g/m^2.d$	C. R. mm/a	C. R. mpy
Brass	+89	+89.8	4.21	330.4	315.3	1200	49.0	1929

β_{anodic} of 330.4 mV/dec describes the voltage required to increase the current density tenfold, while $\beta_{cathodic}$ of 315.3 mV/dec describes the voltages for the reduction reactions.

2. Corrosion Current Density: The corrosion current density, i_corr, is 4.21 mA/cm², and this value is useful for the evaluation of stability in relation to the electrochemical system about possible problems with corrosion.

3. Optimum Conditions: By integrating the Tafel data with the Pourbaix diagrams, this work determined the operating conditions that were optimal for copper recovery, in particular, where efficient extraction occurred within bounds of applied potential from 0.8 V and 1 V at pH values between 1 and 7.

4. Improving Recovery Efficiency: The understanding developed from the Tafel analysis has scopes of application for improvement in copper recovery efficiency by:

· Adjusting Voltage and Current: The operators can change the voltage and current density in order to optimize copper's deposition rates and purity.

· Reducing Side Reactions: Knowledge of reaction kinetics also helps reduce the side reactions that lead to the formation of unwanted compounds.

· Tailoring Electrolyte Composition: This analysis could present some insight with respect to any changes in the electrolyte composition for accommodating any of the specific reactions desired, thereby reducing impurities.

At last, Tafel analysis is a powerful tool for realizing the entire electrochemical process of copper recovery from wastes of brass, which can further improve the recovery efficiency.

Pourbaix Diagrams: These diagrams illustrate copper's thermodynamic stability under various pH and potential (Eh) conditions. For instance:

· **At pH = 2.5 and E = 0.8 V:** Cu^{2+}ions dominate, indicating copper dissolution.

· **At pH = 8 and E = 0.8 V:** Cu(OH) precipitation occurs, hindering copper recovery.

By integrating these analyses, ensures effective copper deposition without forming unwanted compounds like $Cu(OH)_2$ and achieving a purity to obtain a high-quality recovery. To enhance the copper recovery process and minimize the formation of unwanted byproducts like $Cu(OH)_2$, several electrochemical parameters can be optimized:

pH Control: The pH value should be optimized. A lower pH, at about 2.5, favors the existence of Cu^{2+} ions, and thus supports dissolution, while higher pH values allow $Cu(OH)_2$ precipitation. A balanced pH may reduce the formation of byproducts.

Voltage Control: Voltage is a very critical parameter in the process. If the operating voltage is near 0.9 V, reduction of Cu^{2+} ions to metallic copper will be favored when conditions for hydroxide formation are minimized.

Electrolyte Concentration: Copper sulfate ($CuSO_4$) and sulfuric acid (H_2SO_4) concentrations must be optimized for the result of recovery rates of copper to be increased and the production of hydroxides reduced.

A thorough understanding the electrochemical behavior of brass through open-circuit potential (OCP) and Tafel analyses lays a solid foundation for refining recovery processes and improving overall efficiency in copper extraction.

3.3 Electro-deposited Recovery of Copper Powder

Electrodeposition is an adaptative technique to achieve copper powders with defined properties. Proper control of deposition parameters will allow the copper powder to meet the requirements of most applications. This method can reach very high purity for copper powder, by which one can meet the requirements of different applications that require very strict control of metal characteristics. The interaction plot shows that purity is being improved

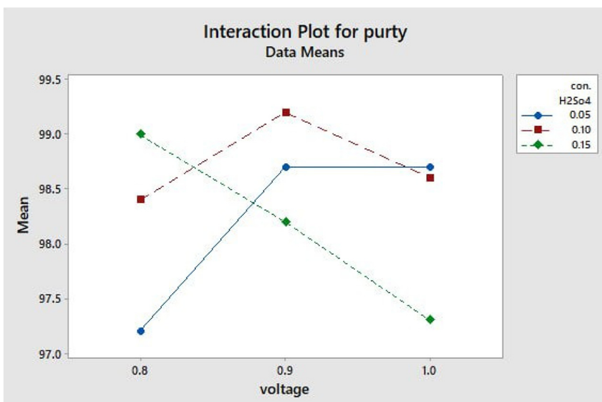


Fig. 6. Effect of cell voltage on copper deposited at the cathode and copper recovery

Table 3. Comparative Analysis of Copper Recovery Techniques: Purity and Electrolyte Information

Study	Technique	Copper Purity	Electrolyte/Leaching Agent
Current Study	Electrolysis with $CuSO_4$ and H_2SO_4	99.2%	1M of sulphate solution
Chen et al. (2023)	$NH_3{\cdot}H_2O–NH_4Cl$ slurry electrolysis	98.47%	$NH_3{\cdot}H_2O–NH_4Cl$ slurry
Liu et al. (2022)	pH-neutral ethylene glycol electrolyte	99.1%	pH-neutral ethylene glycol

by three most important variables: voltage, concentration of sulfuric acid (H_2SO_4), and awareness of copper sulfate ($CuSO_4$) vs its contents, as presented in Fig. 6.

Fig. 6 shows that a purity of 99.2% Cu at an optimized parameters applied intermediate voltage of about 0.9 V with 0.1 M H_2SO_4 solution. Analyzing, one can deduce the following from this batch of experimental results:

· **Optimal Voltage**: A voltage of 0.9 V is ideal for copper deposition, as it effectively prevents the formation of unwanted compounds, such as $Cu(OH)_2$.

· **Copper Purity**: Under these conditions, copper recovery can reach a purity of 99.2%, producing a high-quality product.

· **Copper Deposition**: A significant copper powder deposit of 2.5 g can be achieved when a voltage of 1 V is applied.

Table 3 compares current research associated with proposed work of recovery of copper.

The data from Fig. 6 and Table 3 illustrate that:

1. Current Study: Achieves the highest copper purity of 99.2% with the optimal combination of 0.9 V voltage, 0.1 M H_2SO_4, and 0.3 M $CuSO_4$. This demonstrates the effectiveness of this method in enhancing copper purity.

2. Chen *et al.* (2023): achieved a high purity of 98.47% by $NH_3{\cdot}H_2O–NH_4Cl$ slurry electrolysis, relatively low compared to the current study.

3. Liu *et al.* (2022): Achieves 99.1% for their pH-neutral electrolyte of ethylene glycol, which is almost the same as in this study.

In comparison, the electrochemical deposition route used in this work had some advantages over other copper recovery routes:

1. High Purity: The purity of copper obtained with the electrochemical deposition technique was 99.2%, higher than what was obtained with other techniques such as Chen et al. and Liu *et al.* Generally speaking, such high purity is indispensable in applications which require stringent standards for quality.

2. Efficiencies in Recovery: The processes portrayed a sensible deposit yield of 2.5 grams of copper powder, showing that the route is efficient enough in recovering copper from industrial wastes of brass.

3. Conditions Controlled: This will allow for the optimization of parameters such as voltage, pH, and electrolyte composition to precisely control the deposition process and hence limit any side reaction that could lead to such byproducts as $Cu(OH)_2$. This will ensure better material properties and good quality copper.

4. Ultra-fine Powder Production: The ultra-fine copper powder with a small grain size down to 4.3 micrometers obtained by the electrochemical deposition method is promising for many applications requiring fine materials.

5. Adaptability: The electrochemical deposition process is adaptable to several forms of industrial wastes. This makes the technology versatile in recovering copper from various sources.

6. Environmental Benefits: The process may reduce the environmental impact of conventional copper recovery, processes that might involve more hazardous chemicals and processes.

7. Cost-Effective: By making use of wastes and optimizing recovery processes, electrochemical deposition tends to be cost-effective compared to the conventional methods, which may involve extensive processing and purification steps.

Finally, electrolysis involving such concentrations of the electrolyte gives the highest ever recorded purity of copper compared with other new techniques.

3.6 Characterization of Cu Deposited on the Cathode

The electro-deposited copper under the optimal conditions (potential 0.9 V, 0.1 M H_2SO_4, 0.3 M $CuSO_4$ at room temperature for 30 min time length) was collected and analyzed by XRD and SEM-EDS. The XRD analysis presents the diffraction peaks of Cu as the dominant phase with the absence of any other impurities as show in Fig.

7. With regards to the corresponding SEM-EDS results, it shows that the morphology of the obtained Cu powder on the cathode is relatively uniform and the major component is the Cu metal, as show in Fig. 8.

Fig. 7 XRD pattern indicates that the major constituent of the deposited copper product is pure, crystalline copper, as reflected in the sharp and distinctive peaks representative of the (111), (200), (220), and (311) lattice planes. These peaks are representative of FCC, which is expected from copper. This shows a high degree of crystallinity; the intensity and clarity of the peaks denote that the copper is atomically well ordered. In addition, given the existence of those specific planes, it can also be said that there has been enough growth and maturation during deposition to

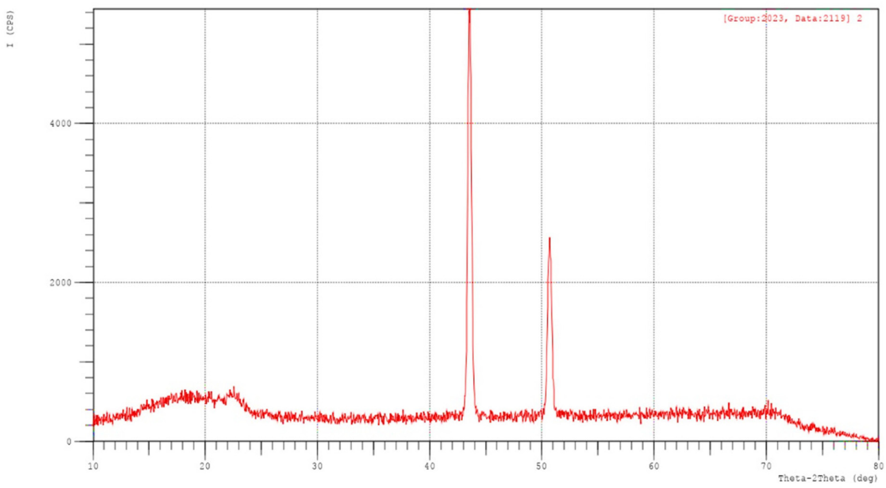


Fig. 7. XRD Analysis of Copper Deposits on the Cathode Under Optimal Conditions

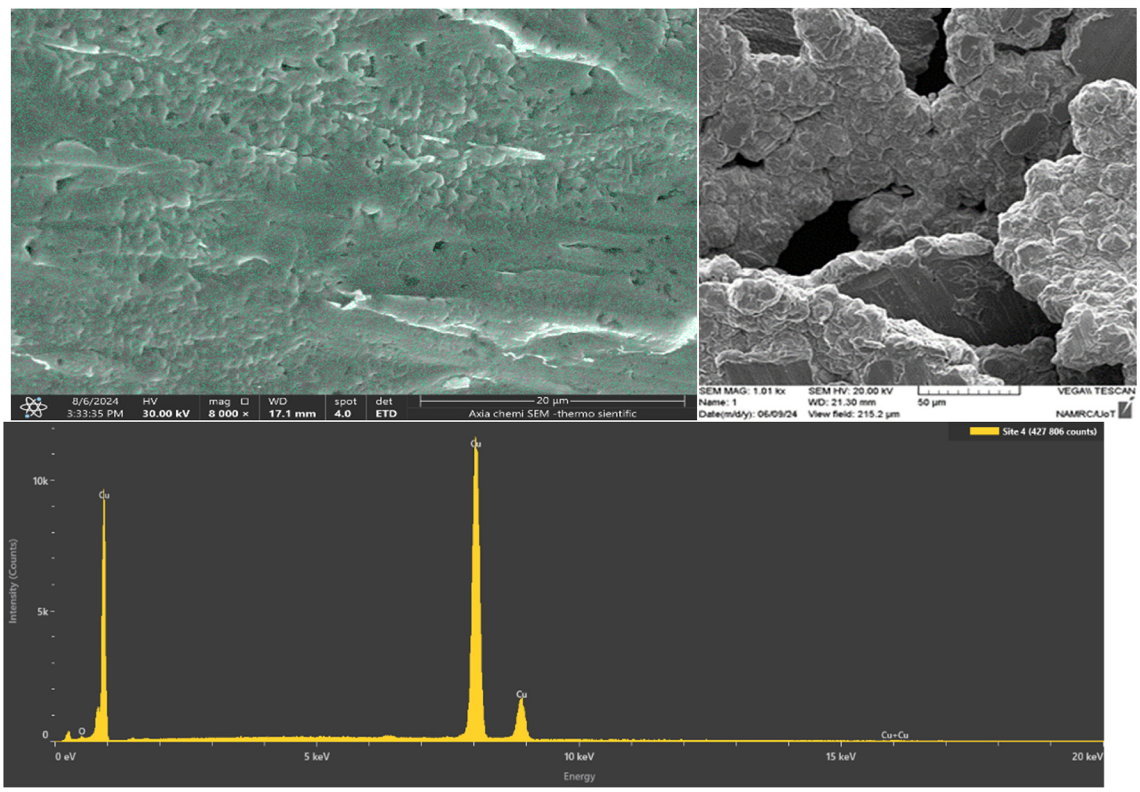


Fig. 8. SEM-EDS Characterization of Copper Deposits on the Cathode Under Optimal Conditions

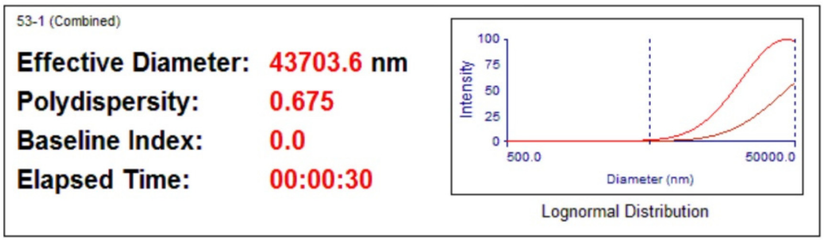


Fig. 9. Particle Size Distribution of Copper Powder Deposited for 30 Minutes Under Optimal Conditions

Table 4. Extracted Parameters from EDS Analysis of Copper Deposits

Element	Weight %	Weight % Error
O	0.8	0.1
Cu	99.2	0.4

provide a higher mean crystallite size and, thus, better improvement of the material properties. The overall confirmation of the quality and purity of the deposited copper by XRD analysis ensures that even after being subjected to various applications, it remains suitable.

Besides, SEM gives handy information on surface morphology of the copper layer; hence, it provides complete observations on texture. Complementarily supporting these, Fig. 8 shows the EDS images of elemental constitution and gives the degree of purity for the deposited material.

Fig. 9. Copper particle distribution at optimum conditions after 30 minutes of deposition information on the sizes of particles obtained during deposition.

Fig. 8 and Table 4 demonstrate that the deposited copper product exhibits a high level of purity, with approximately 99.2% copper content. The remaining 0.8% of the sample is primarily composed of oxygen (O). This high copper purity is a promising outcome, indicating that the electrolysis process used for deposition was efficient in producing a high-quality material.

Fig. 9 shows that the copper powder produced under optimal conditions has a relatively broad particle size distribution, with a majority of particles falling within the smaller size range.

4. Conclusions

The present research has further demonstrated that the electrochemical deposition technique is truly viable for recovering ultra-fine copper powder of very high purity from industrial brass waste. Some concludes as the following:

1. Optimal Voltage-Purity: A voltage of 0.9 V with 0.1 M H_2SO_4 and 0.3 M $CuSO_4$ solution gave the maximum purity for copper as 99.2%. This is the optimum voltage that can prevent the formation of unwanted compounds like $Cu(OH)_2$, thus yielding a superior quality copper product.
2. Copper Deposit Yield: A respectable deposit yield of 2.5 grams of copper powder was realized at 1 V, which goes to underscore the efficiency of the deposition process at higher voltages.
3. Purity-Comparative Performance: The present study stands better compared to the other techniques with regard to purity. The copper purities obtained from the study are higher, 99.2%, compared to Chen et al. at 98.47% and Liu *et al.* at 99.1%. This ascertains that electrolysis used in this study is highly effective and can generate high-purity copper.
4. Ultra-Fine Powder: The use of electrochemical deposition gave an ultra-fine copper powder with a grain size as small as 4.3 micrometers, which was highly faithful to the precision of the method.
5. Electrochemical Behavior: Besides, the precipitation of copper and zinc oxides and other impurities was also mentioned, which might occur during the electrochemical deposition process; hence, again more emphasis was on the deposition conditions with a view to attaining maximum purity.

Finally, the electrochemical deposition technique used in the present research sets a new record in copper recovery from industrial wastes, offering the highest purity ever recorded when compared to other techniques.

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